Remedial Investigation/Feasibility Study Work Plan
GLLA Program – Lower Scajaquada Creek Operable Unit

Buffalo, New York

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Acronyms and Abbreviations

ADD	Average Daily Dose
AOC	Areas of Concern
AWQS	Ambient Water Quality Standards and Guidance
ASP	Analytical Services Protocol
AST	Above-Ground Storage Tank
ASTM	ASTM International (formerly American Society for Testing and Materials)
BCP	Brownfield Cleanup Program
bgs	Below ground surface
BSA	Buffalo Sewer Authority
BTEX	Benzene, Toluene, Ethylbenzene, Xylene
BUI	Beneficial Use Impairment
CAMP	Community Air Monitoring Plan
cm/s	Centimeters per second
COPC(s)	Constituent(s) of Potential Concern
COPEC(s)	Contaminant(s) of Potential Ecological Concern
CSM	Conceptual Site Model
CSO	Combined Sewer Overflow
CVOCs	Chlorinated Volatile Organic Compounds
DER	NYSDEC Division of Environmental Remediation
DNAPL	Dense Non-Aqueous Phase Liquid
DUSR	Data Usability Summary Report
EDDs	Electronic Data Deliverables
EDR	Environmental Data Resources
EIMS	Environmental Information Management System
EPA	United States Environmental Protection Agency
EPC	Exposure Point Concentrations
ESCs	Engineered Source Controls
EPH	Extractable Petroleum Hydrocarbons
FS	Feasibility Study
FSP	Field Sampling Plan
GDIT	General Dynamics Information Technology, Inc.
GEI	GEI Consultants, Inc.
GLLA	Great Lakes Legacy Act
HASP	Health and Safety Plan
HSA	Hollow-Stem Auger
IRM	Interim Remedial Measure
LNAPL	Light Non-Aqueous Phase Liquid
MAH	Monocyclic Aromatic Hydrocarbons
MGP	Manufactured Gas Plant
mg/kg	milligrams per kilogram

NAD83	North American Horizontal Datum 1983
NAPL	Non-Aqueous Phase Liquid
NAVD88	North American Vertical Datum 1988
NOAA	National Oceanic and Atmospheric Administration
NYS	New York State
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
NYSDOT	New York State Department of Transportation
NYSTA	New York State Thruway Authority
OU	Operable Unit
PAH	Polycyclic Aromatic Hydrocarbon
PA	Project Agreement
PCE	Tetrachloroethylene
PID	Photo-ionization Detector
PPE	Personal Protection Equipment
ppm	Parts per million
PVC	Polyvinyl chloride
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
RCRA	Resource Conservation and Recovery Act
REC	Recognized Environmental Condition
RAO	Remedial Action Objectives
RI	Remedial Investigation
SC	Site Characterization
SGVs	Sediment Guidance Values
SVOC	Semi-Volatile Organic Compound
TAL	Target Analyte List
TCL	Target Compound List
TCE	Trichloroethylene
TPH	Total Petroleum Hydrocarbon
TRV	Toxicity Reference Value
UCL	Upper Confidence Limit
USGS	United States Geological Survey
VCP	Volunteer Cleanup Program
VOC	Volatile Organic Compound

1. Introduction

This Work Plan has been developed on behalf of the National Fuel Gas Distribution Corporation (National Fuel) to describe the planned approach for conducting a Remedial Investigation (RI) and Feasibility Study (FS) in the Lower Scajaquada Creek area in Buffalo, New York, identified as the Lower Scajaquada Creek Operable Unit (Scajaquada Creek OU) (Figure 1). The RI focuses primarily on the areas south of the West Avenue bridge and includes upland areas bordering the Lower Scajaquada Creek and the Scajaquada Creek Slip area (also known as the Inlet (Figure 2)).

The New York State Department of Environmental Conservation (NYSDEC) issued an Order on Consent on June 23, 2021, requiring National Fuel to complete a Site Characterization (SC) in response to the discovery of dense non-aqueous phase liquid (DNAPL) believed to originate from historic manufactured gas plant (MGP) operations known as the Iroquois Gas/Westwood Pharmaceutical Terrestrial/Riparian Site that produced water-gas tar (Figure 3). The MGP source area was remediated over two decades ago, and engineering controls were installed to contain and remove mobile DNAPL in areas north of the West Avenue bridge.

The SC investigation (GEI, October 2022) identified DNAPL downstream from the West Avenue bridge in soils above an area-wide, red-brown clay layer, and in shallow creek sediment in the Inlet near the discharge from a City of Buffalo Pumphouse. The RI will determine if the presence of DNAPL above the clay layer in downgradient areas is associated with historic releases (pre-2000) prior to implementation of engineering controls at the Iroquois Gas/Westwood Pharmaceutical Terrestrial/Riparian Sites. Additionally, the SC investigation revealed comingled contamination in groundwater, soils, and sediments from non-MGP sources, including urban runoff, Combined Sewer Overflow (CSO) discharges, and releases from historic industries that operated in the area since the late 1800s, some of which are in the NYSDEC Brownfield Cleanup Program (BCP).

The SC Report recommended a remedial investigation to assess the extent of DNAPL and an evaluation of remedial alternatives through a Feasibility Study. The NYSDEC agreed with this conclusion and supported National Fuel's participation as a partner in an existing Project Agreement (PA) between the NYSDEC and the US Environmental Protection Agency (USEPA) under the Great Lakes Legacy Act (GLLA) program to address contamination in the Black Rock Canal and Scajaquada Creek.

1.1. GLLA Program and Niagara River Area of Concern

The GLLA program is a vital initiative aimed at addressing contaminated sediment in Areas of Concern (AOCs) within the Great Lakes Basin. Scajaquada Creek is a tributary of the Niagara River and discharges to the Black Rock Canal. Scajaquada Creek and the Black Rock Canal are located in the Niagara River AOC. On October 23rd, 2020 EPA entered into a GLLA Project agreement to address sediment contamination in Scajaquada Creek and the Black Rock Canal. The 2020 Project Agreement between USEPA and NYSDEC (NYSDEC Contract No. C011665) was amended in 2024 to add National Fuel as a non-federal sponsor and incorporate work previously completed by National Fuel from 2021 to 2023 related to the Site Characterization (SC, October 2022), Interim Remedial Measures (IRMs, March 2023) completed to mitigate contamination discharge from a City of Buffalo Pumphouse on Niagara Street, and the

completion of the Scajaquada Creek Remedial Investigation (RI) and Feasibility Study (FS). The Project Agreement was fully executed on January 6, 2025.

National Fuel is the Lead Partner for the Scajaquada Creek Operable Unit (OU) SC, IRMs, RI and FS, while the USEPA is the Lead Partner responsible for completing a Feasibility Study for the Black Rock Canal OU. The RI described in this workplan details work that will be performed by National Fuel in the Scajaquada Creek OU. NYSDEC will also conduct separate investigations during a Site Characterization Study in the Lower Scajaquada Creek OU to determine if there are existing sources affecting creek sediments from historic activities unrelated to former MGP operations. Findings from NYSDEC's SC are anticipated to be considered in the RI/FS report to be completed by National Fuel.

1.2. Scajaquada Creek OU RI/FS Objectives and Scope of Work Summary

The remediation of contaminated sediments in the Black Rock Canal and Scajaquada Creek is expected to contribute to the removal of the following Beneficial Use Impairments (BUIs) in the Niagara River Area of Concern:

- *Restrictions On Fish and Wildlife Consumption*,
- *Degradation Of Benthos*,
- *Bird or Animal Deformities or Reproductive Problems*, *and*
- *Degradation of Fish And Wildlife Populations*.

Additionally, these remedial efforts will promote recreational opportunities in Scajaquada Creek and Black Rock Canal.

Remedial investigation and evaluation of remedial alternatives through a feasibility study are necessary to support the design and completion of remedial actions for the Scajaquada Creek Operable Unit (OU). During scoping discussions, National Fuel and NYSDEC agreed on a framework (dated May 5, 2022) to implement a phased investigation and interim remedial measures (IRM) approach under a Project Agreement with the USEPA through the GLLA Program. This included completing the Site Characterization (SC), implementing an IRM to mitigate DNAPL entry into the storm sewer and Pumphouse and minimize re-suspension of contaminated sediments during discharge, and completing an RI and FS for the Lower Scajaquada Creek area.

As described in the May 2022 Framework for the Phased Scope of Work and the Scope of Work (SOW) for the GLLA Project Agreement, the following work is required to complete the RI and FS for the Lower Scajaquada Creek OU:

- Develop RI/FS Work Plan
- Delineate the vertical/horizontal extent of NAPL/DNAPL and associated dissolved phase contamination
- Assess the effectiveness of containment remedies previously installed upstream of West Avenue
- Assess the fate and transport of dissolved and non-aqueous phases of contamination

- Evaluate potential utility/infrastructure migration pathways to the extent feasible, without causing damage or risking worker safety during investigation
- Evaluate potential exposure pathways
- Evaluate existing sediment chemistry and sediment depth data to define the lateral and vertical extent of contamination to support volume estimates for the FS.
- Identify data gaps necessary to successfully complete the feasibility study
- Identify and evaluate potential structural and hydraulic limitations and any other physical factors affecting the remedial decision-making process. For the Inlet, the evaluation should include, at a minimum, the eastern wall adjacent to 1660 Niagara Street and the I-190 expressway piers
- Obtain additional data on the area-wide clay layer below the Inlet
- Prepare a data usability report
- Summarize RI findings with tables and figures
- Evaluate bioavailability of contaminants, as appropriate
- Develop remedial action objectives (RAOs) based on New York State sediment guidance values and other applicable sediment screening values that are consistent with AOC Beneficial Use Impairments (BUIs) and other appropriate considerations including planned future uses of adjacent areas
- Identify options for remedial actions that have the potential to fulfill the RAOs
- Identify the necessary technologies and general process options for implementing each of the general remedial actions
- Estimate costs associated with each remedial action option
- Based on comparative analysis of the remedial action options, and consultation with project stakeholders, select a preferred remedial action
- Evaluate historical sources of contamination to sediments, as well as ongoing sources of contamination that have the potential to re-contaminate the sediments after remediation
- Meet with NYSDEC/USEPA to discuss progress and management of agreement/contract activities
- Prepare Draft and Final Reports that summarize the findings of RI/FS
- Conduct a public meeting to present FS findings and incorporate comments received by the public into final FS.

As described in Section 1.1, the NYSDEC will be undertaking separate Site Characterization investigation in the Lower Scajaquada Creek OU as part of its cost-sharing responsibilities under the GLLA Project Agreement. This investigation is intended to delineate the nature and extent of Chlorinated Volatile Organic Compounds (CVOCs) on the property at 1660 Niagara Street and ascertain whether CVOC contamination from the site is a source of sediment contamination.

1.3. Work Plan Organization

This RI/FS Work Plan outlines the project objectives, provides background information on historical Site use and current conditions, identifies areas of environmental concern at the Site and adjacent areas, and describes the strategies and methodologies to be implemented during the RI and FS. The remainder of this RI/FS Work Plan includes the following:

- Section 2 – Site Description: Summarizes relevant background information including site location and surrounding land use, topography, geology/hydrogeology, surface water hydrology, wetlands, and waterways.
- Section 3 – Site Investigation Summary and Known Extent of DNAPL: Summarizes the SC investigation findings and known extent of DNAPL that supports the scope of the RI and FS activities.
- Section 4 – Interim Remedial Measures (IRMs): Describes IRMs completed at the Pumphouse to date and contamination that may remain.
- Section 5 – Initial Conceptual Site Model (CSM) and Data Gaps: Introduces the initial CSM, which identifies potential sources of contamination, release mechanisms, exposure media, and exposure routes based on current and potential future site use. The CSM presented in this section is preliminary and will be refined based on data from the RI. This section also identifies data gaps to be addressed in the RI and FS based on available prior studies and data needs.
- Section 6 – Remedial Investigation Scope of Work: Describes work to be performed during the RI for the Scajaquada Creek OU, including management of investigation derived waste (IDW). Additionally, this section references relevant documents including the Field Sampling Plan (FSP) describing field methods, the Quality Assurance Project Plan (QAPP) with data management and submission requirements, and the project Health and Safety Plan (HASP).
- Section 7 – RI Data Summary Report: Provides an outline of the Data Summary Report.
- Section 8 – Qualitative Human Health Exposure Assessment – Describes the performance of risk evaluation for human health.
- Section 9 – Fish and Wildlife Resource Impact Analysis – Describes the performance of the ecological resource characterization and ecological impact assessment per NYSDEC guidance.
- Section 10 – Feasibility Study: Describes the tasks associated with preparing the FS that will develop and assess various potential remedial action alternatives.
- Section 11 – Schedule: Outlines the proposed sequence and timing of RI/FS tasks through preparation and submittal of the FS Report.
- Section 12 – References

Various tables, figures, and appendices are also attached to this document and referenced throughout the text where appropriate.

2. Scajaquada Creek OU Description

A comprehensive overview of the Scajaquada Creek OU was prepared as part of the Site Characterization Report (GEI, October 2022). This report is provided in Appendix A and describes:

- historical and current land uses,
- contamination identified at several nearby properties in NYSDEC's Brownfield Cleanup Program (BCP) and other historic industrial properties with documented environmental impacts,
- geologic and hydrogeologic conditions,
- contaminant impacts to various media (soil, groundwater, creek sediments), and hydrologic conditions in Scajaquada Creek.

This information is summarized below and described in Section 3.

2.1. Area Description and Land Use

The Scajaquada Creek OU encompasses the lands adjacent to Scajaquada Creek, extending from south of the West Avenue Bridge to the creek's mouth where it discharges into the Black Rock Canal. The Scajaquada Creek Slip Area (Inlet Area), which was historically the creek's discharge location before being blocked during the construction of the I-190 New York State Thruway in the 1950s, is also part of the OU.

Upland areas include properties owned by the former Penn Central Railroad (now CSX Transportation), New York State, and City of Buffalo that are used for transportation and infrastructure corridors including a railroad passenger and freight train corridor, the NYS Thruway I-190, the Route 198 Scajaquada Expressway, and City of Buffalo roadways (West Avenue, Tonawanda Street, and Niagara Street). The upland areas also include privately owned properties, many of which were former industrial properties with environmental concerns. Some of those properties have been investigated, remediated, and/or developed for reuse under the NYSDEC Brownfield Cleanup Program (BCP) or the former Voluntary Cleanup Program (VCP) (Figure 3). Most properties in this area are zoned for commercial/industrial use.

2.2. Topography

The ground surface in the Lower Scajaquada Creek area has low topographic relief and gently slopes toward the Creek and Inlet. As shown in Figure 2, the highest elevations in the area are approximately 600 feet (NAVD 88) in the upland areas east of West Avenue. The elevation decreases to about 580 feet (NAVD 88) near the tops of the banks adjacent to the Creek. The creek bank then slopes steeply to the water's edge in most locations, reaching an elevation of 570 to 572 feet (NAVD 88), which corresponds to the typical surface water elevation of the creek.

2.3. Geology and Hydrogeology

2.3.1. Geology

The geology of the Lower Scajaquada Creek area has been characterized through logging of soil and sediment borings. During the 2021/2022 Site Characterization and 2022 City of Buffalo Pumphouse pre-design for the IRM, nine soil and nine sediment borings were completed. Additional data from over a dozen soil borings performed by other parties supported the geologic interpretation.

The geology consists of a layer of fill overlying native soils of alluvial and lacustrine origin. The fill thickness in borings closest to the creek and Inlet ranged from 10 to 19.5 feet and decreased with distance from the creek's edge. The fill materials varied in composition, including reworked red-brown clay soils, black silty sand, and anthropogenic materials like foundry sands, concrete, cinders, and wood debris.

Below the fill, alluvial deposits were identified, becoming coarser with depth. These deposits typically consisted of dark gray to black silt or sand with occasionally decayed woody materials. Deeper alluvium transitioned to black silty sand and fine gravel with wood pieces and shells (gastropods). The alluvium, associated with historic creek sediment deposition, was generally free of anthropogenic materials and ranged from 1 to 22.5 feet in thickness. The thickest alluvium layers were found near the Pumphouse and properties at 1660, 1654, and 1626 Niagara Street, where the historic creek channel had scoured the area-wide red-brown clay layer before depositing the alluvium. The alluvium thickness decreased with distance from the current creek channel.

The bottom of the alluvium was marked by a sharp contact with an area-wide red-brown clay layer which was encountered in all borings completed in the Lower Scajaquada Creek area. The red-brown clay's composition was consistent across borings, and its surface elevation sloped toward the creek in most areas. However, near Niagara Street, the surface elevation of the clay surface is deeper on the north of the Inlet suggesting that the ancestral channel of Scajaquada Creek was situated farther north than its current location. Also, as shown in Figure 4, the width of the alluvial sediments indicates the creek channel was historically broader near the intersection of Niagara and Tonawanda Streets.

Two soil borings advanced to bedrock during the 1995 Remedial Investigation (RI) at the Westwood Former MGP Terrestrial Site and 1956 NYSDOT borings completed for design of the route NY-198 Scajaquada Expressway and I-190 Thruway encountered bedrock at elevations of approximately 500 and 520 feet msl indicating that the area-wide red-brown clay is at least 50 feet thick throughout the Lower Scajaquada Creek area. The boring logs indicate the presence of a sandy till layer above the bedrock. Boring logs are included with the Site Investigation Report (Appendix A).

Within the creek, sediment borings identified a layer of recent fluvial deposits overlying red-brown clay or historic alluvial deposits. These recent deposits were described as black, soft silt mixed with sand and gravel, containing anthropogenic materials such as glass, foundry sand, wood, and occasional zebra mussels. Historic alluvial deposits, similar in composition to those described in soil borings, were identified in several sediment borings below the fluvial deposits.

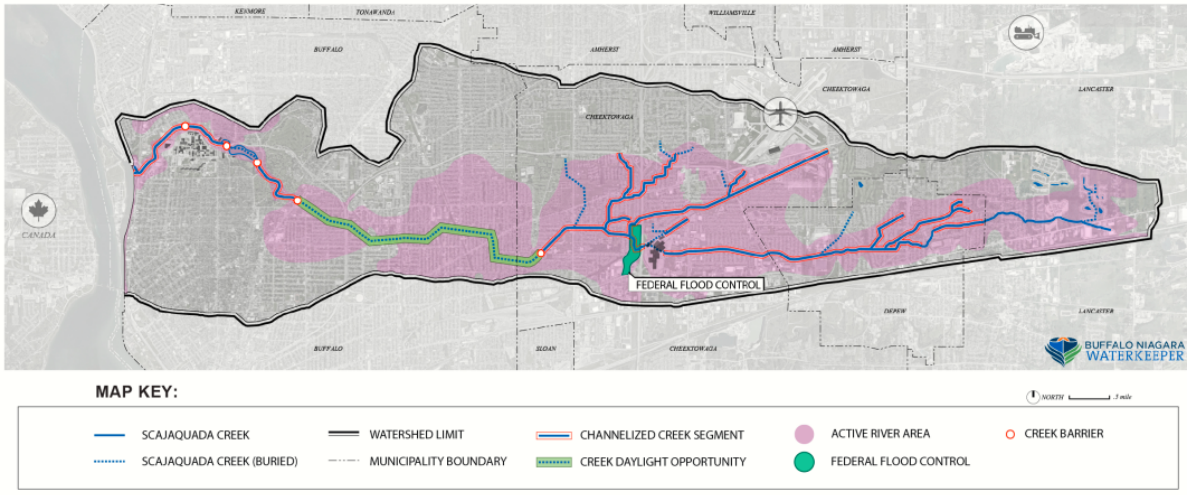
2.3.2. Hydrogeology

Groundwater in the area occurs under unconfined conditions in saturated fill and alluvium. Groundwater and surface water elevation data were recorded in monitoring wells, showing a correlation between creek surface elevations and groundwater levels. Groundwater typically occurs within 10 feet of the ground surface. The groundwater contour maps presented in the Site Characterization Report (Appendix A) indicate a convergent flow pattern toward the creek, with a depressed area of groundwater elevations near Niagara Street and the City of Buffalo Pump Station. The bedrock groundwater encountered during geotechnical NYSDOT drilling, sampling, and rock coring in 1956 for NY-198 Scajaquada Expressway design (see logs in Appendix A) identified artesian groundwater conditions in the bedrock with estimated flow rates up to 15 gpm at the top of the drill stem.

As described in Section 2.3.1, historical changes in the creek channel's positioning have impacted shallow groundwater flow in the creek area. Historical maps of the Black Rock area (see Section 3.2) show that the creek was wider than its current widths. Soil boring data confirm that alluvium soils are thicker beneath the parcels at 31 Tonawanda Street and 1660 Niagara Street. Hydraulic conductivity estimates obtained from well MW-PS-1 and MW-PS-2, located near the City of Buffalo Pumphouse reported values of 3.8 $X10^{-4}$ cm/s and 5.3$X10^{-5}$ cm/s, respectively for the alluvial soils (Appendix A).

2.4. Waterways and Surface Water Hydrology

The Scajaquada Creek watershed covers an urban area of 29 square miles. The source of Scajaquada Creek is in the Town of Lancaster, approximately 18 miles east of the Black Rock Canal at its mouth. A map of the Scajaquada Creek Watershed is shown in the figure below.



The course of the creek has been highly modified and channelized over its length. East (upstream) of the Lower Scajaquada Creek area, a portion of the creek is contained below urbanized areas of Buffalo in a three-mile-long tunnel. Upstream of the tunnel, the creek runs through developed commercial and residential areas of the Towns of Cheektowaga and Lancaster. Downstream of the tunnel, the creek passes through Delaware Park and then enters the former Black Rock industrial area. Only the lowermost portion of the creek (lower ½-mile) has been historically navigable for small boats that serviced former industries along the creek. The mouth of the creek was historically used as a small port

and supported naval vessels during the War of 1812 including the rehabilitation of watercraft. Prior to industrial development of the Black Rock area, the creek was wide, shallow, and meandering, surrounded by extensive marshes and meadows. Over the past two centuries, the Scajaquada Creek channel has been altered as urban and industrial development progressed in the City of Buffalo. Boat and barge traffic historically transported materials to and from the various industries located along the Lower Scajaquada Creek during the late 1800s and early 1900s. During that time, the creek was channelized and the wetland areas surrounding the creek were filled to accommodate industrial development. Boat slips lined the northern side of the creek adjacent to the 1660 Niagara Street parcel. The historic outlet of the creek was closed during the mid-1950s by backfilling during the construction of the NYS Thruway I-190 and the Scajaquada Creek Expressway (NY Route 198), when a new outlet to Black Rock Canal was constructed approximately 450 feet upstream. The modified creek outlet location created the more stagnant "Slip" or "Inlet" segment, which in-filled with depositional material over time.

Scajaquada Creek flows toward the Black Rock Channel under normal weather conditions. However, storm and wind events can cause flow in the Lower Scajaquada Creek area to temporarily reverse direction as lake levels at the eastern end of Lake Erie rise producing higher water levels in the Niagara River. More importantly, the surface water elevation in the Lower Scajaquada Creek is strongly influenced by the surface water elevation of the Black Rock Canal which is controlled by a lock at the north end of the canal. Creek elevation monitoring by GEI shows daily surface water elevations changes of one to two feet and often as much as four feet depending on wind speeds and direction. During high intensity wind and storm events, canal and creek level changes can become extreme. During a seiche event on December 23, 2022, a six to seven-foot rise in surface water elevation was recorded by the Black Rock USGS station and GEI pressure transducers monitoring the creek level and near-shore groundwater elevations. The event produced localized flooding on Niagara Street as surface water in the Inlet overtopped its banks on 1660 Niagara Street at an elevation of approximately 577 msl. These lake seiche conditions are temporary, typically lasting less than 48 hours, and occur more frequently during the late fall and early winter seasons in Western New York.

There are no active stream gauges on Scajaquada Creek. Stream flow at the entrance to the upstream tunnel located approximately 8 miles upstream from the mouth was historically recorded by USGS (gauge 04216200) from March 1, 1957, to September 30, 1994. The measured flow rates during this monitoring period ranged between 3 cubic feet per second (ft/s) (1992) and 292 cubic ft/s (1994).

Scajaquada Creek is navigable from the Black Rock Canal into the Slip Area and the downstream side of the CSX railroad bridge. Between the railroad bridge and the West Avenue bridge, multiple low clearance bridges associated with NY Route 198 (Scajaquada Expressway) make the creek passible by only small boats, kayaks, and canoes.

2.5. Wetlands

State-regulated wetlands were not identified in the Lower Scajaquada Creek area. The portion of Scajaquada Creek downstream from the West Avenue Bridge to the mouth, including the Inlet, as well as the Black Rock Canal is identified as a Riverine Wetland environment on US National Wetlands Inventory mapping.

2.6. Description of Fish and Wildlife Resources

Fish and wildlife resources have been identified in the Lower Scajaquada Creek area through desktop study and site reconnaissance. An onsite habitat assessment was conducted on October 23, 2024 in Lower Scajaquada Creek, focusing on the area primarily south of the West Avenue Bridge and the Scajaquada Creek Slip area. The Lower Scajaquada Creek area is characterized as a highly urbanized, degraded aquatic habitat within a heavily developed watershed, primarily consisting of a channelized creek with limited natural vegetation, and bank cover type dominated by staghorn sumac (*Rhus typhina*), tall goldenrod (*Solidago altissima*), box elder (*Acer negundo*) and various dogwood species (*Cornus* sp.). Dense stands of invasive species including Japanese knotweed (*Reynoutria japonica*) were also observed covering large sections of the banks.

Water at the time of the assessment was clear and the creek substrate was easily observable. The substrate of Lower Scajaquada Creek within the assessment area was observed to have limited areas of depositional sediment, although a few areas of sporadic depositional material were observed adjacent to the banks. The majority of the channel bottom, excluding the Inlet, is instead dominated by pebbles, cobbles, and sporadic boulders. Due to the nature of the observed substrate, submerged aquatic vegetation was limited to the shallower depositional areas of sediment along the creek banks thus significantly reducing fish cover. Observed fish species included rock bass (*Ambloplites rupestris*), smallmouth bass (*Micropterus dolomieu*) and striped shiner (*Luxilus chrysocephalus*). The banks of Lower Scajaquada Creek ranged from gently sloping areas dominated with terrestrial herbaceous vegetation and sporadic tree cover to engineered shorelines.

A US Fish and Wildlife Service Information for Planning and Consultation (IPaC) desktop review was conducted on October 21, 2024. The IPaC report was prepared for Lower Scajaquada Creek west of the West Avenue Bridge to its confluence with the Black Rock Canal. The IPaC report identified no known listed Federally threatened or endangered species within the specified reach of Lower Scajaquada Creek. One Federally proposed species, the salamander mussel (*Simpsonaias ambigua*) was identified in the IPaC report.

A review of the New York State Environmental Resource Mapper identified one state-listed threatened species, the lake sturgeon (*Acipenser fulvescens*) as a potential receptor within the reach of Lower Scajaquada Creek west of the West Avenue Bridge to its confluence with the Niagara River. The portion of Lower Scajaquada Creek in the OU is a Class B designated stream.

Further information regarding potential fish and wildlife receptors is discussed in Section 9.

3. Investigation History, Known Extent of DNAPL, And Historic Industrial Properties

Properties mentioned in this section are situated within the Lower Scajaquada Creek OU and are shown on Figure 3.

3.1. Investigation History

The Lower Scajaquada Creek area has been investigated by GEI and others over the past several decades. GEI completed a Site Characterization of the Lower Scajaquada Creek area and documented the findings in the Site Characterization Report (GEI, October 2022) which supports the basis for additional investigation to fill data gaps.

The 2022 Site Characterization (SC) Report, including text, figures, tables, and boring logs, is provided in Appendix A. The objective of the site characterization was to identify the source area(s) of coal-tar-related DNAPL in the Lower Scajaquada Creek. The NYSDEC suspected that the area of a former MGP gas holder, located on property owned by the New York State Thruway Authority near the current creek outlet (referred to as the P-Site), was a source of coal-tar-related DNAPL. However, the investigations did not identify this property as a DNAPL source area. Investigations did identified coal tar-related DNAPL contamination in deeper soils above the area wide clay layer adjacent to the creek near the Iroquois Gas/Westwood Pharmaceutical Riparian site (NYSDEC well on the bike path), in the subsurface soil below properties adjacent to the creek and below the creek between the West Avenue Bridge and the Inlet Area, and in shallow creek sediments near the discharge from a City of Buffalo Pumphouse. This pumphouse operates to remove groundwater and stormwater from Niagara Street below the CSX railroad bridge, including the storm sewer system and pumphouse wet well.

Additionally, dissolved-phase chemical constituents were detected in the groundwater, and commingled contamination, unrelated to coal tar, was also identified. The SC Report incorporated information from other investigations, detailed in publicly available reports for several nearby BCP Sites. These findings necessitated the development of this RI/FS Work Plan.

Key findings of prior investigations are summarized below.

3.2. DNAPL Observations and Extent

DNAPL, related to coal tar, was identified in coarser-grain alluvial soil and sediment samples, and in eight monitoring wells near the creek installed during NYSDEC site investigations and three wells installed during the GEI 2021/2022 Site Characterization. The DNAPL with a coal tar-like odor was found in deeper coarser-grain alluvial soils above the area-wide red-brown clay layer which is a barrier to downward migration of DNAPL. The area of borings where DNAPL was encountered is shown on Figure 4. Some existing wells, particularly those installed during earlier BCP site investigations, were not deep enough to intersect the top of the red-brown clay layer and are of limited use in establishing the extent of DNAPL presence.

DNAPL has been primarily observed in the deeper alluvial soils above the area-wide clay layer. A forensic evaluation of MAH (Monoaromatic Hydrocarbons) and PAH (Polycyclic Aromatic Hydrocarbons) chemical patterns in soil and sediment samples containing DNAPL identified similarities to those from historic gas manufacturing at the Iroquois Gas/Westwood Pharmaceutical sites (see Appendix A).

DNAPL was identified in three soil borings/wells located near the creek and in four shallow sediment borings downstream of the Niagara Street bridge. DNAPL was present in two sediment borings completed to the top of clay in the creek between West Avenue and Niagara Street. DNAPL was also observed in two Niagara Street manholes that convey stormwater/groundwater to the City of Buffalo Pumphouse and in a wet well (vault) in the Pumphouse where DNAPL accumulated in sediment and debris within the concrete vault. The DNAPL impacts identified in land-based borings/wells were limited to the deeper alluvial soil above the red-brown clay. The geometry of the clay surface and hydraulic gradients influence the distribution of DNAPL in the Lower Scajaquada Creek area. Figure 4 shows sediment and soil borings where DNAPL was observed in the Lower Scajaquada Creek area.

The Site Characterization also included a preliminary DNAPL mobility assessment, concluding that DNAPL migration in the Lower Scajaquada Creek area is primarily driven by hydraulic gradients, gravity, and the surface elevations of the area-wide red-brown clay layer (clay surface geometry). Figure 4 shows the contoured surface of this clay layer, which was historically scoured by the past flow of Scajaquada Creek.

The contoured surface of the clay layer plays a significant role in the location of DNAPL. Because DNAPL is heavier than water, it tends to sink until encountering a confining layer and then migrates via gravity-driven forces along the top of the confining layer. Therefore, the historic flow path of Scajaquada Creek, which scoured the surface of the area-wide clay layer, serves as a sink for DNAPL because it limits further downward migration and limits lateral movement away from the deepest portions of the historic creek channel.

In more recent times (post-1850s industrial development of the lower creek), the water's edge was much wider farther upstream as shown on the figure below.

Figure Above: *Recent aerial photography overlain on an 1856 map of the Black Rock Area to illustrate historic extents of Scajaquada Creek relative to its current channel (source: Georeferenced map provided by NYSDEC, 2024). The star identifies the upstream extent of the Lower Scajaquada Creek OU at the West Avenue bridge.*

Based on known geology, the limits of DNAPL presence would not be expected to extend much beyond the aerial geometry of the creek.

In the Lower Scajaquada Creek area, the DNAPL accumulates on top of the area-wide clay layer. However, due to its specific gravity being only marginally above that of water, DNAPL can become buoyant where vertical hydraulic gradients are sufficiently upward. This was evidenced by the finding of DNAPL in the infrastructure associated with the City of Buffalo Pumphouse. Near the City of Buffalo Pumping Station, groundwater underdrains and deeper storm sewers artificially depress the water table, creating an upward hydraulic gradient. This gradient can potentially cause DNAPL to migrate upward into storm sewers, particularly where the integrity of sewer manholes is compromised. The Site Investigation identified DNAPL in two Niagara Street storm sewer manholes and in the connected City of Buffalo Pumphouse wet well, which discharges into Scajaquada Creek.

DNAPL was removed from the manholes and wet well, and repairs were made to the manholes and piping during the IRMs completed in 2022 (manhole repairs) and 2024 (pipe sleeving) to eliminate this migration pathway. Additional details regarding these interim remedial measures are provided in Section 4.

Groundwater Contamination with DNAPL Presence:

Benzene, toluene, ethylbenzene, and xylenes (BTEX) and polycyclic aromatic hydrocarbon (PAH) compounds were detected above New York State's Ambient Water Quality Standards (AWQSs) in groundwater samples near the creek where DNAPL was present. PCBs and metals were either not detected or elevated above AWQS in groundwater. At several well locations, groundwater also contained chlorinated VOCs, 1,4-dioxane, and per- and polyfluoroalkyl substances (PFAS) above screening criteria. A stormwater/groundwater discharge sample collected from the City of Buffalo Pumphouse during the Pumphouse PDI/IRM (described in Section 4) also contained concentrations of BTEX compounds, PAHs, chlorinated VOCs and 1,4-dioxane above AWQS.

Sediment Contamination with DNAPL Presence:

DNAPL was identified in shallow sediments near the discharge location of the City of Buffalo Pumphouse in the Inlet. DNAPL was not identified in shallow sediments between the West Avenue Bridge and the current creek outlet. The PAH concentrations were highest in shallow sediment borings nearest the discharge point from the City of Buffalo Pumphouse, with one sample (SED-LSC-3 (0-0.5')) showing nearly 500 times the levels of other samples. Elevated levels of volatile and extractable petroleum hydrocarbons, PCBs, and metals (mercury, copper, lead, nickel, zinc) were detected at this location. Where sheens on Inlet surface water were observed prior to the Pumphouse IRM, chemical constituents associated with the sheen were deposited on surface soil along the creek bank along the water line.

The GEI 2021/2022 Site Characterization described the presence of DNAPL downstream of the West Avenue bridge as a result of DNAPL migration from historic MGP operations. These operations began in the late 1800s and continued until approximately 2000, when engineering controls were installed to mitigate migration from the source area (Iroquois Gas/Westwood Pharmaceuticals Terrestrial/Riparian Site). The RI scope of work (Section 6) will assess the effectiveness of source controls installed and operated from this site (see Section 3.3 for remedial actions).

3.3. Historic Industrial Properties in the Scajaquada Creek OU

Several historic industrial properties lie within the boundary of the Lower Scajaquada Creek OU (Figure 3). Both National Fuel and NYSDEC will be conducting RI work to determine if there are existing sources of contamination that are impacting sediment quality.

As described in Section 3.2 and Section 6, National Fuel will be assessing the adequacy of upstream engineering controls to contain coal tar-related DNAPL at the Iroquois Gas/Westwood Pharmaceuticals Site. That property was used for manufacturing gas from a carbureted water gas process using coke, oil, and coal feedstocks from about 1897 until the mid-1950s. A Remedial Investigation/Feasibility Study (RI/FS) was conducted in the mid-1990s by GeoTrans to determine the nature and extent of impacts. In a NYSDEC Consent Order, the site was divided into two portions: the Terrestrial Site (Site #915141) where gas manufacturing occurred, and the Riparian Site (Site #915141B) along the creek. The Terrestrial Site Remedy included:

- Capping of impacted soil
- In-situ biotreatment of soils
- On-site hydraulic control of NAPL and groundwater using vertical pumping wells and treatment
- Placement of a sheet pile barrier along the creek bank
- Site operation and monitoring

In 2017, vertical wells were replaced by a horizontal groundwater collection trench upgradient of the sheet pile barrier to improve hydraulic control.

The Riparian Site Remedy included:

- Excavation and removal of contaminated sediments from historic operations at the Terrestrial Site
- Capping of the creek bottom from approximately 50 feet east of the West Avenue Bridge to 1,600 feet upstream to the abandoned New York Central Railroad bridge

- Placement of a vertical sheet pile barrier near the West Avenue Bridge Long-term collection of DNAPL from coarse-grained deposits below the cap and creek sediments upgradient from the vertical barrier
- Long-term monitoring of the sediment cap

The Riparian remedy also included the installation of a subsurface sheet pile barrier perpendicular to the creek alignment upstream of the West Avenue Bridge, a sediment cap, and two angled collection wells into deeper creek sediments to collect mobile DNAPL (RETEC Engineering, P.C., August 2000).

While National Fuel's work will focus primarily assessing the extent of DNAPL and potential migration pathways, samples collected during the RI will also include a suite of analytes to assess COCs previously observed on adjacent properties and in sediment.

NYSDEC will be assessing potential source areas from other historic industrial properties namely the 1660 Niagara Street BCP site and property on the north side of Niagara Street near Tonawanda Street. Properties with known CVOC contamination are described below.

1660 Niagara Street: Historical use of this property since the 1940s included gasoline sales, underground petroleum storage, vehicle repair and collision shop, and boat sales. Soil and groundwater testing during a Phase II environmental site assessment (ESA) identified contamination from historic property use and detected the following Constituents of Concern (COCs): petroleum VOCs and chlorinated VOCs (i.e., tetrachloroethylene [PCE] and degradation products), PAHs, arsenic, and lead. Groundwater samples analyzed from monitoring wells located at the southern end of the property reported concentrations of chlorinated VOCs approaching 1% of the solubility limit of their parent compounds (tetrachloroethene and trichloroethene) indicating a potential source of non-coal-tar-related DNAPL.

31 Tonawanda Street: The Fedders Corporation constructed a large manufacturing facility on the property and historically used various chemicals, oils, solvents, and other materials in their manufacturing process. The processes at the property included metal stamping, soldering, brazing, welding, painting, acid washing and degreasing. Industrial waste was reported to include solder dross, degreasing still bottoms including TCE and PCE, petroleum-based lubricating fluids and other products and wastes. Percent level concentrations of chlorinated VOCs (PCE and breakdown products) were identified in groundwater at the 31 Tonawanda Street property and property west of the site at 1660 Niagara Street near the CSX railroad tracks.

Other historic industrial properties shown on Figure 3 that contained contamination but not considered an ongoing source include:

- 1675 Niagara Street
- 1626 Niagara Street
- 57-71 Tonawanda Street
- 68 Tonawanda Street
- 75 Tonawanda Street
- 189-205 Tonawanda Street
- 1451 West Avenue
- 166 Dart Street

Each of these sites are described in Appendix A.

4. City of Buffalo Pumphouse IRMs

In December 2021, the NYSDEC requested that National Fuel investigate and implement IRMs to address a petroleum-like sheen on Scajaquada Creek during discharge cycles of the City of Buffalo Pumphouse. Sheens had been observed in 2014 and 2019, and during site investigations by GEI in 2021 and 2022. Inspections identified DNAPL and contaminants including BTEX, CVOCs, 1,4-dioxane, and PAHs in the storm sewer system and Pumphouse wet well. National Fuel agreed to develop a scope of work to investigate tar entry mechanisms and implement IRMs collaboratively with NYSDEC. Emergency response actions included replacing booms, piezometer installations and groundwater sampling, sealing stormwater infrastructure, inspecting subsurface infrastructure, and managing contaminated media.

A pre-design investigation (PDI) performed by GEI identified several critical findings and recommendations for the design and construction IRM. Consistent with the Site Characterization Report (October 2022), it was observed that groundwater elevations are notably depressed in the area surrounding Niagara Street and the storm sewer piping leading to the pumphouse. Additionally, vertical hydraulic gradients in the water-bearing zone above the area-wide, red-brown clay layer near the Pumphouse and storm sewer were found to be upward. The investigation identified that the manhole bottoms within the stormwater conveyance system were poorly constructed or deteriorated, allowing contaminated groundwater and DNAPL to enter. Seepage of grossly contaminated materials was particularly noted in areas with these construction defects. Furthermore, it was determined that contaminated groundwater with elevated concentrations of chlorinated solvents from nearby sites also contributes to the contamination of the storm sewer system.

To effectively mitigate the sheen observed on Scajaquada Creek, it was essential to eliminate the entry of grossly contaminated materials into the storm sewer system and minimize the disturbance of near-shore sediment in the creek during discharge cycles. Epoxy and grout sealants were used to minimize the entry of contaminated groundwater and DNAPL into the manholes below Niagara Street and the Pumphouse wet well also cleaned and sealed. Water quality testing of the manholes and Pumphouse discharge identified seepage of contaminated groundwater (primarily CVOCs and BTEX) through a short section of the conveyance piping from manholes between the wet well and Niagara Street. Slip lining of certain stormwater piping was performed by a NYSDEC contractor to mitigate the infiltration of groundwater along this segment. The NYSDEC also installed a harbor boom across the southern end of Inlet to contain sheens from entering the main channel of Scajaquada Creek.

On behalf of National Fuel, GEI developed and implemented an IRM to address the sheen on Scajaquada Creek. This sheen occurrence was determined to be largely caused by the disturbance of heavily contaminated shallow creek sediments during Pumphouse discharges. The Pumphouse discharges at a rate of approximately 2,250 gallons per minute, causing re-suspension of contaminated sediments and hydrocarbon-like sheen on the surface water near the point of discharge. The selected IRM approach involved constructing a permeable wall of gabion baskets around the outfall, supported by a concrete slab on helical piles, and additional rip-rap armoring and a sheen mitigation barrier to dissipate flow velocity and protect the structure. IRM construction was completed on January 17, 2023. Details of investigations and IRMs completed at the City of Buffalo Pumphouse are documented in a report entitled "*Design Investigation and Interim Remedial Measures (IRM) Report - Lower Scajaquada Creek City of*

Buffalo Pumphouse - NYSDEC Spill #1406307" (GEI, March 2023). The figure below is from the GEI IRM Report and shows the layout of the storm sewer system below Niagara Street, manhole locations and IDs, the Pumphouse, and gabion wall constructed to dissipate turbulent flow from Pumphouse discharge cycles to minimize disturbance to near-shore shallow creek sediments.

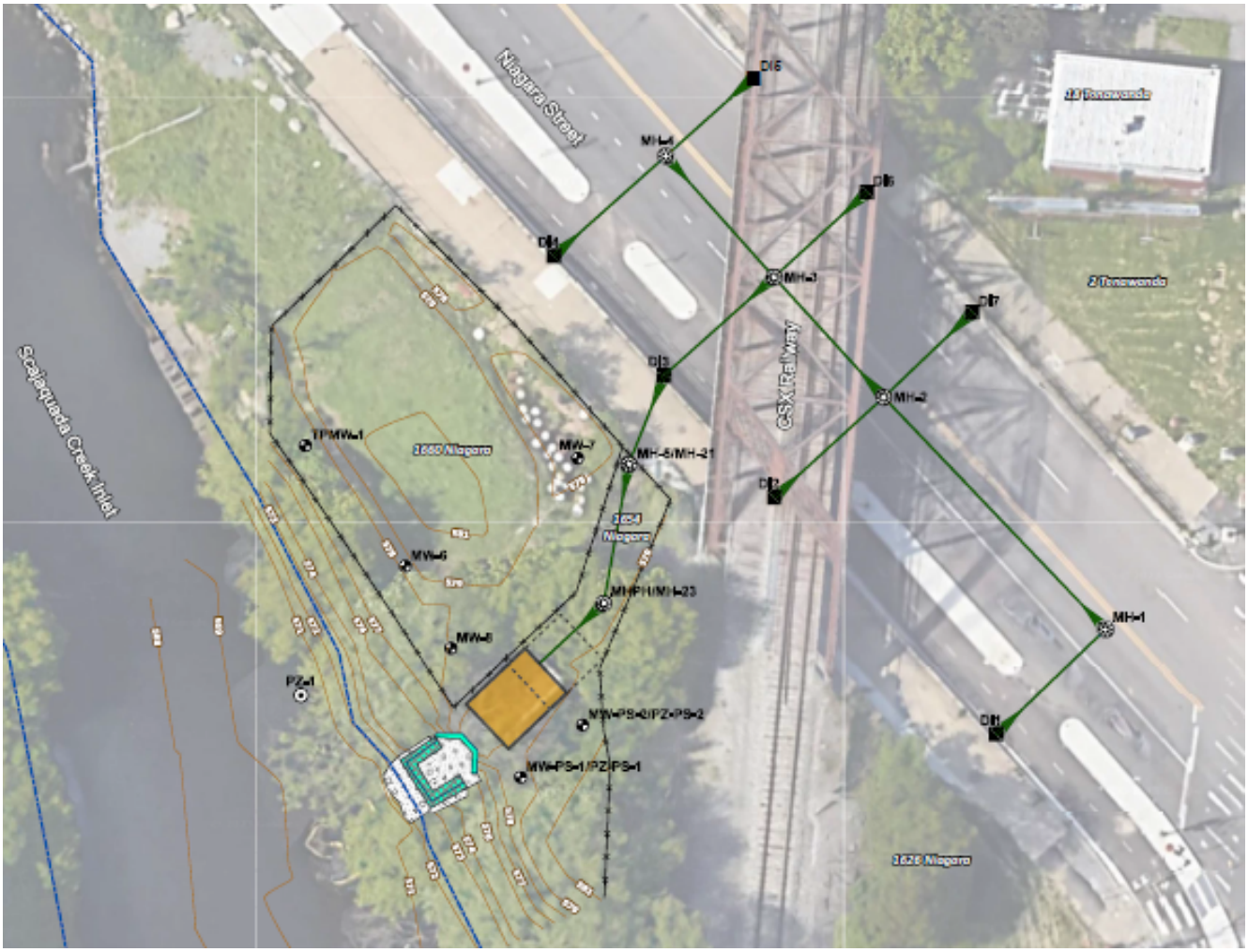


***Figure above**: an excerpt from the IRM Report (GEI, 2023).*

5. Preliminary Conceptual Site Model and Data Gaps

5.1. Preliminary Conceptual Site Model

The 2021/2022 Site Characterization (SC) of the Lower Scajaquada Creek area identified contamination in soils, groundwater, creek sediments, and City of Buffalo infrastructure. This contamination is linked to historic DNAPL transport from former MGP operations. Additionally, investigations by other parties and the NYSDEC at BCP sites revealed contamination of soil, groundwater, and sediments due to past industrial property use, described by the NYSDEC as commingled contamination.

Information from these investigations was used to develop a preliminary CSM for the Lower Scajaquada Creek OU. The CSM outlines potential sources of contamination, release mechanisms, exposure media, and exposure routes based on current and potential future land use. The CSM will be refined based on data collected in the RI.

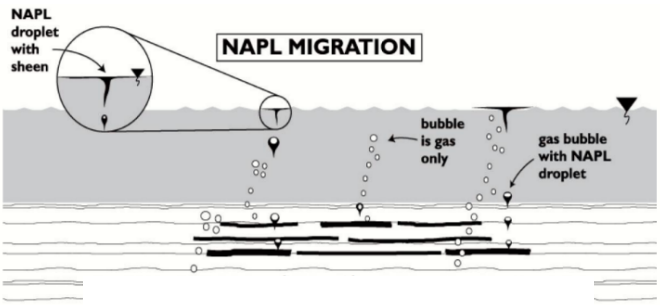
Previous investigations identified various chemical constituents in lower alluvial soil, groundwater, sediment, and City of Buffalo Pumphouse discharge in the Lower Scajaquada Creek OU. These environmental media could potentially serve as exposure media. The CSM provides a qualitative assessment of potential exposure via each medium for various types of receptors.

Data and findings from prior investigations indicate that sources of contamination affecting the Lower Scajaquada Creek originate from the former MGP operation, historic industrial use of nearby properties, and discharge to the creek from CSOs and the City of Buffalo Pumphouse. Contributions from upstream point and non-point sources are also likely given the highly urbanized status of the watershed.

Contamination release mechanisms refer to the processes by which contamination could be transferred from sources to environmental media. Contamination release mechanisms considered as part of the CSM are described below.

- Volatilization: VOCs from NAPL, contaminated soils, and/or groundwater that are released into air, including soil gas (i.e., the air entrained between soil particles in the vadose zone).
- DNAPL Migration: Gravity driven transport of DNAPL occurred as a result of product with specific gravity greater than 1.0 migrating vertically downward until encountering a low-permeability zone (i.e., clay) that precluded further vertical migration. When sufficient source existed, the DNAPL also spread laterally along the top of the clay confining layer, affecting a zone of coarser soil above the clay confining layer.
- Solubilization: Groundwater quality is affected through solubilization of contaminants from pooled or residual DNAPL. Once solubilized into groundwater, constituents are also transported with groundwater flow and may discharge into surface water.
- Discharges to the Creek: Surface water and groundwater collected in the City of Buffalo Pumphouse wet well was discharged to surface water as part of normal pumphouse operations (Pumphouse IRMs mitigated DNAPL entry into the system).

- Ebullition transport: NAPL migration from shallow DNAPL in creek sediments near the Pumphouse to the creek surface driven by gas bubbles caused by changes in hydraulic pressures (e.g., as created by Black Rock Canal lock operation). This contaminant release mechanism produces observed sheen on the surface water in the creek. Ebullition is conceptually shown graphically in the adjacent schematic.



Source: USEPA, 2015

Potential exposure describes the mechanism (exposure route) by which human receptors can be exposed to contamination during current or potentially foreseeable uses of the Lower Scajaquada Creek area. Such receptors include commercial workers, utility workers, construction workers, and passive recreational users of the creek and land adjacent to the waterway. Direct contact, ingestion, inhalation, and vapor intrusion/exposure are all exposure routes that could potentially impact receptors, depending on the nature of their specific activities and exposure areas.

Primary and secondary contact recreation and fishing exposure (i.e., consumption of fish caught from the creek) are pathways that could potentially impact human receptors. DNAPL-impacted soils are typically deeper than 15 feet below the ground surface, so human exposure to DNAPL in soil would be limited to construction workers involved in deep excavation work such as major utility infrastructure development or building construction with deep foundations. Under these scenarios, human exposure routes for chemicals include:

- inhalation of VOCs volatized from groundwater (particularly CVOCs in the area of Niagara Street);
- dermal contact, inhalation, and incidental ingestion of soil/sediment particulates; and
- dermal contact and incidental ingestion of groundwater/surface water.

Groundwater in the area is not a source of drinking water. Scajaquada Creek surface water is also not a drinking water source.

Ecological exposure mechanisms in the Lower Scajaquada Creek are direct exposure to impacted sediments and pore water in the observed depositional areas and assimilation by a species (e.g., prey/food item) at one trophic level and transferred to a higher trophic level species through consumption. For certain types of chemicals, bioaccumulation can exacerbate this pathway at certain levels in the food chain. Based on the evaluation of exposure pathways, the key receptors of concern are benthic invertebrates, fish and semi-aquatic piscivorous mammals and avian species.

The key findings from the Site Characterization are summarized below and establish the preliminary CSM for the investigation of the Lower Scajaquada Creek OU.

5.1.1. Soil Contamination

Soil Quality and DNAPL Presence: Pesticides, PCBs, herbicides, and cyanide were not detected in Site Characterization soil samples above NYSDEC's soil guidance values, regardless of the presence of DNAPL

impacts. However, VOCs and SVOCs (primarily PAHs) were found in soils affected by DNAPL. Certain metals, including arsenic, copper, lead, mercury, nickel, and zinc, were detected in some soil samples above soil guidance values. The presence of chemical impacts associated with DNAPL contamination did not correlate with elevated metal concentrations. Grossly contaminated soil and DNAPL were primarily found in deeper alluvial soils directly above the red-brown clay layer. DNAPL accumulation was observed in the sumps of several monitoring wells near Scajaquada Creek, with well screens monitoring the bottom of the alluvial soils above the clay layer.

The uniformity, thickness, and low permeability of the area-wide red-brown clay layer indicate that this layer acts as a confining layer, preventing the downward vertical migration of DNAPL. DNAPL impacts extending into the red-brown clay layer were not identified during the Site Characterization. The extent to which the clay layer serves as a competent and contiguous vertical migration barrier will be further evaluated as part of the RI.

Source Analysis: Forensic analysis of DNAPL and grossly contaminated soil samples indicate a water gas tar source of pyrogenic origin. DNAPL impacts are confined to deeper alluvial soil due to the surface geometry of the red-brown clay and hydraulic gradients. DNAPL was not identified in shallow soil, suggesting lateral migration of DNAPL from an upgradient source on top of the red-brown clay layer. The DNAPL source is presumed to be historic migration from the Iroquois Gas/Westwood Pharmaceutical Terrestrial/Riparian Site, prior to the installation of engineering controls that contain and remove DNAPL from areas upstream of the West Avenue Bridge.

Chemical fingerprinting of the DNAPL found in borings and wells downstream from the Terrestrial/ Riparian Site engineering controls is similar to that recovered at the Iroquois Gas/Westwood Pharmaceutical Sites. DNAPL has not been detected in shallower soils or sediments near the West Avenue Bridge, suggesting no ongoing releases are occurring. However, this will be further investigated as part of the RI.

There is a possibility of CVOC DNAPL associated with historic industrial usage at BCP sites located at 31 Tonawanda St. and 1660 Niagara St. This is based on dissolved phase concentrations in groundwater of several chlorinated volatile organic compounds approaching 1% of their individual solubility limits. NYSDEC is investigating CVOC contamination in Lower Scajaquada Creek OU.

5.1.2. Groundwater Contamination

MGP Constituent Detection: Site Characterization groundwater samples collected from the Lower Scajaquada Creek area contained BTEX and PAH compounds of pyrogenic origin above New York State ambient water quality standards (AWQS). These constituents were detected in wells with wells screens intercepting the area-wide red-brown clay and where DNAPL was identified to accumulate in the bottom of the well.

Additional Contaminants: Petroleum-related (petrogenic) VOCs, CVOCs, 1,4-dioxane, and PFAS were also detected above AWQS in some wells located on and near BCP properties and near the Inlet. Where present with pyrogenic VOCs and PAHs, their presence is identified as comingled contamination. CVOCs and 1,4-dioxane were also present in the City of Buffalo Pumphouse discharge water at concentrations

above AWQS. As described in Section 4, an IRM was completed in 2022 to mitigate entry of DNAPL and comingled contamination into the manholes and conveyance piping.

Pesticides and herbicides were not detected in groundwater samples analyzed during the Site Characterization. RCRA metals were not detected above AWQS in most wells and none containing DNAPL.

5.1.3. Sediment Contamination

Shallow Sediments: DNAPL impacted recent fluvial deposits were observed in shallow sediments (0-6" depth) in the creek Inlet near the City of Buffalo Pumphouse discharge outlet. PAH concentrations in some shallow sediment samples were elevated, with the sample collected directly adjacent to the Pumphouse discharge outlet (SED-LSC-3; total PAH-16 concentration of 5,649 mg/kg). DNAPL was not identified in shallow sediments elsewhere in the creek during the Site Characterization.

Contaminant Levels: Shallow sediments exhibited elevated levels of volatile and extractable petroleum hydrocarbons, PCBs, and metals (copper, lead, nickel, zinc) when compared to sediment guidance values. Deeper sediments also showed significant contamination, particularly in the creek Inlet area, with high concentrations of BTEX, PAHs, PCBs, and metals (copper, lead nickel, mercury, zinc). DNAPL or sheens/ staining associated with suspected water gas tar residual was identified in deeper alluvial-derived soils below the recent fluvial deposits above the area-wide clay in five of nine sediment borings.

5.2. Data Gaps

Additional data are required to assess the nature and extent of impacts more fully in soil, groundwater, and sediments to evaluate remedial alternatives for the Lower Scajaquada Creek OU. Data gaps with RI objectives are summarized in the following subsections.

5.2.1. Adequacy of DNAPL Source Control

Data Gap: The adequacy of engineering controls upstream of the West Avenue Bridge to contain mobile DNAPL requires demonstration to support the assumption of source control.

RI Objective: Assess the presence and mobility of DNAPL in the vicinity of the West Avenue Bridge to evaluate the effectiveness of upstream physical barriers and DNAPL containment and/or collection and removal systems in preventing DNAPL transport to the south. While residual DNAPL is expected, it will be important to assess mobility and its potential to be a continuing source.

Data Gap: The longer-term performance of Pumphouse IRMs has not been monitored.

RI Objective: Inspect IRM work completed to seal the manholes and conveyance piping for DNAPL entry and groundwater infiltration, as feasible. Water quality will be evaluated by collecting samples from the stormwater conveyance manholes and water discharged at the Pumphouse outfall.

5.2.2. Extent of DNAPL in Soil

Data Gap: The horizontal limits of DNAPL in the subsurface have not been fully characterized below the creek sediments and adjacent upland area above the area wide red-brown clay layer. Construction of Expressway piers may have acted as a temporary migration pathway for vertical transport below the area-wide clay layer.

RI Objective: Establish the horizontal and vertical extent of DNAPL in the OU in consideration of potential migration pathways in natural geologic materials and manmade pathways associated with buried urban infrastructure. This would include evaluating the lateral extents adjacent to the creek, the northern extent of DNAPL in soils beyond the 1660 Niagara Street BCP site, and the soil located near the former outlet of Scajaquada Creek to the Black Rock Canal below the I-190 Thruway. Additional characterization of the surface of the area-wide red-brown clay layer in the lower Scajaquada Creek Area is important as it plays a major role in the fate and transport of DNAPL. Investigation of the sand unit below the area-wide clay layer is warranted.

5.2.3. Identification of Utilities and Infrastructure as DNAPL Migration Pathways

Data Gap: The lower Scajaquada Creek area contains many underground utilities and it is not known whether any of these, other than the Niagara Street storm sewer and City of Buffalo Pumphouse, represent potential preferential pathways for migration away from the creek.

RI Objective: Identify subsurface utilities that occur at or below depths where DNAPL has been identified and assess their potential as a pathway for migration.

5.2.4. Extent of DNAPL in Shallow Creek Sediments

Data Gap: The extent of DNAPL presence in shallow sediments in the Inlet area of the Creek near the Pumphouse discharge outfall.

RI Objective: Use probing and borings to map the distribution DNAPL presence in shallow sediments. While DNAPL presence in shallow sediments was primarily observed in the creek Inlet, a comprehensive probing survey of the shallow creek sediments is needed for verification between the West Avenue Bridge and the Black Rock Channel, including other areas of the Inlet.

Data Gap: Ebullition transport and re-suspension of shallow sediments containing DNAPL near the Pumphouse outfall were observed, producing a sheen that was deposited on creek bank soils. This phenomenon was identified as a migration pathway. The extent of this impact requires further characterization.

RI Objective: Characterize the composition of shallow sediments, including total organic carbon content and the lateral and vertical extents of DNAPL. Additionally, assess the hydraulic influences from daily water level fluctuations in the Black Rock Canal and the impact on surface soils along the creek bank in the Inlet area.

5.2.5. Sediment Quality and Ecological Risks

Data Gap: The quality of creek sediment, the risk posed to benthic organisms, fish, and wildlife, and the data needed to evaluate FS remedial alternatives is required to develop a remedy that could allow removal of Beneficial Use Impairments (BUIs) in the creek.

RI Objective: Assess sediment quality in the Lower Scajaquada Creek and Inlet with a detailed emphasis on known COCs including volatile and extractable petroleum hydrocarbons (EPH), PAHs, PCBs, and metals (mercury, copper, lead, nickel, zinc) to evaluate the risks to benthos, fish, and wildlife. Identify the information needed to evaluate remedial alternatives aimed at removing BUIs in the creek.

5.2.6. Shallow Groundwater Quality

Data Gap: The impact of subsurface DNAPL on shallow groundwater quality.

RI Objective: Wells installed during the 2021/2022 SC were equipped with well screens extending through the deeper alluvial soils into the top of the clay layer. Assess whether the presence of deeper DNAPL affects the groundwater quality near the water table, which is significantly shallower than the area-wide clay layer.

5.2.7. Information to Support the FS

Data Gap: Existing data requires supplementation from the filling of RI data gaps described above. Structural and hydraulic limitations exist that could affect the decision-making process to address contaminated soils and sediments. To inform the development, evaluation, and selection of remedial actions for the Lower Scajaquada Creek OU, each of the data gaps identified above needs to be addressed. In addition, other site-specific data collection and observations will be performed as part of the RI to support the FS and subsequent remedial design phases.

RI Objective: Data should be collected in and near the Inlet to evaluate the eastern wall adjacent to 1660 Niagara Street, the sheet pile wall adjacent to the I-190 Thruway, and piers adjacent to the creek. Data will also be collected to characterize potential treatment and disposal options for media that may be considered for soil/sediment removal under FS alternatives.

5.2.8. CVOCs in Soil and Groundwater

Data Gap: Chlorinated Volatile Organic Compounds (CVOCs) were identified in the upland areas adjacent to the Lower Scajaquada Creek during Brownfield Cleanup Program (BCP) site investigations and the 2021/2022 Site Characterization (SC). These CVOCs are not associated with coal tar-derived DNAPL contamination. The source and extent of CVOCs in the media of Lower Scajaquada Creek remain incompletely understood.

RI Objective: The NYSDEC is leading a Site Characterization on property located at 1660 Niagara Steet including properties located on the northern side of Niagara Street to delineate the nature and extent of CVOCs in soil and groundwater and to determine whether CVOC contamination is a source of sediment contamination in Lower Scajaquada Creek OU. Their findings will be incorporated into the RI/FS.

6. Remedial Investigation Work Scope

The RI will be conducted consistent with EPA and NYSDEC DER-10 guidance and the following supporting project plans:

- Field Sampling (FSP) - provides field sampling methods and procedures to be used during the investigation (Appendix B).
- Quality Assurance Project Plan (QAPP) specifies data quality objectives, and criteria, data acquisition, data management and data submission requirements, laboratory analytical procedures, quality control measures, data validation, and assessment and oversight details (Appendix C).
- Site-Specific Health and Safety Plan (HASP) - Outline procedures that will be undertaken to protect site workers, visitors, and the public in the areas adjacent to the site from potential hazards that may exist as a result of the field work performed during the RI (Appendix D)
- The HASP also includes a Community Air Monitoring Plan (CAMP) to describe methods to monitor and, if necessary, control the potential release of airborne constituents at the downwind perimeters of the investigation work areas. Additionally, an odor control plan is included with the CAMP if intrusive activities produce noticeable odors outside the work area.

As discussed in previous sections, DNAPL associated with former MGP operations is present in the subsurface and locally in shallow sediments. Although significant data have been collected, data gaps identified in Section 5 need to be addressed. The scope of the RI is designed to fill the identified data gaps and support the preparation of the Feasibility Study to evaluate remedial alternatives. The RI scope includes:

- Source Control Assessment
- Subsurface Soil Investigation
- Groundwater Investigation
- Creek Sediment Investigation
- DNAPL Mobility Assessment
- Evaluation of DNAPL Migration Pathways Associated with Infrastructure
- Groundwater/Surface Water Elevation Monitoring

Prior to initiating field work, administrative functions such as obtaining property access agreements and applying for required environmental permits will be necessary. These will be completed prior to initiating drilling and other field work.

Environmental samples collected during the RI will be analyzed by Pace Analytical Laboratory, an ELAP-approved laboratory certified by New York to perform the specified analyses. The laboratory will provide a New York Analytical Services Protocols (ASP) - Level IV laboratory deliverable package for the applicable results. The data will be reviewed by a data validator for conformance to laboratory contract

requirements and provided to General Dynamics Information Technology, Inc. (GDIT) for data validation as described in the QAPP (Appendix C). Data will be provided in electronic data deliverables (EDDs) in SEDD2 format and EQuIS format that are compatible with EPA and NYSDEC requirements.

6.1. Source Control Assessment

The RI will assess the performance of Engineered Source Controls (ESCs) to verify the effectiveness of the Iroquois Gas/Westwood Pharmaceutical Terrestrial/Riparian Site Remedies (described in Section 3) in containing DNAPL in the upstream adjacent area of the Lower Scajaquada Creek OU. This evaluation will include a review of DNAPL collection records from the Southern DNAPL collection system at the Riparian Site, subsurface soil explorations, and assessments of DNAPL mobility where identified. DNAPL is expected to be present on the downgradient/stream side of the engineering controls. This expectation arises because the ESCs were installed during the late 1990s/early 2000s, whereas the release and migration of DNAPL likely began soon after the MGP startup in 1898 and continued until the mid to late 1990s, when the Iroquois Gas/Westwood Pharmaceutical Terrestrial and Riparian remedies were implemented. Therefore, subsurface soil investigations will be completed on land (Sections 6.2 and 6.3) and in the creek and Inlet (Section 6.4) to assess the lateral and vertical extent of DNAPL impacted soils. Borings and wells to be installed to investigate ESCs are summarized in Table 1 and are shown on Figure 7.

In addition to the source control assessment of ESCs upstream of the Scajaquada Creek OU, the performance of interim remedial measures completed at the City of Pumphouse adjacent to the Inlet creek area described in Section 4 will be evaluated. The scope is described in Section 6.6 with the assessment of infrastructure in the Scajaquada Creek OU.

6.2. Subsurface Soil Investigation

Soil borings will be completed at locations shown in Figures 5 through 7 and summarized in Table 1 to evaluate the lateral and vertical extent of DNAPL presence in the subsurface soil. A total of 28 borings are identified as either Primary or Contingency borings. Primary borings will be completed to refine the extent of known DNAPL presence and address other objectives identified in Table 1. If DNAPL is identified at the primary boring location, a contingency boring will be completed at a “stepped out location” to satisfy the objective of mapping the extent of DNAPL. At select locations, soil borings will be completed as groundwater monitoring wells to evaluate groundwater quality and for the assessment of DNAPL mobility. The NYSDEC oversight team will be consulted during the investigation. The borings and monitoring wells to be completed are described below.

6.2.1. Primary and Contingency Soil Borings

Including primary borings to be converted to monitoring wells, 22 primary borings (inclusive of 2 creek borings T26A and T26B to evaluate Source Control) will be completed during the investigation. As shown on Figures 5 through 7, groundwater monitoring wells will be installed in seven of the soil borings. Monitoring well installation methods are summarized in Section 6.3 and described in detail in the FSP (Appendix B). As many as seven Contingency soil borings may be added at step-out locations if needed to satisfy investigation objectives in the Lower Scajaquada Creek OU. If DNAPL is identified in a

Contingency boring, another boring will be completed at a step-out location with agreement from the NYSDEC. Boring termination depths for Primary and Secondary soil borings are summarized in Table 1.

At each location, the boring will be advanced by either a direct-push (Geoprobe™) drilling rig equipped with 2-inch diameter Macro-Core™ samplers, or by a conventional hollow-stem auger (HSA) drill rig equipped with 2-inch diameter split-spoon samplers as described in the FSP (Appendix B). Each boring will be completed through fill and native soil and then a minimum of four feet into the red-brown clay unit. If DNAPL is observed within the top four feet of the native red-brown clay unit, the soil boring will be advanced until no visual evidence of impact is observed. At soil boring locations SB-RI-9 and SB-RI-11, the boring will be advanced through the native red-brown clay unit into the underlying sand unit which directly overlies bedrock. These two borings are anticipated to be drilled to a depth of approximately 70 feet below ground surface. The objective of the deeper soil borings is to evaluate the potential for DNAPL impacts below the native red-brown clay unit. Soil drilling and logging methods including screening (with a PID) are described in the FSP (Appendix B).

6.2.2. Soil Boring Sampling and Analytical

A minimum of two soil samples will be collected from each boring and submitted for laboratory analysis. A sample will be collected from the most apparently impacted interval based on PID screening and field observations. If impacts are not observed, a sample will be collected from the 1-foot interval immediately below the water table, and a second sample will be collected from the bottom of the boring presumed to be from the upper red-brown clay unit. At deeper soil boring locations SB-RI-9 and SB-RI-11, an additional soil sample will be collected from the sand unit directly underlying the red-brown clay unit. Soil sample collection methods including screening (with a PID) are described in the Field Sampling Plan (Appendix B). The laboratory sample analytical program for soil samples is as follows (Table 2).

- TCL VOCs (EPA SW8260D/5035)
- PAH-17 (EPA SW8270E)
- COC Metals (arsenic, copper, lead, mercury, nickel, and zinc) (EPA 6020B/7471B)
- PCBs (EPA 8082A) (granular soils only – not clay)
- Grain Size analysis (ASTM D422), Standard Penetration Testing (SPT) (ASTM D1586), TOC (EPA Method 9060A/Lloyd Kahn)

If light NAPL (LNAPL) or DNAPL is encountered in any soil boring exhibiting properties inconsistent with prior findings of NAPL (characteristic of coal-tar-like gross contamination), a sample will be collected and submitted for laboratory forensic analysis by method EPA SW8270M/8015M as summarized in Table 2.

6.3. Groundwater Investigation

6.3.1. Monitoring Well Installation

The goal of the groundwater investigation is to assess groundwater quality in the unconfined water-bearing zone where DNAPL is known to exist. In areas where DNAPL presence is to be investigated, well

screens will be positioned to monitor the bottom of the alluvial soils directly above the area-wide clay layer that forms a barrier to vertical downward migration of DNAPL. These will also support DNAPL mobility testing (Section 6.5) if DNAPL is present. To achieve this objective, five new monitoring wells (MW-RI-1 through MW-RI-5) will be installed at the proposed locations shown on Figures 5, 6, and 7 to supplement the existing network of monitoring wells in the Lower Scajaquada OU. Each well will be constructed using 2-inch diameter Schedule 40 polyvinyl chloride (PVC) with a screened section consisting of 0.02-inch sized slot openings. The bottom of the well will terminate in the area-wide red-brown clay layer between approximately elevations 550 to 558 feet (NAVD88). Wells monitoring the top of the clay layer will be equipped with a 2-foot DNAPL collection sump extending into the area-wide clay layer below the well screen. As described in the FSP (Appendix B), the annular space surrounding the sump with be sealed with bentonite prior to installation of the sand pack.

In areas where DNAPL-impacted soils were identified and shallow wells are not present, proposed monitoring wells MW-LSC-2S-RI and MW-LSC-4S-RI will be installed with well screens positioned to monitor the upper 5 feet of the saturated zone at an approximate elevation of 565 feet (NAVD88), or 5 feet below the top of the water table as observed during drilling. These wells will have 5-foot screen lengths to assess groundwater quality in shallow groundwater near the water table.

The objective and rationale for each monitoring well installation is provided in Table 1. Monitoring well installation construction details are provided in the FSP (Appendix B).

6.3.2. Monitoring Well Development

Prior to development, the wells will be checked for NAPL presence (LNAPL and DNAPL). If NAPL is encountered, the field observations (i.e., thickness and description) will be documented on the well development forms. New wells will be developed no sooner than 48 hours after completion to remove fine-grained sediments that may have accumulated in the well during construction and to promote the hydraulic communication between the well and surrounding soil formation. For wells that do not contain NAPL, wells will be developed by surging and pumping techniques. Monitoring well development methods are described in the FSP (Appendix B). If NAPL is present, well development will utilize the methods described in the FSP that minimize the movement of NAPL to prevent contact with the riser and screened portion of the well not already in contact with DNAPL.

Following well development, an assessment of NAPL presence and mobility will be conducted at each new monitoring well location as discussed further in Section 6.5.

6.3.3. Groundwater Quality Sampling and Analysis

Following well development, new monitoring wells will be allowed to stabilize for at least two weeks and then sampled using low flow sampling methods. Prior to sampling, wells will be checked for the presence of NAPL (both LNAPL and DNAPL).

The groundwater samples collected will be analyzed for the following parameters:

- TCL VOCs by EPA Method 8260D

- TCL SVOCs by EPA Method 8270E including 1,4-Dioxane by EPA Method 8270E SIM with isotope dilution
- PFAS by EPA Method 1633
- PCBs by EPA Method 8082A
- TAL Metals by EPA Method 6020B/7470A
- Total Cyanide by EPA Method 9012B

If NAPL is encountered in new monitoring wells, and if observations based on olfactory and visual evidence, along with groundwater analytical testing results, indicate that the NAPL is inconsistent with previous findings (e.g., coal-tar-like odor with elevated BTEX and naphthalene concentrations in the aqueous phase), a sample of the NAPL will be collected and submitted for forensic analysis using EPA Methods 8270E-SIM/8015D(M).

Additional information regarding the collection methods and analysis to be used during groundwater sampling is included in the FSP (Appendix B). The analytical summary for groundwater sampling is provided in Table 2.

6.3.4. Hydraulic Conductivity Testing (New and Existing Wells)

Estimates of saturated soil hydraulic conductivity will be obtained from RI monitoring wells and monitoring wells installed during the 2021/22 Site Characterization by performing slug tests. Slug testing will be performed using rising head methods. A known volume of water will be displaced to effect head change by removing a volume of water with a hand bailer. Water level recovery will be measured automatically via instrumentation temporarily installed in the well, such as a down-hole pressure transducer. Hand measurements will also be periodically recorded to demonstrate at least 80% recovery during the test. Slug test displacement and recovery data will be analyzed using AQTESOLV (or similar) software by the Bouwer and Rice Method (1976) to yield estimates of hydraulic conductivity in each well.

6.4. Creek and Inlet Area Sediment Investigation

6.4.1. Inlet Creek Bank Surface Soil

Surface water sheens have historically (prior to IRMs) been observed on the creek bank due to daily fluctuations in surface water levels in the Inlet. When sheen was present on the surface water in the Inlet Area near the City of Buffalo Pumphouse, the sheen residue was observed to be deposited on the shoreline at the water/bank interface. Surface soil samples will be collected from the creek bank at 10 locations identified on Figure 8. The surface soil samples will be analyzed for the following parameters:

- TCL VOCs by EPA Method 8260D/5035
- PAH-17 by EPA Method 8270E
- PCBs by EPA Method 8082A
- COC Metals (arsenic, copper, lead, mercury, nickel, and zinc) by EPA Method 6020B/7471B

The creek bank surface soil sample collection involves probing surface soils in proposed sampling locations at an elevation of approximately 574 to 575 feet as described in the FSP (Appendix B). Table 2 provides an analytical summary.

6.4.2. Bathymetric Survey

Mapping and surveying work will be performed by a combination of standard Geographical Information Service (GIS) mapping combined with survey-grade GPS methods and bathymetric surveying.

A bathymetric survey of the investigation area will be performed using a single-beam bathymetric survey methodology with GPS location. Sufficient points along the shore will also be obtained so that the bathymetric survey can be tied to the upland topographic survey and a unified map of land and creek-bottom elevations prepared. The survey results will be used to create a contour figure of the creek-bottom, and the data will be provided in digital GIS-compatible format.

Mapping from the bathymetric survey will be obtained before probing and coring, so that the results of the survey can be used to modify the sampling plan, as appropriate, to consider current field conditions at the time of the work.

6.4.3. Sediment Probing Transects

Systematic sediment probing will be performed to assess the thickness and quality of sediment within the investigation area and observe for sheen generation. Probing will be conducted along 26 transects, spaced approximately 50 to 100 feet apart, as shown on Figures 8, 9, and 10. The endpoints of each transect, each probing location along the transect, and the water surface elevation will be established using survey-grade GPS. The depth of water at each location will be measured by a tape or probe with a flat plate to prevent the end from sinking into the sediment. Once the endpoints of each transect have been located, a rope will be strung across the creek between the endpoints to help the probing crew stay on the transect as they probe and sample across the length of the transect. Probes will be attempted at approximately 10-foot intervals along transect lines. Sediment probing is described in the FSP (Appendix B).

6.4.4. Sediment Sampling

Sediment borings will be completed in the Scajaquada Creek main channel and Inlet along 26 transects to evaluate sediment quality. Target sediment sampling locations are provided on Figures 8 through 10. The final location of each sediment boring may be adjusted in the field based on the presence of utilities buried beneath the Creek channel, accessibility with the sampling barge, obstructions (rip-rap, large debris), or if poor sample recovery occurs. Sediment sample locations will generally be collected at the nearest (relative to target locations on Figures 8-10) 10-foot probing locations along each transect, and the sediment sampling effort will replace the physical probing (Section 6.4.3 above) at those locations.

Sediment borings will be advanced using 3-inch diameter flush-jointed casing or 2.75-inch diameter hollow stem augers. Samples will be collected using a 4-foot Macrocore sampler or 2-inch diameter split spoon samplers. If sample recovery in sediment borings is below 70%, sampling methods will be modified in order to achieve a minimum 70% recovery goal. These modifications may include increasing

the sampler diameter, adjusting the cutting shoe on the sampler, varying core-catcher type, or modifying the Macrocore liner type or configuration. The rate of sampler advancement may also be modified to maximize sediment sample recovery. Sediment boring and sampling procedures are provided in the FSP (Appendix B). At all sediment boring locations, the full length of recovered sediment and underlying clay will be visually characterized. At certain locations, sediment will be collected from selected intervals for laboratory analyses, as indicated on Table 4. The target intervals for sampling are generally the upper two feet of sediment and the most impacted interval observed in the core. In the event the 0- to 2-ft depth interval is also the most visibly impacted, or if no depth interval appears more impacted than others, the second sample will be collected from the 2-foot interval overlying the red-brown clay. Each sediment boring will be advanced a minimum of 4 feet into the area-wide red-brown clay layer which occurs more than 15 feet below the soft sediment surface.

Each sediment boring location will be surveyed at the time of completion using a hand-held GPS capable of sub-meter accuracy. GPS coordinates will be recorded on sediment boring logs and reported in NAVD88 format.

6.4.5. Sediment Analysis

Sediments collected from Scajaquada Creek will be analyzed for chemical, biological, waste profiling, and geotechnical purposes; specific analyte lists will vary based on specific locations and objectives. The analyses to be performed on the samples from the specific sampling locations shown on Figures 8 through 10 are identified in Table 4. The various sampling activities, rationale, and analytes are summarized in the following subsections.

6.4.5.1. COC Delineation Samples

As indicated on Figures 8 through 10, there are 29 locations where analyses target a list of COCs potentially affecting the lower Scajaquada Creek area based on the findings of prior investigations in and near the creek. The primary purpose of these samples is to delineate the nature and extent of impacts. At each of those locations, two sample intervals are proposed for analysis, totaling 58 samples (excluding QA/QC samples). Of those, 47 are proposed for analysis of the focused COC Parameters analyte list and 11 are proposed for analysis of an expanded Full Suite COC analyte list. The analytes associated with each list are indicated on Table 4 and summarized below:

List	Analytes Included
COC Parameters (focused list)	TCL VOCs, PAH17, Selected metals (arsenic, copper, lead, mercury, nickel, and zinc), TOC
Full Suite COCs (expanded list)	TCL VOCs, TCL SVOCs, EPH, expanded NOAA PAH34 list, PCBs, TAL metals, total cyanide, and TOC

Where Table 4 indicates a location where samples are subject to both COC Parameters and Full Suite COCs analyses, the shallow (0- to 6-inch depth) will be subject to the Full Suite analytical list and the second sample (collected from a deeper interval) will be analyzed for the more focused COC Parameters list.

6.4.5.2. Benthic Toxicity Analysis

Sediment samples will be collected from the uppermost six inches of sediment (i.e., the most biologically active zone) at a subset of 26 of the 29 locations where COC-related sampling is planned. In addition to collecting COC-related information at these locations, additional parameters will also be analyzed to better assess other factors that may affect benthic toxicity apart from identified COCs. The additional analytical parameters at these locations are indicated on Table 4 and summarized below:

List	Analytes Included
Ecological Risk Assessment Parameters	Ammonia, TOC (if not already part of the Full Suite analyses at that location), sulfide, Acid Volatile Sulfide / Simultaneously Extracted Metals (AVS/SEM), and grain size

In addition, sediment will be collected from the upper six inches of sediment at three locations for the purpose of laboratory toxicity testing. The three target locations include T9B, T15B, and T26B (Figures 8, 9, and 10), although adjustments may be made based on the results of the Ecological Risk Assessment Parameters analysis indicated above. Standard toxicity testing will include conducting chronic, 28-day whole-sediment toxicity test with the amphipod, *Hyalella azteca*. Endpoints measured in this chronic test will include effects on survival, growth, and reproduction from 28-day exposure to site sediments per "Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates" (USEPA 2000) and "A Guidance Manual to Support the Assessment of Contaminated Sediments in Freshwater Ecosystems" (USEPA 2002). Sediment toxicity tests will include collection of bulk sediment from three pre-determined locations and will also incorporate laboratory provided control samples and a reference site/background sample for statistical comparison.

6.4.5.3. Waste Characterization

The evaluation of waste management costs in the FS requires evaluation of chemistry and characteristics for sediment that may be dredged from the creek and subject to offsite disposal. Three sediment boring locations are proposed for collection of waste characterization samples. These locations (T2B, T6B, and T23A; Figures 8 and 10) focus on the Inlet area where the greatest volume of deposition sediment is anticipated and where prior data suggests the majority of chemical impacts are present. These analyses, combined with the suite of area-wide chemical tests listed above, will allow for evaluation of both landfill and thermal treatment disposal options. Analyses for waste characterization will be performed on composite samples created from the uppermost 4 feet or full thickness of depositional material retrieved at each location, whichever is less. One composite sample will be collected at each of the three locations; three samples in total will be analyzed for waste characterization parameters. The waste characterization analytes, which are listed on Table 4, reflect typical waste profiling requirements at nearby commercial disposal facilities. The analytical parameters are summarized below:

List	Analytes Included
Waste Characterization	TCL VOCs, TCL SVOCs, TAL Metals, Pesticides/herbicides, PCBs, hazardous characteristics (pH, ignitability, reactivity, and corrosivity), total cyanide, TPH, sulfur content, and BTU content

As indicated in Table 4, both total and TCLP analyses will be performed for certain of these parameters (VOCs, SVOCs, and metals).

6.4.5.4. Geotechnical

The evaluation of certain remedial technologies for the creek as part of the planned FS may require an assessment of the effects of each technology on the stability of roads and structures along the creek. Knowledge of geotechnical properties may also be pertinent to designing support facilities, sediment dewatering, excavation support, and other ancillary activities that may be needed to implement certain remedial technologies. To support this assessment, limited geotechnical testing is proposed at six locations as part of the sediment investigation scope. The six locations were selected to be adjacent to structures that may be most susceptible to being destabilized by certain remedial activities, and focus on the following areas:

- Along the bulkhead supporting highway I-190 and closing the former creek outlet (T2A, T3A, and T5A)
- Between the State Route 198 on-ramp and Niagara Street bridge (T16B)
- Adjacent to the West Avenue Bridge (T26A and T26B)

At each of these locations, samples will be collected from the clay at the bottom of the sediment core and analyzed for the following:

List	Analytes Included
Geotechnical Parameters	Grain size, TOC, Atterberg Limits, and permeability (if intact Shelby tube samples can be recovered)

6.5. DNAPL Mobility Assessments

DNAPL mobility assessments will be conducted at:

- Wells installed during the RI containing DNAPL
- Temporary wells installed at creek borings T26A and T26B (if DNAPL is observed)
- Six existing wells monitored during 2021/2022 SC that contained DNAPL (DEC-MW-100, MW-LSC-2, MW-LSC-4, MW-LSC-5, 1660-MW-7, 1660-MW-8)

The mobility assessment will be conducted after the completion of monitoring well development, sampling, and hydraulic conductivity testing. The procedure involves removing accumulated DNAPL from the 2-foot sump in each well and measuring the rate of DNAPL recovery over time until the sump is completely refilled (approximately 1/3 of a gallon). Details are described in the FSP (Appendix B).

The frequency of testing will be well-specific:

- Daily DNAPL removal will occur in wells for approximate one week if the gravity recovery of DNAPL in the well is sufficiently rapid to re-fill the entire sump and additional DNAPL removal is necessary to allow continued gravity drainage of DNAPL.
- The testing will be reduced if DNAPL recovery rates are slower.

The duration of DNAPL mobility testing will extend for a maximum of 6 months after RI monitoring wells are sampling and hydraulic conductivity testing is complete.

Planned Mobility Assessment Schedule:

Initial Phase: Daily assessments for the first week to establish recovery rates.

Intermediate Phase: Adjust frequency based on initial findings; wells with rapid recovery may continue daily checks (for a maximum of one week), while others may shift to weekly.

Final Phase: Monthly assessments for all wells for the last three months, focusing on wells with slower recovery rates and DNAPL recovery in more rapidly re-filling wells.

6.6. Evaluation of DNAPL Migration Pathways Associated with Infrastructure

The 2022 Site Characterization identified numerous utilities and below-grade infrastructure in the Lower Scajaquada Creek area. This included the evaluation of the Niagara Street stormwater sewers below the CSX Railroad Bridge and the conveyance piping to the City of Buffalo Pumphouse. As described in Section 4, an IRM was implemented to minimize contaminated groundwater from infiltrating the storm sewer system and to mitigate the entry of DNAPL, including accumulated DNAPL in the Pumphouse Wet Well (vault). Although sampling was conducted shortly after the IRM's completion, including NYSDEC pipe slip-lining, the performance of the IRM has not been quantified through analytical testing.

The following will be conducted to assess DNAPL/contaminated groundwater entry into the Niagara St. storm sewer servicing the City of Buffalo Pumphouse (see Figure in Section 4):

- Manholes #1 through #4 in Niagara Street, Manholes 21 and 23, and the Pumphouse vault will be inspected and probed with a white, PVC rod for DNAPL presence.
- Water samples will be collected from Manholes 21 and 23 and the Pumphouse discharge and analyzed for parameters specified in Table 2 (full suite of compounds).

During RI/FS work plan preparation, design drawings provided by the Buffalo Sewer Authority (BSA) showing infrastructure (e.g., sewer and utility lines), as well as drawings provided by others, were reviewed with the objective of identifying potential migration pathways for the transport of DNAPL away from the Lower Scajaquada Creek area. These included:

- The Buffalo Sewer Authority (BSA) interceptor sewer that parallels the NYS Thruway I-190 and passes beneath the former and current creek outlet,
- Buried high-voltage electric transmission lines beneath Niagara Street, and

- BSA Stormwater and sanitary sewers along Niagara Street and Tonawanda Street.

The presence of these utilities and features and the ability to act as preferential pathways for DNAPL migration away from the Scajaquada Creek area will be evaluated following completion of investigations described in Section 6.1 through 6.5. For example, if DNAPL is identified in boring SB-RI-14, then manholes located along Wayne Street and Niagara Street will be probed for DNAPL presence. Decisions to investigate infrastructure outside the Scajaquada Creek OU will be made after consultation with the representatives from the Project Coordination Team.

6.7. Groundwater and Surface Water Elevation Monitoring

Groundwater elevation measurements (synoptic rounds) will be collected monthly for a duration of six months and quarterly for one year afterward. The measurements will be collected from the following monitoring wells:

RI Monitoring Wells:		
Well ID	**Top of Riser Elevation (EL.)[1]**	**Location**
MW-RI-1	To be surveyed upon Completion	Under 198 expressway, west of southern DNAPL recovery vault
MW-RI-2		Northeastern corner of 31 Tonawanda Street parcel, near West Avenue Bridge
MW-RI-3		Former location of 31-MW-4R.
MW-RI-4		1660 Niagara Street
MW-RI-5		Next to 190, on Jesse Kregal pathway
MW-LCS-2S-RI		Next to MW-LCS-2
MW-LCS-4S-RI		Next to MW-LCS-4
Existing Monitoring Wells:		
Well ID	**Top of Riser Elevation (EL.)[1]**	**Location**
1660-TPMW-1	582.20	1660 Niagara Street
1660-MW-1	578.79	1660 Niagara Street
1660-MW-2	577.65	1660 Niagara Street
1660-MW-3R	578.63	1660 Niagara Street
1660-MW-5R	577.61	1660 Niagara Street
1660-MW-6	577.20	1660 Niagara Street
1660-MW-7	578.34	1660 Niagara Street
1660-MW-8	580.80	1660 Niagara Street
1675-MW-1	577.51	1675 Niagara Street
1675-MW-2	580.29	1675 Niagara Street
MW-100	578.56	Bike Path
MW-103	578.37	Bike Path
MW-106	579.84	Bike Path
68-MW-2	585.55	68 Tonawanda Street
68-MW-3	584.26	68 Tonawanda Street
68-MW-4	579.22	68 Tonawanda Street
31-MW-2	581.59	31 Tonawanda Street
31-MW-3R	578.93	31 Tonawanda Street
57-MW-1	577.70	57-71 Tonawanda Street
57-MW-3	579.15	57-71 Tonawanda Street
57-MW-4	582.75	57-71 Tonawanda Street
57-MW-5	584.00	57-71 Tonawanda Street
MW-BGL-1	581.74	On Bike Path, under Route 198 ramp and I-190 Expressway
MW-BG1-3	579.82	Off Bike Path, under I-190 Expressway
MW-LCS-1	585.69	Between Route 198 ramp and West Avenue
MW-LCS-2	585.35	Under Route 198 ramp

MW-LSC-3	586.95	Under Route 198 ramp
MW-LSC-4	582.13	Between the on and off Route 198 ramps
MW-LSC-5	582.48	Corner of Niagara Street and Tonawanda Street
MW-PS-1	578.34	East of pumphouse
PZ-PS-1	578.33	East of pumphouse
MW-PS-2	579.42	East of pumphouse
PZ-PS-2	579.42	East of pumphouse

Notes: 1. Elevations reported in NAVD88 datum.

Monitoring well locations are shown on Figures 5, 6, and 7.

Two surface water elevation monitoring stations will be temporarily installed at an upstream location (US-Surface-1) and a downstream location (DS-Surface-2) along Scajaquada Creek. The stations will be constructed using a PVC standpipe fastened to post. A transducer/data logger will be installed at each surface water monitoring location. Exact locations will be field determined considering safe access and physical protection of the station. Surface water elevations will be manually measured and documented daily during drilling activities.

The groundwater and surface water elevation measurements will be analyzed to determine a direct correlation between the two.

6.8. Survey

A survey of the RI sampling points and important site features will be performed by a NYS licensed surveyor. All horizontal locations will be reported in the applicable NYS horizontal coordinates (North American Horizontal Datum 1983 (NAD83, NYS West Zone) and latitude and longitude coordinates. All vertical measurements will be reported in NAVD88.

6.9. Management of Investigation Derived Waste

Waste materials generated from field investigation activities including drill cuttings, sediments and plastic used for surface containment of spoils and fluids, purge water, drill cuttings, personal protective equipment, and disposable sampling equipment will be containerized in separate 55-gallon drums. Management of IDW is described in the FSP (Appendix B).

6.10. Community Air Monitoring

Community air monitoring will be performed to provide real-time measurements of total VOCs and particulate (airborne dust) concentrations in air at the downwind perimeter of each designated work area when intrusive investigation activities (i.e., drilling activities) are in progress. The monitoring provides protection for the downwind community, including residences and businesses and on-site passersby not directly involved with the RI work activities. The procedures follow methods described in the CAMP, which is included with the HASP (Appendix D), and are consistent with NYSDOH guidance.

6.11. CVOC Source Characterization

The NYSDEC is leading a SC to delineate the nature and extent CVOCs on the property at 1660 Niagara Street (the site) in the City of Buffalo and to determine whether CVOC contamination from the site is a

source of sediment contamination in lower Scajaquada Creek. The planned investigation will include surface soil sampling, soil borings and the installation and sampling of groundwater monitoring wells. The SC will include:

- Surface Sampling – Collection of five surface soil samples from the mound area on the southwestern end of the 1660 Niagara St. property.
- Soil Borings - Install 12 soil borings to the depth of the area-wide brown clay to determine the vertical extent of CVOC contamination in the overburden soils on site and collect two samples from each boring for laboratory analysis.

Soil samples collected during the SC will be analyzed for: TAL Metals, PCBs, 8260 VOCs, 8270 SVOCs, 1,4-dioxane and PFOA/PFAS.

- Groundwater Investigation – Install five overburden monitoring wells to the depth of the area-wide brown clay.

Samples collected during the SC will be analyzed for: TAL Metals, PCBs, 8260 VOCs, 8270 SVOCs, 1,4-dioxane and PFOA/PFAS.

Site Characterization findings will be summarized by the NYSDEC and incorporated into RI Data Summary Report (see Section 7).

7. RI Data Summary Report

The RI Data Summary Report will be prepared to support the Feasibility Study. National Fuel will coordinate with NYSDEC to incorporate findings from their Site Characterization activities into an appendix of the RI Data Summary Report. A preliminary outline is provided below:

1. Executive Summary - Brief overview of the site, objectives, key findings, and recommendations.
2. Introduction - Background information on the site, purpose and scope of the investigation, regulatory framework, and project objectives.
3. Site Description and History - Location and physical setting, historical site use and previous investigations.
4. Sampling and Analysis Summary - Description of sampling locations and rationale, types of samples collected, work plan deviations, analytical methods and laboratory procedure, quality assurance/quality control (QA/QC) discussion, data validation and usability assessment.
5. Data Summary, Findings, and Interpretation - Presentation of data in tables, graphical summaries, and maps, interpretation of analytical results, comparison with regulatory standards and guidelines.
6. Updated Conceptual Site Model – The preliminary CSM will be updated with information obtained from investigations completed in the Lower Scajaquada Creek OU.
7. Conclusions and Recommendations.
8. References.

The RI Data Summary Report will also include tables, figures, and attachments to document the completed work and summarize the results and findings.

8. Qualitative Human Health Exposure Assessment

The Qualitative Human Health Exposure Assessment (or the exposure assessment) for the Lower Scajaquada Creek OU will focus on potential human exposure to DNAPL constituents under current land use conditions of the Lower Scajaquada Creek area. The exposure assessment will be completed after the RI Summary Report and will be incorporated into the Feasibility Study (see Section 10) and will include:

- A description of the constituent source;
- An explanation of the release and transport mechanisms;
- Identification of all potential exposure point(s) where actual or potential human contact with an impacted medium may occur;
- Description(s) of the route(s) of exposure (i.e., ingestion, inhalation, dermal absorption); and
- A characterization of the receptor populations who may be exposed to DNAPL constituents at a point of exposure.

These items represent the five elements of an exposure pathway. An exposure pathway is complete when all five elements of an exposure pathway are documented; a potential exposure pathway exists when any one or more of the five elements comprising an exposure pathway are unknown. An exposure pathway may be eliminated from further evaluation when any one of the five elements comprising an exposure pathway has not existed in the past, does not exist in the present, and can reasonably be anticipated to never exist in the future.

The NYSDEC and NYSDOH will integrate the exposure assessment findings related to coal tar-derived DNAPL and associated constituents of potential concern (COPCs) into their overall exposure assessment of the upland areas in the Lower Scajaquada Creek OU. This comprehensive assessment will include non-coal tar-related COPCs identified during their investigation. The findings will be summarized in the Feasibility Study Report described in Section 10.

9. Fish and Wildlife Resource Impact Analysis

A Fish and Wildlife Resource Impact Analysis (FWRIA) will be performed as part of the RI by qualified personnel as defined within the NYSDEC Fish and Wildlife Impact Analysis for Inactive Hazardous Waste Sites guidance document (NYSDEC, 1994). An initial habitat assessment conducted as a component of FWRIA Part 1 - Resource Characterization, was completed during the preparation of this workplan. The remaining components of the Part 1 - Resource Characterization and Part 2- Ecological Impact Assessment will be completed using information collected from the sediment investigation and information in this work plan and incorporated into the FS Report (see Section 10).

In accordance with the FWRIA guidance, a qualitative evaluation of actual or potential impacts to fish and wildlife resources from site-related constituents will be performed. Available information and the resource descriptions developed from the evaluation will be used to characterize the exposure setting, identify migration pathways, and evaluate contaminant fate and transport and potential effects to fish and wildlife resources. This information will also be used to identify contaminants of potential ecological concern (COPECs) via comparison of the existing site media data to ecologically based screening values and/or criteria.

An onsite habitat assessment was conducted on October 23, 2024 in Lower Scajaquada Creek, focusing on the area primarily south of the West Avenue Bridge and the Scajaquada Creek Slip area. The Lower Scajaquada Creek area is characterized as a highly urbanized, degraded aquatic habitat within a heavily developed watershed, primarily consisting of a channelized creek with limited natural vegetation, and bank cover type dominated by staghorn sumac (*Rhus typhina*), tall goldenrod (*Solidago altissima*), box elder (*Acer negundo*) and various dogwood species (*Cornus* sp.). Due to the nature of the observed substrate, submerged aquatic vegetation was limited to the shallower depositional areas of sediment along the creek banks thus significantly reducing fish cover. Observed fish species included rock bass (*Ambloplites rupestris*), small mouth bass (*Micropterus dolomieu*) and striped shiner (*Luxilus chrysocephalus*). The banks of Lower Scajaquada Creek ranged from gently sloping areas dominated with terrestrial herbaceous vegetation and sporadic tree cover to engineered shores lines.

Scajaquada Creek in the Lower Scajaquada Creek OU transitions from a Class C to a Class B stream and flows into the Niagara River, (Class A-Special stream). Sensitive species potentially present in this segment of Lower Scajaquada Creek include the salamander mussel (Simpsonaias ambigua) and the lake sturgeon (Acipenser fulvescens). The Niagara River, to which Lower Scajaquada Creek discharge, is listed as a NYS freshwater mussel screening stream.

9.1. Initial Sediment Characterization

As part of the FWRIA, sediment data collected during the RI/FS will initially be evaluated through a comparison to NYSDEC screening benchmarks, including the NYSDEC (2014c) Class A and Class C freshwater Sediment Guidance Values (SGV). SGV are not available for all contaminants; therefore, for contaminants where SGV are not available, the SGV will be supplemented with benchmarks from additional sources. The research for alternate screening benchmarks will include a general literature search and query of multiple databases, including the following:

- National Oceanic and Atmospheric Administration (NOAA) Screening Quick Reference Tables (SQuiRT) (Buchman, 2008)
- USEPA Region 4 Ecological Risk Assessment Supplemental Guidance (March 2018 update)
- USEPA Region 3 sediment screening benchmarks (2006); and
- USEPA Region 5 Resource Conservation Recovery Act (RCRA) Corrective Action – Ecological Screening Benchmarks (2003)

The screening approach initially identifies a preliminary list of COPEC that includes constituents detected above conservative benchmarks, specifically the Class A SGV, which will be retained for further evaluation as detailed in the following sections.

9.2. COPEC Refinement

An ecological CSM will be developed following the RI sediment characterization. The ecological CSM will describe the predicted relationships between ecological receptors and stressors to which they may be exposed. The CSM will also describe the primary, secondary, and tertiary exposure pathways or co-occurrences among exposure pathways, ecological effects, and ecological receptors (USEPA, 1998). The CSM will summarize how COPECs may be transported through potentially impacted sediments and result in exposure to ecological receptors. Based on the observations made during the October 2024 habitat assessment, the primary mechanisms of exposure to COPECs in the Lower Scajaquada Creek are direct exposure to impacted sediments in the observed depositional areas and assimilation by a species (e.g., prey/food item) at one trophic level, bioaccumulated by that trophic level, and transferred to a higher trophic level species through consumption. Bioavailability considerations, including total organic carbon and AVS/SEM modeling, will be incorporated into the exposure assessment process. Based on the proposed exposure pathways, the key receptors of concern are benthic invertebrates, fish and semi-aquatic piscivorous mammals and avian species.

In order to assess the potential for impacted sediments to pose ecological harm to receptors within Lower Scajaquada Creek, surrogate species, including American mink (*Neogale vison*) and belted kingfisher (*Megaceryle alcyon*) have been identified as representative species to evaluate exposure to mammalian and avian receptors based on feeding guild. Surrogate species for benthic invertebrates and fish were not selected as these receptors were evaluated on a community rather than individual level. The following sections discuss the identification of endpoints that will be utilized to evaluate the ecological effects associated with the potential exposure of ecological receptors to COPECs.

9.2.1. Assessment and Measurement Endpoints

Assessment endpoints are statements of the characteristics or attributes of the environment that are to be protected. The following assessment endpoints were developed based on the selection criteria described in the USEPA Environmental Risk Assessment Guidance for Superfund (USEPA, 1997).

- Assessment Endpoint #1: Evaluate the potential for adverse changes in the survival, reproduction, and growth of benthic invertebrate and fish populations on a community level resulting from potential exposure to COPECs in depositional sediments.

- Assessment Endpoint #2: Evaluate the potential for adverse changes in the survival, reproduction, and growth of populations of semi-aquatic mammal species potentially utilizing the Site resulting from exposures to COPECs in sediments and prey (fish and benthic invertebrates).
- Assessment Endpoint #3: Evaluate the potential for adverse changes in the survival, reproduction, and growth of populations of semi-aquatic avian species potentially utilizing the Site resulting from exposures to COPECs in sediments and prey (fish and benthic invertebrates).

Because the above assessment endpoints generally cannot be measured directly, measurement endpoints have been identified. There are three types of measurement endpoints, or lines of evidence, that are used to assess the status and potential changes in the attributes of the environment. The lines of evidence will include:

- Determination of the potential for ecological effects by the comparison of COPEC concentrations to media-specific ecotoxicological benchmarks derived from the literature, when available;
- Estimation of the potential for ecological effects on higher trophic level organisms (American mink and belted kingfisher) from COPECs based on dietary exposure; and
- Toxicity studies to directly measure the effects of contaminants on local biota.

9.2.2. Risk Characterization

The analytical results for sediment COPECs observed to exceed the initial sediment characterization (Section 9.1) will be utilized to calculate Exposure Point Concentrations (EPCs) that will be used to further refine the potential for COPEC exposure to ecological receptors. The EPCs for ecological receptors in sediment will be developed based on calculation of the 95% Upper Confidence Limit (UCL) of each COPEC.

The direct contact exposure evaluation includes the comparison of EPC of detected COPECs to direct contact ecological criteria for the analysis of risks to benthic macroinvertebrate and fish communities will be conducted, as will estimations of potential exposures to the surrogate wildlife receptor (American mink and belted kingfisher). The exposure to COPECs in sediment and prey will be calculated through the development of an average daily dose (ADD), which is the amount of COPEC that an individual organism may be exposed to daily through direct or indirect exposure pathways within the Site area.

The calculated ADDs will then be compared to industry recognized Toxicity Reference Values (TRVs). TRVs will be selected for each COPEC to develop Hazard Quotients to assess the potential presence of risk to ecological receptors from sediments with Lower Scajaquada Creek.

10. Feasibility Study

The FS for the Scajaquada Creek OU will evaluate potential remedial actions to address DNAPL and other COC contamination in creek sediments, soils, and groundwater as necessary to meet remedial objectives. The FS will include the following:

- Summarize RI Findings: Provide a summary of site investigation information describing the extent of DNAPL and other COCs, the fate and transport of contamination, and identification of complete exposure pathways that could impact human health, and ecological risk findings.
- The RI Summary Findings will be coordinated with NYSDEC to incorporate findings from RI work conducted by National Fuel and by NYSDEC.
- Identify Remedial Action Objectives (RAOs): Establish specific goals for the protection of human health and the environment, considering the nature and extent contamination. This will include consideration of New York State sediment guidance values and other applicable screening values that are consistent with Niagara River AOC BUIs and other appropriate considerations including planned future uses.
- Identify Response Actions and Development of Remedial Alternatives: A range of potential remedial actions will be formulated to address the RAOs. This will include but not limited to, removal and containment strategies, practical treatment technologies, and institutional controls.
- Screen and Evaluate Alternatives: Conduct a detailed analysis of each remedial alternative based on criteria such as effectiveness, implementability, and regulatory compliance. Each alternative will include a cost estimate.
- Conduct a Comparative Analysis: Compare the shortlisted alternatives against each other to determine the most suitable remedial action. This analysis will consider long-term effectiveness, reduction of toxicity, mobility, or volume of contaminants, short-term effectiveness, and overall protection of human health and the environment.
- Recommend the Preferred Alternative: Recommend the most appropriate remedial action based on the comparative analysis, stakeholder input, and regulatory requirements. This will include involvement of community members, regulatory agencies, and other stakeholders in the decision-making process to ensure transparency.

The FS findings will be incorporated in a Draft RI/FS Report. The draft report will summarize the RI findings and FS activities identified above. The RI/FS findings will be presented at a public meeting. Comments received from the public will be incorporated into the Final Remedial Investigation/Feasibility Study Report for the Scajaquada Creek OU.

11. RI/FS Schedule

RI/FS Schedule
GLLA Program - Lower Scajaquada Creek OU
Buffalo, NY

Task Name	2025 Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	2026 Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	2027 Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov
Complete Draft Work Plans		■		■																															
Submit Final Work Plans						◆																													
Subcontractor Agreements		■	■																																
Access Agreements/Permits			■	■																															
RI Landside Field Activities					■	■	■	■	■	■	■	■																							
Quarterly RI Landside Activities														■			■			■			■												
RI Sediment Field Activities							■	■																											
Laboratory Analysis/Validation/Data Compilation						■	■	■	■	■	■	■	■	■																					
RI Figures/Mapping Preparation/Findings Summary								■	■	■	■	■	■	■	■	■																			
RI Data Submission Report																	◆																		
Qualitative Human Health Risk Assessment																	■	■	■																
Fish and Wildlife Resource Impact Analysis							■	■	■	■	■	■	■	■	■	■	■	■	■																
Develop RAOs Consistent with AOC BUIs																				■	■														
RA Options/Technology Screening																						■	■												
Remedial Alternatives Costing/Evaluations																								■	■	■	■	■							
Select Preferred Alternative																													◆						
Draft RI/FS Report Preparation								■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■		
Public Meeting																																	●		
Submit Final RI/FS Report																																		◆	
PCT Meetings		●	●	●		●	●	●		●	●	●		●	●	●		●	●	●		●	●	●		●	●	●		●	●	●	●	●	

12. References

Buchman, M. F., 2008. Screening Quick Reference Table (SQuiRTs). NOAA OR & R Report ; 08-1

GEI Consultants, Inc. P.C. , May 11, 2022. Email submittal to NYSDEC for GLLA Project Agreement – Framework for Phased Scope of Work. NYSDEC acceptance email May 16, 2022.

GEI Consultants, Inc. P.C. August 2021. Site Characterization Work Plan Former Buffalo Gaslight Iroquois Gas Corp. Site Buffalo, New York NYSDEC Site #915351

GEI Consultants, Inc. P.C. June 2022, Revised October 2022. Site Characterization Report Former Buffalo Gaslight Iroquois Gas Corp. Site and Lower Scajaquada Creek Buffalo, New York NYSDEC Site #915351

GEI Consultants, Inc. P.C. March 2023, Pre-Design Investigation, and Interim Remedial Measures Report - Lower Scajaquada Creek City of Buffalo Pumphouse -NYSDEC Spill #1406307

NYSDEC, October 1994. Fish and Wildlife Impact Analysis for Inactive Hazardous Waste Sites.

NYSDEC, June 2014. Screening and Assessment of Contaminated Sediment.

RETEC Engineering, P.C., August 2000. Final Engineering Report, Scajaquada Creek Sediment Remediation Buffalo, NY NYSDEC #91514B

USEPA, June 1997. Ecological Risk Assessment Guidance for Superfund: Process for Designing and Conducting Risk Assessments

USEPA, March 1998. Region 4 Ecological Risk Assessment Supplemental Guidance

USEPA, March 2000. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants with Freshwater Invertebrates, Second Edition.

USEPA, December 2002. A Guidance Manual to Support the Assessment of Contaminated Sediments in Freshwater Ecosystems Volume II - Design and Implementation of Sediment Quality Investigations.

USEPA. 2006. Region 3 Ecological Screening Benchmarks. Available at: http://epa.gov/reg3hscd/risk/eco/index.htm

USEPA. 2003a. Region 5 RCRA Corrective Action – Ecological Screening Levels. Available at: http://www.epa.gov/reg5rcra/ca/edql.htm

Tables

Table 1. Soil Boring and Monitoring Well Summary

Table 2. Laboratory Sample Analytical Summary for Soil, Groundwater, and NAPL

Table 3. Sediment Investigation Transects and Objectives

Table 4. Sediment Investigation Boring and Analytical Summary

Table 1. Soil Boring and Monitoring Well Summary
GLLA Scajaquada Creek OU
Buffalo, New York

Location	Primary/ Contingency*	Objective/Rationale	Boring Termination Depth [1,2]	Property Owner/Access Requirements
Primary Borings/Monitoring Wells				
MW-RI-1	Primary	Groundwater Quality/Verification of Source Control and Evaluate Extent of DNAPL.	AWRBCL	NYSDOT
MW-RI-2	Primary	Groundwater Quality/Verification of Source Control and Evaluate Extent of DNAPL (combine boring information from Creek Sediment Investigation Transect T-26).	AWRBCL	31 Tonawanda Street, LLC
MW-RI-3	Primary	Groundwater Quality/Evaluate Extent of DNAPL/Geotechnical Data (destroyed 31 Tonawanda well MW-4R).	AWRBCL	31 Tonawanda Street, LLC
MW-RI-4	Primary	Groundwater Quality/Evaluate Extent of DNAPL and Assess Potential for Northern Migration Pathway/Geotechnical Data.	AWRBCL	1660 Niagara Street
MW-RI-5	Primary	Groundwater Quality/Evaluate Extent of DNAPL and Assess Potential Historic Migration Pathway (former Scajaquada Creek outlet).	AWRBCL	City of Buffalo/NYSDOT
MW-LSC-2S-RI	Primary	Evaluate groundwater quality in upper saturated zone above DNAPL impacted soil.	Screen upper 5 feet of Saturated Zone (Elevation 565 ft)	City of Buffalo/NYSDOT
MW-LSC-4S-RI	Primary	Evaluate groundwater quality in upper saturated zone above DNAPL impacted soil.	Screen upper 5 feet of Saturated Zone (Elevation 565 ft)	City of Buffalo/NYSDOT
Soil Borings				
T26A	Primary	Creek Boring - Evaluation of Source Control and assess extent of DNAPL (temporary well install/testing for potential mobility).	AWRBCL	in waterway
T26B	Primary	Creek Boring - Evaluation of Source Control and assess extent of DNAPL (temporary well install/testing for potential mobility).	AWRBCL	in waterway
SB-RI-1	Contingency	Evaluation of Source Control and assess extent of DNAPL/Step-out location if MW-RI-1 impacts are observed.	AWRBCL	Pratt & Lambert
SB-RI-2	Primary	Evaluate Source Control and assess extent of DNAPL. If DNAPL encounted install well to assess mobility.	AWRBCL	NYSDOT
SB-RI-3	Contingency	Evaluation of extent DNAPL.	AWRBCL	NYSDOT
SB-RI-4	Primary	Evaluation of extent if DNAPL.	AWRBCL	31 Tonawanda Street, LLC
SB-RI-5	Primary	Evaluate extent of DNAPL.	AWRBCL	NYSDOT
SB-RI-6	Contingency	Evaluation of extent if DNAPL is observed at SB-RI-5.	AWRBCL	NYSDOT
SB-RI-7	Primary	Evaluate extent of DNAPL (install well if alluvial soils are identified and contain DNAPL).	AWRBCL	City of Buffalo
SB-RI-8	Contingency	Evaluation of extent if DNAPL is observed at SB-RI-7.	AWRBCL	City of Buffalo
SB-RI-9	Primary	Evaluate extent of DNAPL - (install well if alluvial soils are identified and contain DNAPL) Complete Evaluation of Sand below AWRBCL if no DNAPL.	BCL	City of Buffalo
SB-RI-10	Contingency	Evaluation of extent if DNAPL is observed at SB-RI-9.	AWRBCL	City of Buffalo
SB-RI-11	Primary	Evaluate Extent of DNAPL - Complete Evaluation of Sand below AWRBCL.	BCL	NYSDOT
SB-RI-12	Contingency	Evaluation of extent if DNAPL is observed at MW-RI-5 in Historic Creek Outlet.	AWRBCL	NYSDOT
SB-RI-13	Primary	Evaluate Northern Extent of DNAPL near Historic Creek Outlet.	AWRBCL	City of Buffalo/NYSDOT
SB-RI-14	Primary	Assess Potential for Northern Migration Pathway.	AWRBCL	City of Buffalo
SB-RI-15	Primary	Assess Potential Migration Pathway toward Black Rock Canal.	AWRBCL	City of Buffalo
SB-RI-16	Primary	Assess Potential Migration Pathway toward Black Rock Canal.	AWRBCL	City of Buffalo
SB-RI-17	Primary	Evaluate shallow impact at MW-LSC-3	AWRBCL	City of Buffalo/NYSDOT
SB-RI-18	Primary	Evaluate Extent of DNAPL.	AWRBCL	57-71 Tonawanda Street, LLC
SB-RI-19	Primary	Evaluate Extent of DNAPL.	AWRBCL	57-71 Tonawanda Street, LLC

Notes
1. AWRBCL - Area-wide red brown clay layer at assumed elevation between elevation 550 and 558 ft. Boring will be terminated a minimum depth of 4 ft into the clay: beyond any visual evidence of DNAPL.
2. BCL - Below Clay Layer (AWRBC)L. Boring will completely penetrate AWRBCL until till layer above bedrock is encountered.
*Primary wells and borings to be completed duirng the RI, Contingency boring locations to completed if RI objective is not satisfied by Primary.

Table 2. Laboratory Sample Analytical Summary for Soil, Groundwater, and NAPL
GLLA Scajaquada Creek OU
Buffalo, New York

Matrix	Parameter	Analytical Method	Field Samples				QC Blanks		
			Field Samples[a]	Field Duplicate	MS/MSD[b] (Total)	Sub- Total	Trip Blank	Equipment Blank	Total
Subsurface Soil Samples	TCL VOCs	EPA SW 8260D/5035	44	3	3	56	NA	NA	56
	PAH-16/17	EPA SW 8270E	44	3	3	56	NA	NA	56
	COC Metals (arsenic, copper, lead, mercury, nickel, and zinc)	EPA SW 6010D/6020B/7471B	44	3	3	56	NA	NA	56
	PCBs	EPA SW 8082A	21	2	2	29	NA	NA	29
	Grain Size analysis, Standard Penetration Testing (SPT), TOC	ASTM D422 - sieve & hydrometer, D1586 SPT, TOC - 9060A/Lloyd Kahn	MW-RI-3 (2) MW-RI-4 (2)	NA	NA	4	NA	NA	4
Creek Bank Surface Soil Samples	TCL VOCs	EPA SW 8260D/5035	10	1	1	12	NA	NA	12
	PAH-16/17	EPA SW 8270E	10	1	1	12	NA	NA	12
	PCBs	EPA SW 8082A	10	1	1	12	NA	NA	12
	COC Metals (arsenic, copper, lead, mercury, nickel, and zinc)	EPA SW 6010D/6020B/7471B	10	1	1	12	NA	NA	12
Groundwater Samples (Monitoring Wells)	TCL VOCs	EPA SW 8260D	7	1	1	9	1	1	14
	TCL SVOCs	EPA SW 8270E	7	1	1	9	NA	1	13
	Total Cyanide	EPA SW 9012B	7	1	1	9	NA	1	13
	TAL Metals	EPA SW 6010B/6020B/7470A	7	1	1	9	NA	1	13
	1,4 Dioxane	EPA SW 8270E - SIM	7	1	1	9	NA	1	13
	PCBs	EPA SW 8082A	7	1	1	9	NA	1	13
	PFAS	EPA SW 1633	7	1	1	9	NA	1	13
Groundwater Samples (City of Buffalo Pumphouse/Conveyance Piping)	TCL VOCs	EPA SW 8260D	3	1	1	5	1	1	7
	TCL SVOCs	EPA SW 8270E	3	1	1	5	NA	1	6
	Total Cyanide	EPA SW 9012B	3	1	1	5	NA	1	6
	TAL Metals	EPA SW 6010B/6020B/7470A	3	1	1	5	NA	1	6
	1,4 Dioxane	EPA SW 8270E - SIM	3	1	1	5	NA	1	6
	PCBs	EPA SW 8082A	3	1	1	5	NA	1	6
	PFAS	EPA SW 1633	3	1	1	5	NA	1	6
NAPL Samples	Forensic Analysis	EPA SW 8270M/8015M	TBD	NA	NA	TBD	TBD	NA	TBD

Notes:

(a) The number of samples assumes two soil samples collected from each Primary boring/well location. If Contingency borings are needed, additional soil samples will be collected. QC samples will be collected as specified in the Quality Assurance Project Plan.

(b) Matrix spike / matrix spike duplicate for organic analyses; matrix spike and laboratory duplicate for inorganic analysis.

TBD - To be determined based on areas where NAPL is encountered and if inconsistent with prior charcteristics suggestive of source material associated with water gas tar.

Table 3. Sediment Investigation Transects and Objectives
GLLA Scajaquada Creek OU
Buffalo, New York

Sample Location	DNAPL Delineation		Sediment-related objectives targeted							FS Support	Source control evaluation	Notes/Comments
	Vertical NAPL Delin (visual)	Horizontal NAPL Delin (visual)	Sed Vol / Thickness (visual)	Vert Extent of Contam (analytical)	Eco Risk Assessment Analytical	Benthic Tox	Geotech Properties	Waste Profiling	Migration Pathway Eval			
Transects 1-26 Probing	X	X	X						X	X		Exploratory probing using a PVC pipe or steel rod to assess for sheen along transect.
T1A	X	X	X	X						X		
T1B	X	X	X	X						X		
T2A	X	X	X	X			X			X		
T2B	X	X	X	X	X			X		X		Benthic toxicity &/or waste profile samples in Inlet may be collected from two depth intervals
T2C	X	X	X							X		
T3A	X	X	X				X			X		
T3B	X	X	X							X		
T4A	X	X	X	X	X					X		
T4B	X	X	X							X		
T5A	X	X	X				X			X		
T5B	X	X	X							X		
T6A	X	X	X							X		
T6B	X	X	X	X	X			X		X		
T7A	X	X	X							X		
T7B	X	X	X							X		
T8A	X	X	X	X	X					X		
T8B	X	X	X							X		
T9A	X	X	X	X	X					X		
T9B	X	X	X			X				X		
T10A	X	X	X							X		
T10B	X	X	X							X		
T11A	X	X	X	X	X					X		
T11B	X	X	X	X	X					X		
T12A	X	X	X	X	X					X		
T12B	X	X	X							X		
T12C	X	X	X							X		
T13A	X	X	X	X	X					X		
T13B	X	X	X	X	X					X		
T14A	X	X	X	X	X					X		
T14B	X	X	X							X		
T14C	X	X	X	X	X					X		
T15A	X	X	X	X	X					X		
T15B	X	X	X	X	X	X				X		
T16A	X	X	X	X	X					X		
T16B	X	X	X	X	X		X			X		
T17A	X	X	X	X	X					X		
T17B	X	X	X							X		
T18B	X	X	X	X	X					X		
T18A	X	X	X							X		
T19A	X	X	X	X	X					X		
T19B	X	X	X	X	X					X		

Table 3. Sediment Investigation Transects and Objectives
GLLA Scajaquada Creek OU
Buffalo, New York

Sample Location	DNAPL Delineation		Sediment-related objectives targeted							FS Support	Source control evaluation	Notes/Comments
	Vertical NAPL Delin (visual)	Horizontal NAPL Delin (visual)	Sed Vol / Thickness (visual)	Vert Extent of Contam (analytical)	Eco Risk Assessment Analytical	Benthic Tox	Geotech Properties	Waste Profiling	Migration Pathway Eval			
T20A	X	X	X							X		
T21A	X	X	X	X	X					X		
T22A	X	X	X							X		
T22B	X	X	X							X		
T23A	X	X	X					X		X		
T23B	X	X	X	X	X					X		
T24A	X	X	X	X	X					X		
T24B	X	X	X							X		
T25A	X	X	X							X		
T25B	X	X	X	X	X					X		
T26A	X	X	X	X	X		X			X	X	Boring supports assessment of Source Control
T26B	X	X	X	X	X	X	X			X	X	Boring supports assessment of Source Control

Table 4. Sediment Investigation Boring and Analytical Summary
GLLA Scajaquada Creek OU
Buffalo, New York

	Transect Boring ID:	*T1A*	*T1B*	*T2A*	*T2B*	*T2C*	*T3A*	*T3B*	*T4A*	*T4B*	*T5A*	*T5B*	*T6A*	*T6B*	*T7A*	*T7B*	*T8A*	*T8B*	*T9A*	*T9B*	*T10A*	*T10B*	*T11A*	*T11B*	*T12A*	*T12B*	*T12C*	*T13A*
	Visual Assessment/Characterization:	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X
COC Parameters only (0-6" samples and/or DIOI)*	TCL VOCs (8260D/5035)	2	2	2	2				1					2			1			1			1	2	2			2
	PAH17 (8270E)																											
	TOC (9060A/Lloyd Kahn)																											
	COC Metals - Arsenic, copper, lead, mercury, nickel, zinc (6000-7000 series)																											
Full Suite of COCs (not defined on figures) (0-6" samples)	TCL VOCs (8260D/5035)								1								1			1			1					
	TCL SVOCs (8270E), expanded NOAA PAH 34 List																											
	PCBs (8082A)																											
	TAL Metals (6000-7000 series)																											
	Total Cyanide (9012B*)																											
	TOC (9060A/Lloyd Kahn)																											
	EPH (MassDEP MA-EPH)																											
Eco Risk Assessment Parameters (0-6" samples)	Ammonia (SM4500NH3)				1				1					1			1			1			1	1	1			1
	TOC (9060A/Lloyd Kahn)																											
	Sulfide (9030B)																											
	AVS/SEM (EPA Draft method)																											
	Grain Size (ASTM D422 - sieve & hydrometer)																											
Geotechnical Parameters (in clay)	Grain Size (ASTM D422 - sieve & hydrometer)			1			1				1									1								
	TOC (9060A/Lloyd Kahn)																											
	Atterberg Limits																											
	Shelby Tubes (if equip allows)																											
Benthic Toxicity (0-6")	28-day whole-sediment tox test w/ *Hyalella azteca*																			1								
Waste Characterization Parameters (0-4' or full depositional thickness if less)	TCL and TCLP VOCs (8260D)				1									1														
	TCL and TCLP SVOCs (8270E)																											
	TAL and TCLP Metals (6000-7000 series)																											
	Pesticides/Herbicides (8081B/8151A)																											
	PCBs (8082A)																											
	pH, ignitability, sulfide &cyanide reactivity																											
	Corrosivity																											
	Total Cyanide (9012B)																											
	Total petroleum hydrocarbons (8015D)																											
	Percent sulfur (ASTM D4239)																											
	BTU (ASTM D240-87)																											

Notes:
* DIOI = Depth interval of interest = the deeper boring depth selected based on work plan criteria (approximate depth 5 to 10 feet below top of sediments)

Table 4. Sediment Investigation Boring and Analytical Summary
GLLA Scajaquada Creek OU
Buffalo, New York

	Transect Boring ID:	*T13B*	*T14A*	*T14B*	*T14C*	*T15A*	*T15B*	*T16A*	*T16B*	*T17A*	*T17B*	*T18A*	*T18B*	*T19A*	*T19B*	*T20A*	*T21A*	*T21B*	*T22A*	*T22B*	*T23A*	*T23B*	*T24A*	*T24B*	*T25A*	*T25B*	*T26A*	*T26B*	*TOTAL SAMPLE COUNT*
	Visual Assessment/Characterization:	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	X	54
COC Parameters only (0-6" samples and/or DIOI)*	TCL VOCs (8260D/5035)	2	2		2	2	2	2	1	2			2	2	1		1					1	1			1	2	1	47
	PAH17 (8270E)																												
	TOC (9060A/Lloyd Kahn)																												
	COC Metals - Arsenic, copper, lead, mercury, nickel, zinc (6000-7000 series)																												
Full Suite of COCs (not defined on figures) (0-6" samples)	TCL VOCs (8260D/5035)								1						1		1					1	1			1		1	11
	TCL SVOCs (8270E), expanded NOAA PAH 34 List																												
	PCBs (8082A)																												
	TAL Metals (6000-7000 series)																												
	Total Cyanide (9012B*)																												
	TOC (9060A/Lloyd Kahn)																												
	EPH (MassDEP MA-EPH)																												
Eco Risk Assessment Parameters (0-6" samples)	Ammonia (SM4500NH3)	1	1		1	1	1	1	1	1			1	1	1		1					1	1			1	1	1	26
	TOC (9060A/Lloyd Kahn)																												
	Sulfide (9030B)																												
	AVS/SEM (EPA Draft method)																												
	Grain Size (ASTM D422 - sieve & hydrometer)																												
Geotechnical Parameters (in clay)	Grain Size (ASTM D422 - sieve & hydrometer)								1																		1	1	6
	TOC (9060A/Lloyd Kahn)																												
	Atterberg Limits																												
	Shelby Tubes (if equip allows)																												
Benthic Toxicity (0-6")	28-day whole-sediment tox test w/ *Hyalella azteca*						1																					1	3
Waste Characterization Parameters (0-4' or full depositional thickness if less)	TCL and TCLP VOCs (8260D)																				1								3
	TCL and TCLP SVOCs (8270E)																												
	TAL and TCLP Metals (6000-7000 series)																												
	Pesticides/Herbicides (8081B/8151A)																												
	PCBs (8082A)																												
	pH, ignitability, sulfide &cyanide reactivity																												
	Corrosivity																												
	Total Cyanide (9012B)																												
	Total petroleum hydrocarbons (8015D)																												
	Percent sulfur (ASTM D4239)																												
	BTU (ASTM D240-87)																												

Notes:
* DIOI = Depth interval of interest = the deeper boring depth selected based on work plan criteria (approximate depth 5 to 10 feet below top of sediments)

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Figure 2. Scajaquada Creek Operating Unit Area Layout

Figure 3. Scajaquada Creek and Historic Industrial Properties

Figure 4. Mapped Area-Wide Clay Surface and DNAPL

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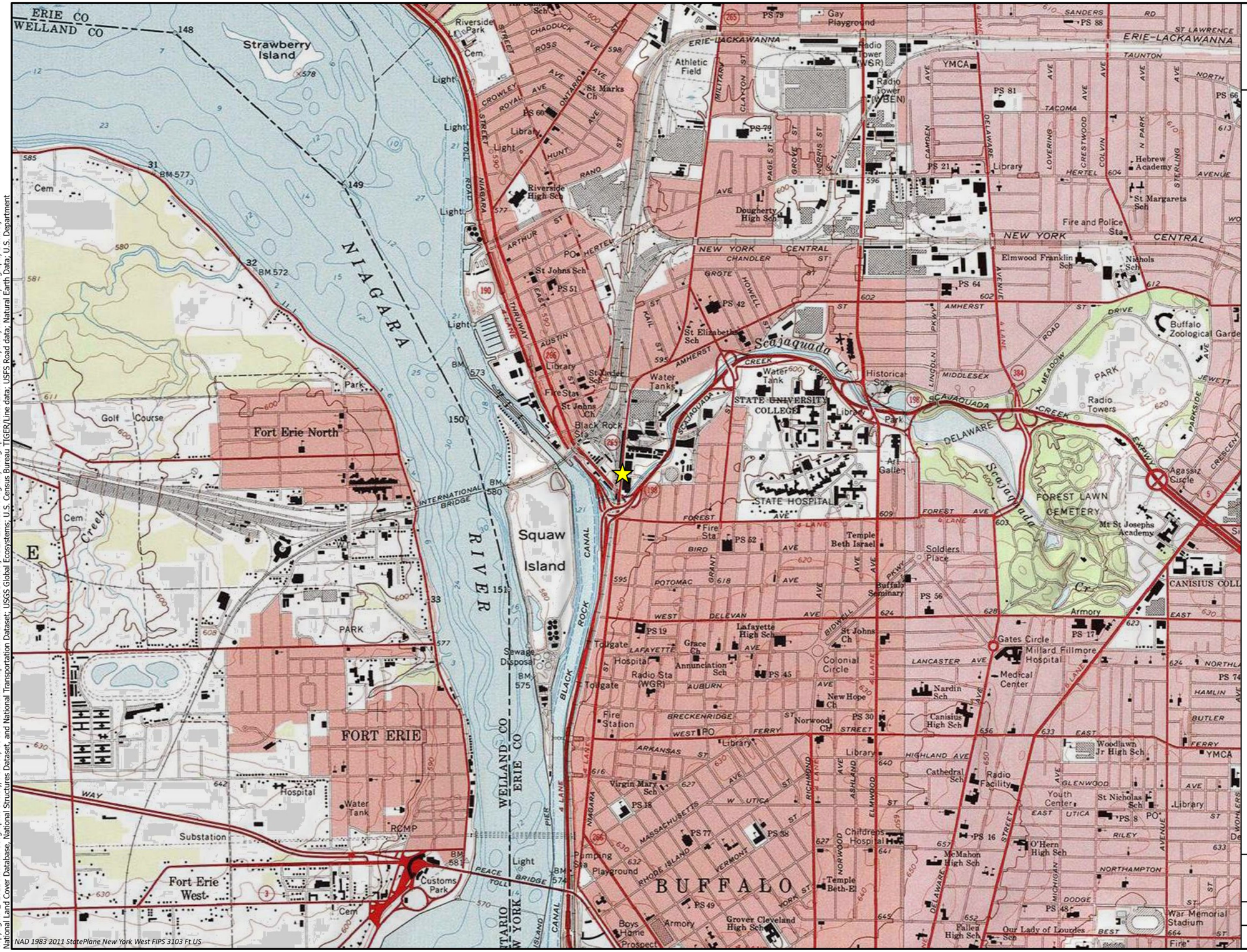
Figure 6. Landside Investigation Southern Area

Figure 7. Landside Investigation Northeastern Area

Figure 8. Creek Sediment Investigation Inlet Area

Figure 9. Creek Sediment Investigation Western End

Figure 10. Creek Sediment Investigation Northern End



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GENERAL SITE LOCATION

LEGEND:

★ General Site Location (Center)

N
W E
S

GEI Consultants

0 2,050 4,100 Feet

Lower Scajaquada Creek
Remedial Investigation

Buffalo, New York

Project 2406810 June 2025 Figure 1

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LOWER SCAJAQUADA CREEK OPERATING UNIT AREA LAYOUT

LEGEND:

- Elevation Contour (USGS)
- Black Rock Canal OU (Northern Portion, Approximate)
- Lower Scajaquada Creek OU (Approximate)
- County Tax Parcel

N
W E
S

GEI Consultants

0 250 500
Feet

Lower Scajaquada Creek
Remedial Investigation

Buffalo, New York

Project 2406810 June 2025 Figure 2

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USGS The National Map: National Boundaries Dataset, 3DEP Elevation Program, Geographic Names Information System, National Hydrography Dataset, National Land Cover Database, National Structures Dataset, and National Transportation Dataset; USGS Global Ecosystems; U.S. Census Bureau TIGER/Line data; USFS Road data; Natural Earth Data; U.S. Department of State HIU; NOAA National Centers for Environmental

NAD 1983 2011 StatePlane New York West FIPS 3103 Ft US

LOWER SCAJAQUADA CREEK AND HISTORIC INDUSTRIAL PROPERTIES

LEGEND:

- Elevation Contour (USGS)
- County Tax Parcel
- NYSDEC Listed Site or Historic Industrial Parcels w/Label

Notes:
BCP - NYSDEC Brownfield Cleanup Program
VCP - NYSDEC Former Voluntary Cleanup Program

N
W E
S

GEI Consultants

0 250 500 Feet

Lower Scajaquada Creek
Remedial Investigation

Buffalo, New York

Project 2406810 June 2025 Figure 3

USGS The National Map: National Boundaries Dataset, 3DEP Elevation Program, Geographic Names Information System, National Hydrography Dataset, National Land Cover Database, National Structures Dataset, and National Transportation Dataset; USGS Global Ecosystems; U.S. Census Bureau TIGER/Line data; USFS Road data; Natural Earth Data; U.S. Department of State; HIU; NOAA National Centers for Environmental

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MAPPED AREA WIDE CLAY SURFACE AND DNAPL

LEGEND:

- CPT-1 Design Boring

Boring Type w/Clay Elevation (famsl)

- 2021 GEI Sediment Boring
- Existing Monitoring Well
- 2021/2022 GEI Monitoring Well
- Boring w/DNAPL Observed or Suspected
- County Tax Parcel
- Clay Surface Elevation Contour (ftamsl)

Elevation Color Map (ftamsl)

580

545

N

W E

S

GEI Consultants

0 150 300

Feet

Lower Scajaquada Creek
Remedial Investigation

Buffalo, New York

Project 2406810 June 2025 Figure 4

USGS The National Map: National Boundaries Dataset, 3DEP Elevation Program, Geographic Names Information System, National Hydrography Dataset, National Transportation Dataset; USGS Global Ecosystems; U.S. Census Bureau TIGER/Line data; USFS Road data; Natural Earth Data; U.S. Department of State HIU; NOAA National Centers for Environmental

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LANDSIDE INVESTIGATION
NORTHERN & WESTERN AREA

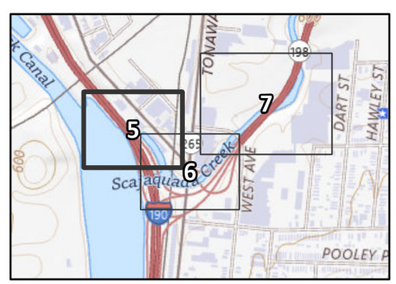
LEGEND:

- Proposed RI Monitoring Well
- Proposed Primary Soil Boring
- Proposed Contingency Soil Boring
- Existing Monitoring Well
- Monitoring Well (Destroyed)
- Sediment Boring (GEI Installed 2021)
- Monitoring Well (GEI Installed 2021/2022)
- Creek Edge of Water
- Creek Edge of Water (Georeferenced from 1936 Buffalo Sewer Authority Drawing)
- City of Buffalo Pump House
- County Tax Parcel

N
W E
S

GEI Consultants

0 75 150
Feet



Lower Scajaquada Creek
Remedial Investigation

Buffalo, New York

Project 2406810 June 2025 Figure 5

USGS The National Map: National Boundaries Dataset, 3DEP Elevation Program, Geographic Names Information System, National Hydrography Dataset, National Land Cover Database, National Structures Dataset, and National Transportation Dataset; USGS Global Ecosystems; U.S. Census Bureau TIGER/Line data; USFS Road data; Natural Earth Data; U.S. Department of State HIU; NOAA National Centers for Environmental

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LANDSIDE INVESTIGATION
SOUTHERN AREA

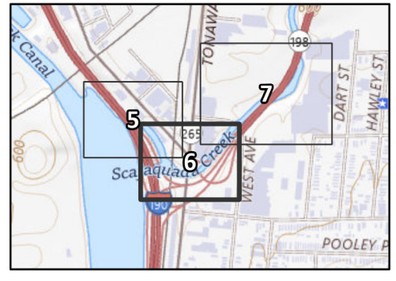
LEGEND:

- Proposed RI Sediment Boring
- Proposed RI Monitoring Well
- Proposed Primary Soil Boring
- Proposed Contingency Soil Boring
- Boring to be Completed Below Clay Layer
- Existing Monitoring Well
- Monitoring Well (Destroyed)
- Sediment Boring (GEI Installed 2021)
- Monitoring Well (GEI Installed 2021/2022)
- Creek Edge of Water
- Creek Edge of Water (Georeferenced from 1936 Buffalo Sewer Authority Drawing)
- City of Buffalo Pump House
- County Tax Parcel

N
W E
S

GEI Consultants

0 75 150
Feet



Lower Scajaquada Creek
Remedial Investigation

Buffalo, New York

Project 2406810 June 2025 Figure 6

USGS The National Map: National Boundaries Dataset, 3DEP Elevation Program, Geographic Names Information System, National Hydrography Dataset, National Land Cover Database, National Structures Dataset, and National Transportation Dataset; USGS Global Ecosystems; U.S. Census Bureau TIGER/Line data; USFS Road data; Natural Earth data; U.S. Department of State HIU; NOAA National Centers for Environmental

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LANDSIDE INVESTIGATION
NORTHEASTERN AREA

North Vault

MW-106

Scajaquada Expy State Rte 198

Scajaquada Creek

CPT-1

MW-103

57-71 MW-2

57-71 MW-3

SB-RI-19

MW-100

SB-RI-18

57-71 MW-1

Scajaquada Creek

State Rte 198 Scajaquada Expy

West Ave

31-MW-5

SB-RI-4

MW-RI-2

T26B

SED-LSC-8

T26A

MW-RI-1

SB-RI-1

South Vault

SB-RI-2

31-MW-4R

MW-RI-3

SB-RI-3

Fernwood Ave

Bradley St

Danforth St

NAD 1983 2011 StatePlane New York West FIPS 3103 Ft US

LEGEND:

- Proposed RI Sediment Boring
- Proposed RI Monitoring Well
- Proposed Primary Soil Boring
- Proposed Contingency Soil Boring
- Monitoring Well (Destroyed)
- Existing Monitoring Well
- Sediment Boring (GEI Installed 2021)
- Creek Edge of Water
- Sheet Pile
- Interceptor Trench
- CPT-1 Design Boring
- DNAPL Well Screen
- Sediment Cap Extent
- Historic Infrastructure
- County Tax Parcel

N

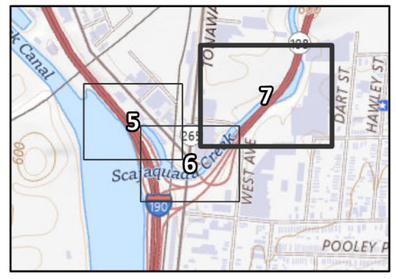
W E

S

GEI Consultants

0 50 100 200

Feet



Lower Scajaquada Creek
Remedial Investigation

Buffalo, New York

Project 2406810 June 2025 Figure 7

USGS The National Map: National Boundaries Dataset, 3DEP Elevation Program, Geographic Names Information System, National Hydrography Dataset, National Land Cover Database, National Structures Dataset, and National Transportation Dataset; USGS Global Ecosystems; U.S. Census Bureau TIGER/Line data; USFS Road data; Natural Earth Data; U.S. Department of State HIU; NOAA National Centers for Environmental

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CREEK SEDIMENT INVESTIGATION
INLET AREA

LEGEND:

Sample Type

- Visually Inspect Boring for NAPL
- Analytical for COC Parameters or Full Suite per Table 4
- Analytical for COC Parameters or Full Suite per Table 4, plus Ecological Parameters
- Locations where additional geotechnical data will be collected
- Locations where Benthic Toxicity Samples will be Collected
- Locations where Waste Characterization Samples will be Collected
- Surface Soil Sample for Sheen Residue Assessment
- 2017 Sediment Boring
- 2021 GEI Sediment Boring
- Creek Edge of Water
- Creek Edge of Water (Georeferenced from 1936 Buffalo Sewer Authority Drawing)
- City of Buffalo Pump House
- County Tax Parcel

N W E S

GEI Consultants

0 25 50 100 Feet

Lower Scajaquada Creek
Remedial Investigation

Buffalo, New York

Project 2406810 June 2025 Figure 8

T1 T1A T1B SCJ-01 T2 T2A T2B T2C SCJ-02 T3B SED-LSC-1 T3 T3A SCJ-03 T4B T4A T4 T5B SED-LSC-2 T5 T5A SCJ-04 T6 T6A T6B SCJ-05 T7 T7A T7B T8 T8A T8B Pump House SCJ-06 SED-LSC-3 T9 T9A T9B SCJ-07 T12 T12A T12B SCJ-08 T12C T11 T11B T13B SCJ-10 SED-LSC-4 T13A T14C T10

I-190 (NY Thruway)

State Rte 266 Niagara St

State Rte 265 Tonawanda St

CSX RR

Scajaquada Creek

NAD 1983 2011 StatePlane New York West FIPS 3103 Ft US

USGS The National Map: National Boundaries Dataset, 3DEP Elevation Program, Geographic Names Information System, National Hydrography Dataset, National Transportation Dataset; USGS Global Ecosystems; U.S. Census Bureau TIGER/Line data; USFS Road data; Natural Earth Data; U.S. Department of State HIU; NOAA National Centers for Environmental

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CREEK SEDIMENT INVESTIGATION
WESTERN END

LEGEND:

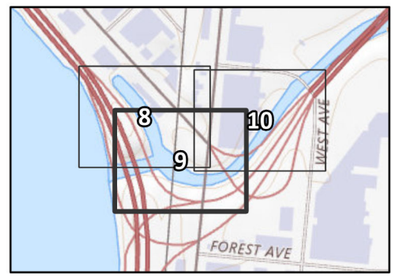
Sample Type

- Visually Inspect Boring for NAPL
- Analytical for COC Parameters or Full Suite per Table 4, plus Ecological Parameters
- Locations where additional geotechnical data will be collected
- Locations where Benthic Toxicity Samples will be Collected
- Locations where Waste Characterization Samples will be Collected
- Surface Soil Sample for Sheen Residue Assessment
- 2017 Sediment Boring
- 2021 GEI Sediment Boring
- Outfall
- Creek Edge of Water
- Creek Edge of Water (Georeferenced from 1936 Buffalo Sewer Authority Drawing)
- City of Buffalo Pump House
- County Tax Parcel

N W E S

GEI Consultants

0 25 50 100 Feet



Lower Scajaquada Creek
Remedial Investigation

Buffalo, New York

Project 2406810 June 2025 Figure 9

NAD 1983 2011 StatePlane New York West FIPS 3103 Ft US

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CREEK SEDIMENT INVESTIGATION
NORTHERN END

LEGEND:

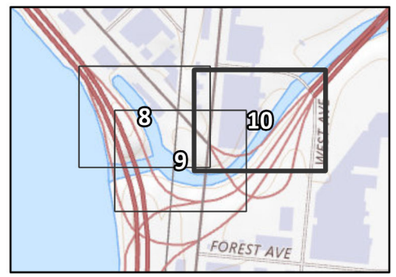
Sample Type

- Visually Inspect Boring for NAPL
- Analytical for COC Parameters or Full Suite per Table 4, plus Ecological Parameters
- Locations where additional geotechnical data will be collected
- Locations where Benthic Toxicity Samples will be Collected
- Locations where Waste Characterization Samples will be Collected
- 2017 Sediment Boring
- 2021 GEI Sediment Boring
- Outfall
- Creek Edge of Water
- County Tax Parcel

N W E S

GEI Consultants

0 25 50 100 Feet



Lower Scajaquada Creek
Remedial Investigation

Buffalo, New York

Project 2406810 June 2025 Figure 10

USGS The National Map: National Boundaries Dataset, 3DEP Elevation Program, Geographic Names Information System, National Hydrography Dataset, National Land Cover Database, National Structures Dataset, and National Transportation Dataset; USGS Global Ecosystems; U.S. Census Bureau TIGER/Line data; USFS Road data; Natural Earth Data; U.S. Department of State HIU; NOAA National Centers for Environmental

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Appendix A Site Characterization Report Former Buffalo Gaslight Iroquois Gas Corp. Site and Lower Scajaquada Creek Buffalo, New York NYSDEC Site #915351 (Text/Figures/Tables and Boring Logs)

GEI Consultants

Consulting
Engineers and
Scientists

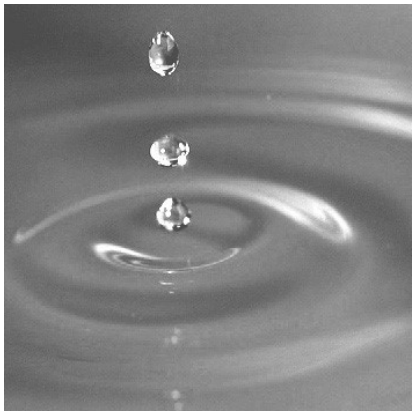
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
and Lower Scajaquada Creek
Buffalo, New York

NYSDEC Site #915351

Submitted to:
National Fuel Gas Distribution Corporation
6363 Main Street
Williamsville, NY 14221

Submitted by:
GEI Consultants, Inc., P.C.
100 Sylvan Parkway, Suite 400
Amherst, NY 14228

June 2022, Revised October 2022
GEI Project 2101620



Richard H. Frappa, P.G.
Senior Consultant/Hydrogeologist

Michael Cummings, P.G.
Project Hydrogeologist

Certification

I, Richard H. Frappa, P.G., certify that I am currently a Qualified Environmental Professional as defined in 6 NYCRR Part 375, and that this Site Characterization Report was prepared in accordance with all applicable statutes and regulations, and in substantial conformance with the NYSDEC Division of Environmental Remediation (DER) Technical Guidance for Site Investigation and Remediation (DER-10).

Richard H. Frappa, P.G.
Senior Consultant

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Abbreviations and Acronyms

AOC	Areas of Concern
ASP	Analytical Services Protocol
AST	Above-Ground Storage Tank
ASTM	ASTM International (formerly American Society for Testing and Materials)
bgs	Below ground surface
BTEX	Benzene, Toluene, Ethylbenzene, Xylene
CAMP	Community Air Monitoring Plan
COC(s)	Constituent(s) of Concern
DER	NYSDEC Division of Environmental Remediation
DNAPL	Dense Non-Aqueous Phase Liquid
DUSR	Data Usability Summary Report
EDR	Environmental Data Resources
EIMS	Environmental Information Management System
EPA	United States Environmental Protection Agency
FOIL	Freedom of Information Law
FSAP	Field Sampling and Analytical Plan
GEI	GEI Consultants, Inc., P.C.
HASP	Health and Safety Plan
HSA	Hollow-Stem Auger
LNAPL	Light Non-Aqueous Phase Liquid
MAH	Monocyclic Aromatic Hydrocarbons
MGP	Manufactured Gas Plant
mg/kg	milligrams per kilogram
NAD83	North American Horizontal Datum 1983
NAPL	Non-Aqueous Phase Liquid
NAVD88	North American Vertical Datum 1988
NYS	New York State
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
NYSDOT	New York State Department of Transportation
NYSTA	New York State Thruway Authority
PAH	Polycyclic Aromatic Hydrocarbon
PCE	Tetrachloroethylene
PID	Photo-ionization Detector
PPE	Personal Protection Equipment
ppm	Parts per million
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
REC	Recognized Environmental Condition
SC	Site Characterization
SCG	Standards, Criteria, and Guidance

Abbreviations and Acronyms (Continued)

SVOC	Semi-Volatile Organic Compound
TLM	Tar-Like Material
TAL	Target Analyte List
TCL	Target Compound List
TCE	Trichloroethylene
TOGS	Technical Operation Guidance Series
TPH	Total Petroleum Hydrocarbon
USGS	United States Geological Survey
UST	Underground Storage Tank
VOC	Volatile Organic Compound

1. Introduction

This Site Characterization (SC) Report has been prepared for National Fuel Gas Distribution Corporation (National Fuel) by GEI Consultants, Inc., P.C. (GEI) for property in the Black Rock area of Buffalo, New York adjacent to Scajaquada Creek. The SC Report describes investigation activities and findings for land formerly owned by a National Fuel predecessor company (Iroquois Gas Corporation), as well as the City of Buffalo, the United States of America, and the State of New York. The land was later developed for construction of the Niagara Section of the New York State (NYS) Thruway I-190 with on-ramp/off-ramp access to Route 198 (Scajaquada Expressway). The New York State Department of Conservation (NYSDEC) assigned the property a "Class P" (Potential listing) classification with Site Number 915351 and is referred to as the "P-Site" or "Site" in this report. In addition to the P-Site, investigation activities also included the lower portion of Scajaquada Creek and lands bordering the creek not owned by National Fuel. This area extends from the West Avenue bridge downstream to the creek outlet and is referred to as the Lower Scajaquada Creek Area.

The scope of SC activities was presented in the Site Characterization Work Plan (Work Plan) (GEI, August 2021). One objective of the investigation was to assess the potential for historical releases at the P-Site and whether or not the Site poses a potential threat to public health or the environment such that further investigation pursuant to NYSDEC DER-10 guidelines may be warranted. A second objective of the investigation was to characterize observations of suspected coal tar previously identified in the Lower Scajaquada Creek Area derived from historic manufactured gas plant (MGP) operations at the Iroquois Gas/Westwood Pharmaceutical Terrestrial Site. The requirement to investigate the P-Site and the Lower Scajaquada Creek Area is specified in an Order on Consent (Order or OC) between National Fuel and the NYSDEC that was signed by the NYSDEC Director of Environmental Remediation on June 23, 2021.

This SC Report summarizes the scope and preliminarily evaluates the resulting data associated with the completed SC activities, which were performed between October 2021 and April 2022. This report is organized as follows:

- Section 2 – Site Description and History – Presents a description of the P-Site and the Lower Scajaquada Creek Area (Figures 1 and 2), Site historical information, current Site use, sites bordering the Lower Scajaquada Creek Area and the creek itself having Recognized Environmental Conditions (RECs) as reported by others for these areas are also identified.
- Section 3 – Summary of Site Characterization Activities – Describes the scope of work, investigation methods, and deviations from the SC Work Plan.

- Section 4 – Site Characterization Findings and Assessment – Describes the investigation findings, preliminary site conceptual model, and the associated potential threat to public health and the environment.

- Section 5 – Conclusions and Recommendation – Summarizes SC conclusions and recommendations.

- Section – References – Provides references to documents cited in this report.

Various tables, figures, and appendices are also included and referenced within the text where appropriate.

2. Site Description and History

This section describes the P-Site and the Lower Scajaquada Creek Area, provides historical information and current property use, and identifies and summarizes properties in the Lower Scajaquada Creek Area with Recognized Environmental Conditions (RECs). Figures 1, 2, and 3 show various site locations and features referenced in the following text.

2.1 Site Description and Use

The P-Site property is a 2.6-acre area located between the Black Rock Channel and the Scajaquada Creek in the Black Rock section of the City of Buffalo, Erie County, New York (Figure 1). The Site does not have a street address, but the location of the former gas holder was located central to the following coordinates: latitude 42 deg 55'45.30" North and longitude 78 deg 53'56.12" West. Figure 2 is an expanded view of the area designated in this report as the Lower Scajaquada Creek Area.

The P-Site property has been developed for vehicular traffic on the Niagara Section of the NYS Thruway I-190, along with elevated on- and off- ramps for the NYS Route 198 Scajaquada Expressway, a City of Buffalo walking/biking trail, and contains an underground wastewater sewer conveyance system operated by the Buffalo Sewer Authority (BSA) known as the BSA interceptor sewer. During construction of the I-190, the property was substantially modified where the natural Scajaquada Creek outlet was filled (creating the Scajaquada Creek Inlet shown on Figure 2), and the creek outlet was relocated by excavating a channel through the property at its current location. Perimeter fencing extends along both sides of the NYS Thruway corridor.

No definitive plans for change of property use by the Niagara Section of the NYS Thruway or the Scajaquada Expressway near the Site or the Lower Scajaquada Creek area have been publicized by the NYS Department of Transportation (NYSDOT). In 2017, the NYSDOT completed a Final Design Report / Final Environmental Impact Statement / Final 4(f) Evaluation for a 2.2-mile segment of the Scajaquada Expressway corridor between the Grant Street interchange to the intersection with Parkside Avenue in the City of Buffalo (NYSDOT, November 2017), herein referred to as the NYSDOT Scajaquada Expressway 2017 Corridor Design/FEIS or NYSDOT Scajaquada project report. While outside the NYSDOT's project development area, the FEIS included a summary of environmental information along the full length of the Scajaquada Expressway corridor from the NYS Thruway I-190/Scajaquada Express exchange (area of the P-Site) to the Grant Street exchange. This summary presents pertinent information including RECs at the P-Site as well as at nearby properties. Following report completion and stakeholder review, NYSDOT Region 5 Director Frank Cirillo issued a brief (January 8, 2018) that stated *"... a consensus could not be reached with the many stakeholders involved. More than two-thirds of the most recent public comments received by*

NYSDOT were not in favor of the proposed Scajaquada Corridor project moving forward. As a result, NYSDOT will rescind work on the Environmental Impact Statement and not move forward with the project in its current form." Since that time, the Greater Buffalo Niagara Regional Transportation Council (GBNRTC) has resumed interest in modifications to the expressway corridor in the Olmsted Park area of expressway near Delaware Park approximately 1.5 miles northeast from the West Avenue bridge (Figure 1).

2.2 Site Ownership, Zoning, and Current Property Use

The City of Buffalo GIS property viewer (https://www.buffalony.gov/543/GIS-Maps) identifies the entire P-Site without a property identifier code indicating the property is currently owned by the State of New York. The NYSDEC provided National Fuel with historic plans (Drawing 301.0-8 Sheet 27 dated 3-21-1955 by the New York State Thruway Authority) that included record plans for constructing a portion of the Niagara Section of the NYS Thruway - Porter Avenue to Ontario Street Part 1. The plans identify the transfer of lands from various owners of the historic P-Site property including Iroquois Gas Corporation, the US Government, and the City of Buffalo to the State of New York. The 1955 drawings designed a new creek channel line as proposed by the US Army Corps of Engineers to re-route the Scajaquada Creek outlet by cutting an approximate 20-foot-deep channel directly across the area of the former gasholder on the NYS-acquired property. The historic natural Scajaquada Creek outlet was shown as being backfilled.

The P-Site property is currently utilized by the New York State Thruway Authority as a traffic corridor for the NYS Thruway System. A portion of the property includes a walking and biking trail used for recreational activity. The property is not zoned.

Properties adjacent to the creek in the Lower Scajaquada Creek Area are not owned by National Fuel but are owned by various public and private entities. Many of these properties have documented Recognize Environmental Conditions (RECs) including sites listed in the NYS Brownfields Cleanup Program (BCP), spill sites, and historic industrial properties. These sites are shown on Figure 3.

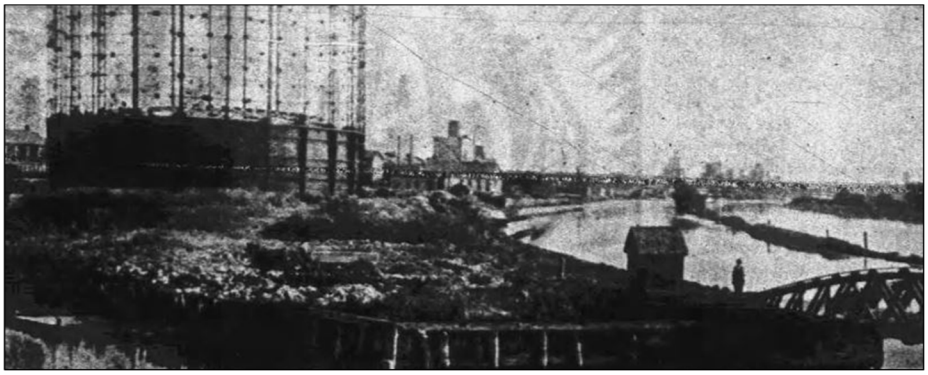
2.3 Historic Industrial Sites, BCP Sites, and Spill Sites

The P-Site property and nearby properties in the Black Rock Area near Scajaquada Creek have a long history of industrial use in the City of Buffalo from the mid-1800s though the mid to late 1900s. As shown in the 1927 aerial photograph below and information found in various environmental database searches and mapping records, industrial use of properties in the Lower Scajaquada/Black Rock area included: metal foundries, coal yards, rail lines, factories and manufacturing plants (i.e., paint and lacquer), gas manufacture and storage, steel works, and other industrial operations.

Photograph - *1927 aerial photograph showing the highly industrialized development of the Black Rock area (note original location of the Scajaquada Creek outlet to the Black Rock Channel and concrete foundation of gasholder at the P-Site property).*

2.3.1 P-Site History

The property was occupied by Shepard Hardware Company around 1883. The Shepard Hardware Company was a foundry that manufactured iron and brass household items, including ice cream freezers, iron piggybanks and other household goods. In May 1893, the foundry was destroyed by an intense fire that leveled the buildings on the property. Buffalo Gas Light Company purchased the property that comprised the Site in February 1896. In April 1896, Buffalo Gaslight Company began the construction of an above ground gasholder (see historic photo below).

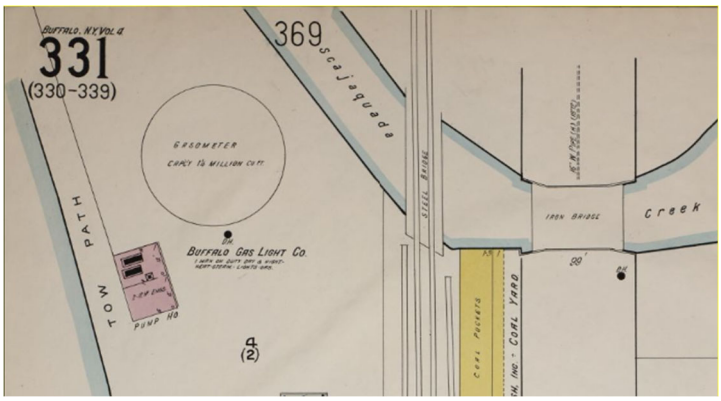


An article in the Buffalo Evening News (April 16, 1896) stated that,

> "*The tank will be situated between the Scajaquada Creek and the Erie Canal on the ground once occupied by the Shepard Hardware foundry. The gasometer will be the largest in the State outside of New York. The interior diameter of the great steel tank will be 132 feet, six*

> *inches and when full will rise to a height of 150 feet. The foundation will be of concrete and will be three feet below the surface. It will be constructed in three sections, which will telescope so that when empty it will be 50 feet high. The capacity of the tank will be 2,500,000 cubic feet of gas which is not under pressure. The gas will be manufactured at the present works and piped to Black Rock."*

As stated in the article above and supported by a review of aerial photographs, historical maps, and Sanborn Maps, gas was not manufactured on the property. Sanborn maps for 1900 and 1916 show a structure containing a pumphouse and boiler near the gasholder. A structure is present south of the P-Site and is not related to operations at the property.



From: Sanborn Map 1916

The gasholder remained on the property until no later than 1928 based on aerial photo review.

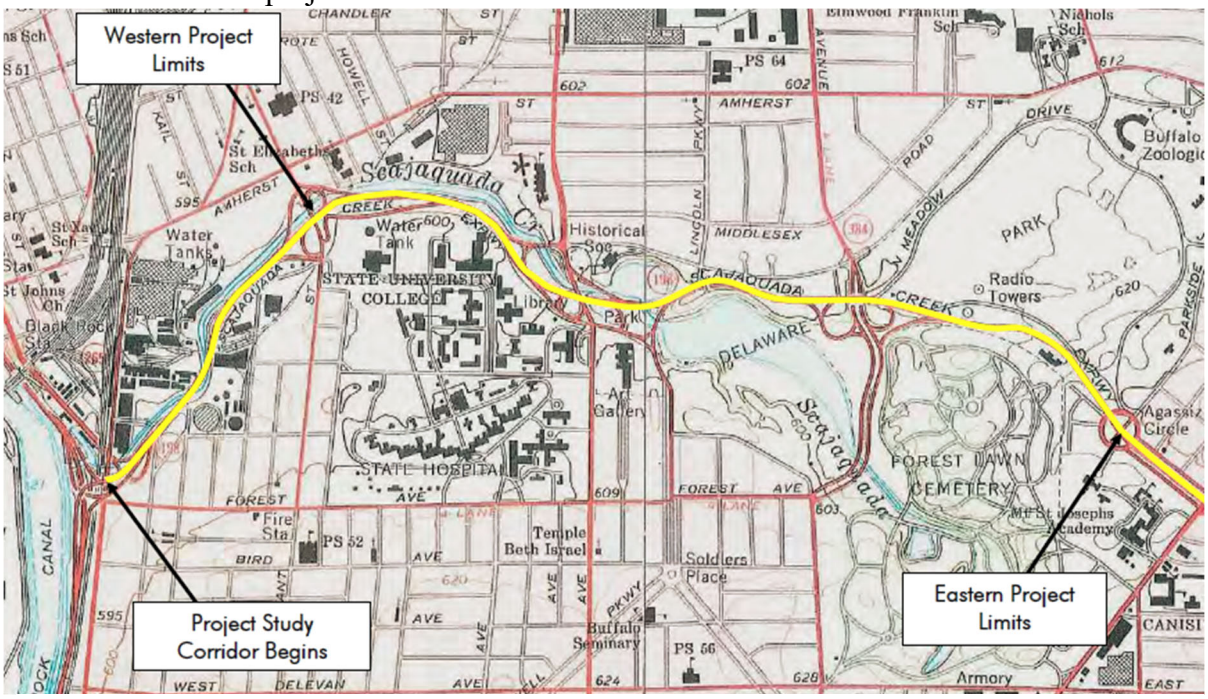
Subsequently, the property became unused until development for the Niagara Section of the NYS Thruway I-90 in the mid-1950s. NYS Thruway Authority design drawings were obtained and reviewed. The as-build drawings show complete excavation and removal of the concrete pad of the former gas holder as well as soil to a depth of 21 feet for relocation of the Scajaquada Creek outlet. Information related to backfilling the natural outlet of Scajaquada Creek has not been identified in the drawing packages reviewed.

Potential related Areas of Concern (AOCs) at the Site include its use as a holder for manufactured gas from an off-site source. Also, the location of the structure containing the pumphouse and boiler is considered an AOC for the property.

2.3.2 Industrial Sites, BCP Sites, and Spill Sites

As mentioned in Section 2.1, a redesign development project was contemplated for the Scajaquada Expressway. The scope of the project necessitated completion of a preliminary design and completion of environmental impact study. The NYSDOT Scajaquada Expressway 2017 Corridor Design/FEIS included the identification of properties with

potential environmental concern in the expressway corridor in the Lower Scajaquada Creek area. The map below identifies the corridor project study limits and Western and Eastern limit of the corridor project.



Map from NYSDOT, November 2017 Scajaquada Expressway 2017 Corridor Design/FEIS or NYSDOT Scajaquada project report NYSDOT Volume 8

Their report identified 25 properties posing an environmental concern along the Scajaquada Expressway corridor between the I-190 and the expressway's Grant Street exchange. The NYSDOT report included the property associated with the P-Site which was identified in the report as, "Site F – I-190 Section" and concluded the following, "Despite redevelopment, the use of this property as a foundry and a gas company with a large gas tank [*sic.* gasometer] indicates the potential for encountering soil contamination from petroleum products and the potential for abandoned USTs in the existing ROW."

During the past several decades, investigations and some remedial activities have occurred on properties in the area of the Lower Scajaquada Creek. Sites are shown on Figure 3 and are listed below with a brief description.

- Property at 1600 (also identified as 1654) Niagara Street (Spill number 1901060) – NYSDEC spill reported on April 30, 2019, at City of Buffalo Pump Station which pumps collected storm water from Niagara Street beneath the depressed section of the roadway beneath the CSX railroad bridge. A petroleum-type sheen was reported on surface water near the outfall discharge from the pump house to Scajaquada Creek. The NYSDEC spill inspector stated that he did not observe a sheen in the water being pumped from the pump house basement which collects stormwater from depressed

roadway below the railroad bridge. The spill report stated that, the rocks near the discharge "had distinct strong creosote odor and coated the rocks." The report also stated, "the coated rocks appear to be releasing petroleum sheen whenever water flows over them." The spill number was closed on May 8, 2019, and according to the Department, was referred to the NYSDEC's remedial section for follow-up work.

- Property at 1660 Niagara Street (BCP Site number C915311) – Historical use of the Site since the 1940s as gasoline sales and underground petroleum storage, vehicle repair and collision shop, and boat sales. Soil and groundwater testing during a Phase II environmental site assessment (ESA) identified contamination from historic property use and detected the following Constituents of Concern (COCs): petroleum VOCs and chlorinated VOCs (i.e., tetrachloroethylene [PCE] and degradation products), PAHs, arsenic, and lead.

- Property at 1626 Niagara Street (no NYSDEC Site No.) – The 2017 NYSDOT Scajaquada Corridor Study identified 1626 Niagara Street as a historic property used as a power station (circa 1912) and an area of concern for redevelopment. Two spills were identified in the EDR search and confirmed by review of NYSDEC spill database. The former on-site building (later used as a substation) was decommissioned during the 1990s.

- Former Pratt & Letchworth malleable iron and steel works, 189-205 Tonawanda St. (Site number 915045) – Operated as an iron steel works plant from the late 1890s through the mid-1900s and utilized "Fletcher Furnaces" for heating steel. Historic aerial photographs, Sanborn Maps, and the NYSDOT Scajaquada project report identified substantial petroleum bulk storage including a 500,000-gallon fuel oil tank installed in 1941; a 2,000-gallon gasoline underground storage tank installed in 1955; and a 30,000-gallon propane aboveground storage tank installed in 1977. A concrete oil tank house was constructed at this site in 1916. Four 20,000-gallon #6 fuel oil aboveground storage tanks (installation date unknown, removed 1995) were listed for this site. An investigation documented subsurface soil, surface soil, and surface water contamination on this site. Contaminants included PAHs, PCBs, phenolics, VOCs, and heavy metals. A petition to delist a portion of the site for the construction of a store and parking lot was submitted and approved. Some PCB contaminated soils have been removed from the site. Following site cleanup, the NYSDEC issued a "No Further Action" Record of Decision in 1995.

- Former Pratt & Lambert Paint and Varnish Plant, 75 Tonawanda St. (no site number) – The past use of the property as a paint and solvent manufacturer, as well as the past presence of over 100 petroleum and chemical storage tanks on the property, indicate the potential for soil and groundwater contamination from petroleum, chemicals,

solvents, and paint products to have been released from the site. Recorded spills and limited soil cleanup have been documented at the site.

- Former Pratt & Lambert Paint and Varnish Plant, 1451 West Avenue (no site number) – The past use of the property for lacquer manufacturing, as well as the past presence of bulk solvent and petroleum storage in above-ground and below-ground tanks on the property, represent potential sources of impact to Scajaquada Creek. Recorded spills and soil cleanups have been documented at the site.

- Property at 68 Tonawanda Street (NYSDEC Site No. C915316) – The property houses the historic New York Central Freight House and Office constructed in the early 1900s. The property is situated adjacent to railroad tracks operated by the New York Central railroad (currently CSX railroad). Railroads are RECs as they typically are associated with environmental impacts from operations which include use of creosote and diesel fuel. The results of a Phase II ESA (Panamerican Environmental, Inc./BE3, February 2017) indicate that semi-volatile organic compounds (SVOCs) (primarily PAHs) and metal compounds were detected throughout the Site at levels above residential and restricted residential soil cleanup objectives (SCOs). VOCs and PCB/Pesticides were detected but at concentrations below SCOs.

- Property at 57-71 Tonawanda Street (NYSDEC Site No. C915024) – The property was a lateral expansion of the Fedders Corporation situated at 31 Tonawanda Street. A portion of the site was listed as a Class 3 site on the NYSDEC's Registry of Inactive Hazardous Waste Disposal Sites in New York State. Soil and groundwater testing on the property identified the following COCs: chlorinated VOCs (TCE and degradation products), petroleum-related VOCs (BTEX), PAHs, PCBs, and heavy metals. The property is currently undergoing cleanup and redevelopment under the BCP.

- Property at 31 Tonawanda Street (NYSDEC Site No. C915299) – The original use of this property included brick making and sewer pipe making during the mid to late 1800s. In the early 1900s, the Fedders Corporation constructed a large manufacturing facility on the property. The Fedders complex historically used various chemicals, oils, solvents, and other materials in their manufacturing process. The processes at the property included metal stamping, soldering, brazing, welding, painting, acid washing and degreasing. Industrial wastes were reported to include solder dross, degreasing still bottoms including TCE and PCE, petroleum-based lubricating fluids and other products and wastes. Percent level concentrations of chlorinated VOCs (PCE and breakdown products) were identified in groundwater at the 31 Tonawanda Street property and property west of the site at 1660 Niagara Street.

- Property at 166 Dart Street (No site number) – Historical property involved in producing structural steel. According to a book by J. N. Larned (1911), Buffalo Structural Steel operated a steel forming facility with a capacity for producing about 8,000 tons of structural steel per year. Little else was found concerning history of the facility other than an expansion of the operation to the north. The property was acquired by the City of Buffalo after plant closure around 2010 and used as a salvage/impound lot. The past use of this property as a steel company and an automobile storage area indicates the potential for soil contamination from petroleum, chemicals, and solvents to exist on site. In addition, a portion of Scajaquada Creek was filled in on this site.

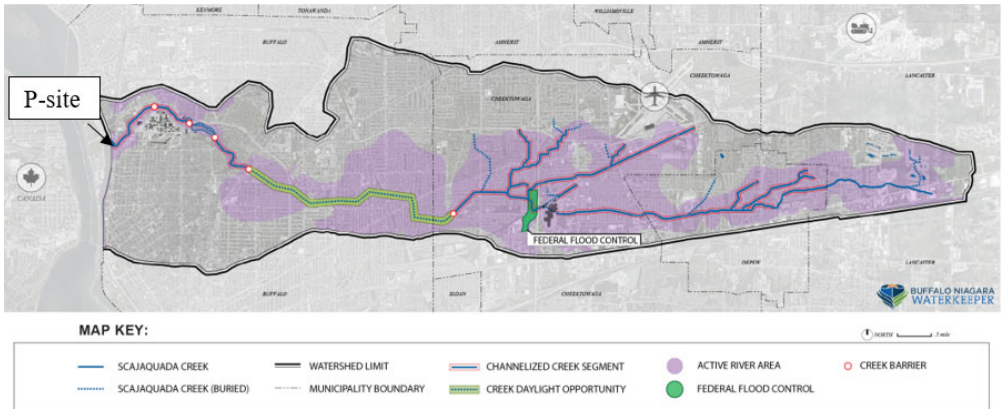
- Property at 120 Dart and 40 Bradley Street (NYSDEC Site No. #915141) – Property located at Dart and Bradley Street was formerly utilized as a manufactured gas plant and identified as the Iroquois Gas/Westwood Pharmaceutical Site. The property was development for manufactured gas generation about 1897 by the People's Gast Light and Coke Company. Feed stocks used in the gas manufacturing process were coke, oil, and coal. MGP operations reportedly ceased in 1955. Iroquois Gas continued to store natural gas at the site until 1972. In 1994, GeoTrans, Inc. completed a Remedial Investigation at the property. The RI characterized geology and distribution of COCs identified in soil, groundwater, and creek sediment. The RI identified benzene, toluene, ethylbenzene, and xylenes (BTEX) and PAHs in site media including the presence of non-aqueous phase liquid (NAPL) classified as coal tar. The remedies for the land side and creek side portions of the property were separated under NYSDEC consent decrees and identified as the Iroquois Gas/Westwood Pharmaceutical Terrestrial Site - #915141 and the Iroquois Gas/Westwood Pharmaceutical Riparian Site - #915141B. The Terrestrial Site remedy included in-situ containment with a sheet pile barrier wall placed between the land side and the creek and hydraulic control with groundwater extraction and treatment, and groundwater quality monitoring. In 2017, vertical groundwater collection wells were replaced by a horizontal groundwater collection trench placed upgradient of the sheet pile barrier to improve hydraulic control. The Riparian Site remedy included excavation and removal of contaminated sediments impacted by historic operations at the Terrestrial Site, capping of the creek bottom beginning approximately 50 feet east from the West Avenue Bridge and extending approximately 1,600 feet upstream to the abandoned New York Central Railroad bridge, placement of a vertical sheet pile barrier near the West Avenue bridge, long-term collection of DNAPL from coarse-grained deposits below the cap and creek sediments upgradient from the vertical barrier, and long-term monitoring of the sediment cap. The remedial measures were in place by 2000.

Documents with site information for many of the sites listed above are available on-line through the NYSDEC DECInfoLocator and https://www.dec.ny.gov/data/DecDocs/ by searching the NYSDEC Site Number. Boring logs/monitoring well construction details

from these investigations relevant to this study (as prepared by others) are included in Appendix B.1.

2.4 Description of Scajaquada Creek and Historic Sampling

The Scajaquada Creek watershed covers an urban area of 29 square miles. The source of Scajaquada Creek is located in the Town of Lancaster, approximately 18 miles east of the Black Rock Channel at its mouth. A map of the Scajaquada Creek Watershed is shown in the figure below.



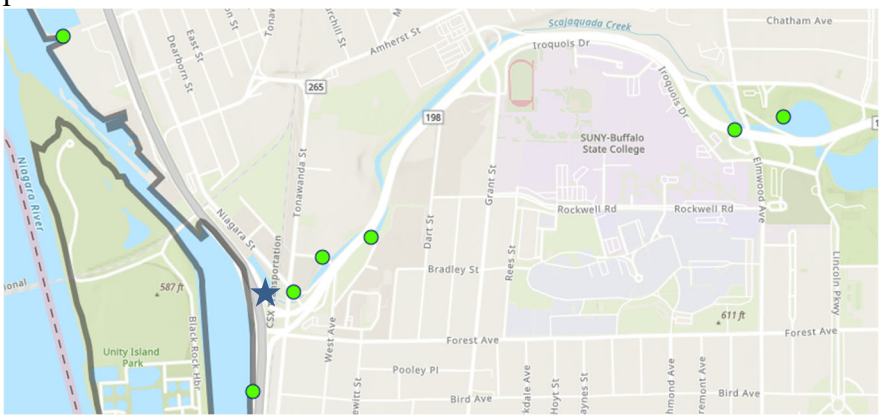
From: Buffalo Niagara Waterkeeper, https://bnwaterkeeper.org/scajaquada-creek/ and annotated by GEI with P-Site location

The course of the creek has been highly modified and channelized over its length. East of the Lower Scajaquada Creek Area, a portion of the creek is contained below urbanized areas of Buffalo in a three-mile-long tunnel, upstream of Forest Lawn Cemetery. Above the upstream entrance to the tunnel the creek runs through highly developed residential areas of the Towns of Cheektowaga and Lancaster. Downstream of the tunnel, the creek passes through Delaware Park, and then enters the former industrial area of Black Rock. The lower portion of the creek has historically been navigable for small boats which serviced industries along the creek. The mouth of the creek historically was used as a small port and supported naval vessels during the War of 1812 including the rehabilitation of watercraft. Prior to industrial development of the Black Rock area, the creek was wide, shallow, and meandering, surrounded by extensive marshes and meadows. Over the past two centuries, the Scajaquada Creek channel has been altered as urban and industrial development progressed in the City of Buffalo. Boat and barge traffic historically transported materials to and from the various industries located along the Lower Scajaquada Creek during the late 1800s and early 1900s. During that time, the creek was channelized and the wetland areas surrounding the creek were filled to accommodate industrial development. Boat slips lined the northern side of the creek prior to closure of the original creek mouth during re-routing of the creek to accommodate the development of the NYS Thruway Authority I-190 highway.

Scajaquada Creek flows toward the Black Rock Channel under normal weather conditions. However, storm and wind events can cause flow in the lower Scajaquada Creek area to

temporarily reverse direction as lake levels at the eastern end of Lake Erie rise producing higher water levels in the Niagara River resulting in a flow reversal in the lower portion of Scajaquada Creek. These lake seiche conditions are temporary and short duration, typically less than 48-hours, and occur more frequently during the late fall and early winter seasons in Western New York.

The NYSDEC stream classification for Scajaquada Creek is Class B-Fresh Surface Waters, Standards B, pursuant to 6 NYCRR Part 701. The best usages for Class B waters are for primary and secondary contact recreation and fishing. The water quality must be suitable for fish propagation and survival. Scajaquada Creek is on the Final New York State 2014 Section 303(d) List of Impaired Waters Requiring a Total Maximum Daily Load (TMDL). These are waters with verified impairments that are expected to be addressed by a segment/pollutant specific TMDL. Pollutants listed include floatables, dissolved oxygen/oxygen demand, phosphorus, PCBs, pesticides, heavy metals, and pathogens. The sources for these pollutants are listed as Combined Sewer Overflows (CSOs) and Urban Runoff. The map below from the Buffalo Sewer Authority (BSA) website (https://buffalosewer.org) shows permitted CSO discharge locations in the Lower Scajaquada Creek Area, areas upstream of the Westwood Riparian Site and the Black Rock Channel within the mapped limits shown below.



- Combined Sewer Overflow discharge locations near the P-Site
- P-Site location

In addition to the direct discharge of pollutants at CSOs, historic activities at industrial properties situated along the creek have impaired sediment quality with debris such as foundry sands and household trash, as well as COCs such as VOCs, PAHs, PCBs, pesticides, and heavy metals (see Section 2.3).

The USEPA designated the Niagara River as a binational Area of Concern (AOC) in 1987 because past industrial discharges and hazardous waste sites caused extensive degradation of aquatic habitats, particularly the degradation of the benthic macroinvertebrate community. In an unrelated study in 1993, the NYSDEC collected sediment samples at eight transect locations in the Lower Scajaquada Creek area to preliminarily assess the impact to sediments near historic industrial properties between Grant Street and the Black Rock Channel (NYSDEC, 1993). The sediment sample collected and analyzed farthest upstream (east of

Grant NS- No sample, ND – None Street) detected a total base neutral acid extractable concentration (BNA) of 191.4 mg/kg. BNA concentrations in sediments downstream opposite industrial properties (shown on Figure 3 and discussed in Section 2.5.3) ranged between 2.76 mg/kg and 580.4 mg/kg with the highest concentration occurrence near the Dart and Bradley site (pre-remediation). BNA concentrations in samples farther downstream (upstream side of the CSX steel railroad bridge) ranged from 14.2 mg/kg to 169.3 mg/kg. At all sample locations, PAHs had the highest BNA constituent concentrations. Pesticides and PCBs were detected at nearly all sample locations with no clear pattern of distribution. PCB concentrations ranged from non-detect to 4.2 mg/kg with the highest concentration detected upstream of the Dart and Bradley property. Arsenic, cadmium, lead, and mercury were elevated at most sample locations.

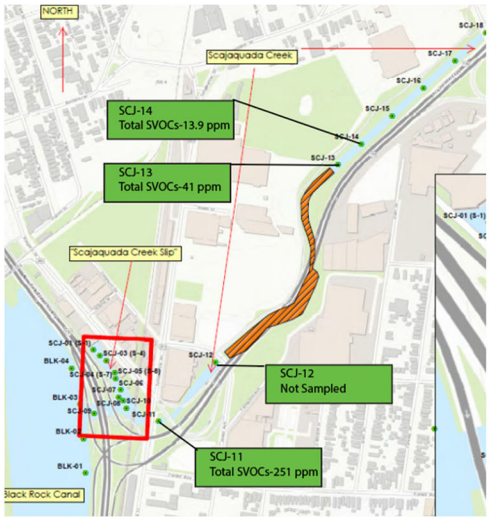
Preliminary studies conducted by the USEPA/USACE in 2014 and 2015 assessed sediment quality in tributaries to the Niagara River. Sediment testing in the Lower Scajaquada Creek Inlet (near the former boat slip area) east of the P-Site indicated sediment toxicity associated with contaminated bed sediments (USGS, 2016). Subsequently, contractors to the NYSDEC conducted additional sediment sampling in Scajaquada Creek primarily in the closed, dead-end portion of the former outlet of Scajaquada Creek (NYSDEC/Empire GEO Services, February 2015). The adjacent aerial photograph shows these sediment sampling locations. Sample results for the 2015 NYSDEC Scajaquada Creek Slip Area (Inlet area) sediment sampling are summarized below.

SAMPLE ID	Total SVOCs (ppm)	TOTAL PAHS (ppm)	Total VOCs (ppm)	Total CVOCs (ppm)	Total PCBs (ppm)	Petroleum Hydrocarbon Fingerprint (ppm)
S-1	959	610.5	27.4	ND	2.17	Fuel Oil #6- 10,000 / Motor Oil- 6,800
S-2	23,284	11,157	3,737	ND	NS	NS
S-3	199.7	109.7	0.16	ND	NS	NS
S-4	216.3	216.3	2.28	ND	NS	NS
S-5	555.7	500.6	0.64	ND	NS	NS
S-6	76.4	76.4	2.32	1.4	NS	NS
S-7	172.3	166.6	49.7	ND	9.01	Fuel Oil #6- 4,600 / Motor Oil- 3,900
S-8	156.7	156.7	75.5	ND	NS	NS
S-9	73.6	73.6	3.06	ND	NS	NS
S-10	137.3	137.3	1.18	ND	3	Fuel Oil #6- 5,300 / Motor Oil- 4,800

ND – Not detected; NS – No sample

Testing indicated the presence of PAHs, petroleum and chlorinated VOCs, and PCBs in shallow creek sediments. The TPH method fingerprints suggested low- to mid-distillate petroleum source material.

In 2017, additional sediment sampling was conducted in Scajaquada Creek by the USACE and the NYSDEC. These studies included sample collection and analysis of sediments from the eastern side of the Black Rock Channel near the P-Site and areas upstream from the mouth to Elmwood Avenue (NYSDEC/Empire GEO Services, June 2017). A portion of the data are summarized below.



Scajaquada Creek 2017 Sediment Sample Summary

SAMPLE ID	Total SVOCs (ppm)	TOTAL PAHS (ppm)	Total VOCs (ppm)	Total CVOCs (ppm)	Total PCBs (ppm)
SCJ-11	251	130	NS	NS	0.003
SCJ-13	41	41	NS	NS	0.99
SCJ-14	13.9	13.5	NS	NS	0.176

NS-Not Sampled

Data excerpt from NYSDEC/Empire GEO Services, 2017 and annotated by GEI.

Sediment sample data collected in 2017 from Scajaquada Creek identified PAHs, pesticides/PCBs, and heavy metals in Scajaquada Creek extending from the mouth of the creek upstream to sample location east of Grant Street (upstream from the mapped area shown in the figure above). The highest concentrations of COCs detected during the study were found in the slip area of the creek which is consistent with 2015 NYSDEC/Empire GEO Services. The elevated COC concentrations in the slip are most likely the result of stagnated flow conditions caused by the closure of the creek's natural outlet and re-routing of the creek channel to accommodate the 1950s construction of the Niagara Section of the NYS Thruway, thus allowing for the accumulation of sediments in this portion of the creek.

2.5 Previous Testing and Observations

Known site conditions prior to implementing the SC Work Plan included the detection of COCs in Scajaquada Creek sediments derived from:

- historic and current activities at properties in the Lower Scajaquada Creek (see Section 2.3);
- historic and active discharges from combined sewer overflows (CSOs); and

- point source and nonpoint source discharges from properties farther upstream within the Scajaquada Creek watershed including other outfalls discharging to the creek not described in this report.

In 2020, the NYSDEC investigated deeper soil adjacent to Scajaquada Creek. The investigation included the completion of soil borings and monitoring wells on properties located at 1660 and 1675 Niagara Street and later along the walking/biking trail on the west side of the Iroquois Gas/Westwood Pharmaceutical Site Riparian Site (see Figures 2 and 3). The NYSDEC investigation occurred after the NYSDEC's finding of "coated rocks with a distinct strong creosote odor" associated with Spill number 1901060 at 1654 Niagara Street associated with the discharge from the vault of the City of Buffalo Pump Station adjacent to the CSX Railroad bridge.

The NYSDEC provided NFG with documents and field records indicating the presence of COCs in soil and groundwater, as well as NAPL suggestive of coal tar. Suspected coal tar-like DNAPL was reported at wells MW-5R, MW-7, and MW-8R installed at 1660 Niagara Street. In addition to elevated PAH concentrations, analytical data provided by the NYSDEC for well MW-7 identified percent levels of chlorinated VOCs (TCE and PCE) suggestive of a mixture of product sources. Petroleum odors were reported at MW-3R installed at 1660 Niagara Street.

Where DNAPL was identified, the NYSDEC installed wells to depths greater than 25 feet below grade having well screens that extended deep enough to fully penetrate coarser grain materials and encounter the underlying aerially extensive red-brown clay layer. Monitoring wells installed at 1660 Niagara Street parcel during a 2016 BCP Phase II investigation (Labella, 2016) reported the detection of petroleum and chlorinated solvent-related contamination. Wells installed during the BCP Phase II investigation were completed to depths less than 20 feet below grade. DNAPL was not detected in the two wells installed at 1675 Niagara Street located northeast of the 1660 Niagara Street property. DNAPL having high PAH content was identified in well MW-100. This well is situated upgradient from the vertical barrier placed on the downstream end of the Riparian Site remedy that includes NAPL collection at the South Vault and is considered to be within the area of remediation of the Iroquois Gas/Westwood Pharmaceutical Riparian Site. NAPL accumulation was not identified farther upgradient in wells MW-103 and MW106 located along the bicycle path (see Figure 8 and discussion in Section 4.2.2). Based on these findings, an AOC was identified in both shallow and deeper soils adjacent to Scajaquada Creek at 1660 Niagara Street parcel.

3. Summary of Site Characterization Activities

This section describes the activities performed during the SC. Each field activity performed during the investigation, grouped by field task or environmental media, is described in the following sections. The SC sample locations and monitoring well locations on nearby properties installed by others are shown on Figure 2.

Representatives of the NYSDEC were present to observe the majority of the field activities, including the boring/monitoring well installation, sediment boring completion and sample collection, monitoring well sampling, and the City of Buffalo Niagara Street Pump Station sampling. A photographic log documenting SC field investigations is included following the text of this report.

3.1 SC Work Plan Documents

The SC was performed according to the methods and procedures described in the Site Characterization Work Plan (August 2021), which was approved by the Department on October 5, 2021. Four companion documents were included as appendices to the Site Characterization Work Plan:

- **Field Sampling and Analytical Plan (FSAP)** – The Site Characterization sampling, unless otherwise noted in the sections below, were conducted in accordance with the methods described in the site-specific FSAP.
- **Quality Assurance Project Plan (QAPP)** – The QAPP identified the quality assurance objectives for the measurement data, the QA/QC procedures to be used in the field, the sample chain-of-custody methods to be used, and the analytical procedures to be followed.
- **Community Air Monitoring Plan (CAMP)** – A CAMP was prepared which was followed during all invasive fieldwork including soil borings and the well installations.
- **Site-Specific Health and Safety Plan (HASP)** – A site-specific HASP was prepared to describe health and safety risks and procedures for the site sampling personnel.

3.2 Property Access and Underground Utility Clearance Activities

The SC field activities were performed beginning in November 2021 and continued through April 2022. As National Fuel is not a current property owner for any of the investigation locations, property access agreements were required to gain entry to complete field work. The following property owners or State/Federal governmental agencies were contacted to obtain access agreements and/or work permits to complete the work:

Property	Current Owner
P-Site Parcel	NYSDOT/ NYS Thruway Authority
1660 Niagara Street	Buffalo Niagara Riverkeeper
Scajaquada Creek	U.S. Army Corp. of Engineers, City of Buffalo, NYS Office of General Services, NYS Dept. of State.
Parcel Underlying State Route 198 and I-190 interchange, southeast of Scajaquada Creek.	NYSDOT/ NYS Thruway Authority
1626 Niagara Street	Niagara Mohawk Power Corporation (National Grid)
Bicycle Path adjacent to the Riparian Site	City of Buffalo

An on-Site meeting was requested by GEI to all utility entities identified during the initial DigSafe call-in. GEI met on-Site with representatives from National Grid and City of Buffalo to review the locations of buried infrastructure ahead of intrusive activities. Other utilities were either clearly identified on the ground surface during the mark-out process or were identified as "clear" from respondents during the Dig Safely New York process.

Additionally, personnel from the Buffalo Sewer Authority (BSA) field-identified the location of a tunnel interceptor sanitary sewer system which traverses the P-Site in a general north-south direction at a depth of approximately 24 feet below the average surface water elevation of Scajaquada Creek. BSA outlined a "no dig zone" in the field such that any intrusive work on the P-Site would remain a safe distance from the sewer. The location of the no dig zone is identified on Figure 2. Soil borings and monitoring wells proposed to be completed in the no-dig zone as part of the SC investigation were omitted or relocated as described in Section 3.13.

Soil boring locations were cleared using a combination of hand excavation and air-knife methods to a depth of approximately 5 feet below ground surface (bgs) in areas where a high density of buried infrastructure was identified to ensure that shallow utilities were not damaged during drilling.

3.3 Geophysical Survey

A subsurface geophysical survey was performed on the P-Site on October 28, 2021, to evaluate the potential for buried relic structures (gas holder foundation, support buildings, piping, etc.) associated with historic site use. An electromagnetic survey was performed using a Geonics EM-61 unit along transects shown on Figure 2. The results of the electromagnetic survey did not

identify any subsurface anomalies suggestive of relic structures on the P-Site. Additionally, soil boring data from P-Site locations MW-BGL-1 and MW-BGL-3 indicated reworked native clay backfill at the surface across the P-Site. Because no buried anomalies were indicated during the initial EM survey, the ground penetrating radar survey was not performed, and test pits to identify underground structures were not necessary . Results of the geophysical survey are presented in Appendix A.

3.4 Subsurface Sampling

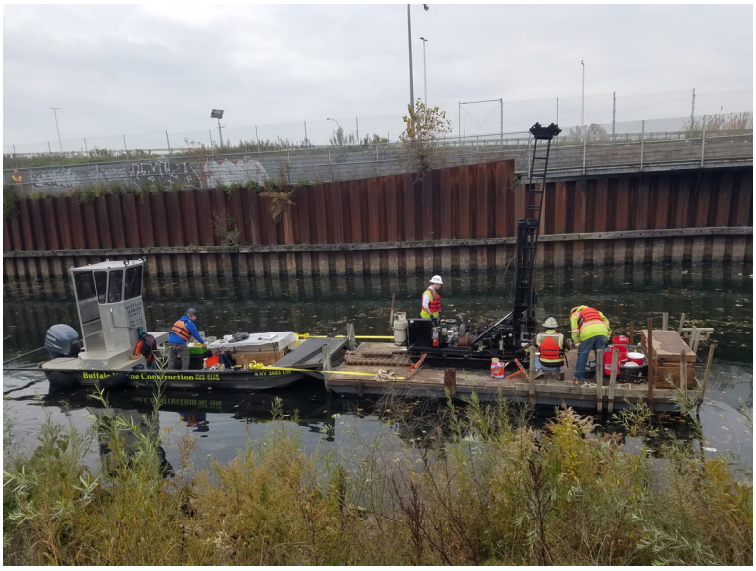
3.4.1 Soil Borings and Soil Sampling

As shown on Figure 2, a total of seven soil borings were completed and converted to monitoring wells during the Site Characterization. Soil samples from boring were submitted for laboratory analyses. Soil borings were completed between November 1 and November 11, 2021, and on January 4 and 5, 2022 (MW-LSC-5) following receipt of a property access agreement on the Niagara Mohawk property at 1626 Niagara Street. Two soil borings (MW-BGL-1 and MW-BGL-3) were completed on the P-Site Buffalo Gas Light (BGL) and five soil borings (MW-LSC-1, MW-LSC-2, MW-LSC-3, MW-LSC-4, and MW-LSC-5) were completed on parcels along the Lower Scajaquada Creek (LSC) corridor. Each boring was completed through fill and native soil into the native, low permeability red-brown (oxidized) clay exhibiting no impacts, consistent with methods outlined in the FSAP of the SC Work Plan. Soil boring logs are presented in Appendix B.2 which include sample collections depths from each boring. Sample collection methods were consistent with the FSAP of the SC Work Plan. A summary of soil samples and analytical methods is discussed in Section 3.12.

3.4.2 Sediment Sampling

Nine sediment borings (designated SED-BGL-1 and SED-LSC-1 through SED-LSC-8) were completed in Scajaquada Creek at locations shown on Figure 2 in accordance with methods described in the FSAP of the SC Work Plan. SJB Services, Inc. completed sediment borings SED-LSC-6, SED-LSC-7 and SED-LSC-8 during the week of November 1, 2021, using a pontoon barge and skid-mounted drill rig with support skiff that was manually launched immediately downstream of the West Ave. bridge. Manually launching the barge to directly access these boring locations was necessary due to height restrictions between the Creek surface and the Niagara Street Bridge and I-198 eastbound entrance ramp. Each of these borings was advanced using 2" diameter split spoon samplers driven by a 140 lb. hammer. Split-spoon sample depths were measured from the barge deck surface and the depth of water was recorded at each sampling location. Samples were

transferred from the pontoon barge to land for logging, sample collection and photo documentation.



The remaining sediment borings were completed west of the Niagara Street bridge by Nothnagle Drilling, Inc. with a support craft provided by Buffalo Marine Construction during the week of November 8, 2021. The barge was launched at a private marina on Niagara St. and shuttled down the Black Rock Canal to Scajaquada Creek. Each sediment boring was advanced using 4¼" hollow stem augers (Diedrich D25) and a dual-tube Macrocore® sampler advanced with an automatic hammer. Macrocore® sample depths were measured from the barge deck surface and the depth of water and deck height above water were recorded at each sampling location. Retrieved samples were transferred to the support craft for logging, sample collection and photo documentation.

At each location, the upper six inches of sediment was collected representing the modern transport and deposition system, and to perform laboratory chemical analyses to assess the sediment quality. Each sediment boring was advanced through historic alluvium and advanced several feet into the native red-brown clay unit. Sediment boring depths are summarized in Table 1B. Samples of deeper sediment/alluvium were collected from each boring to evaluate the quality of deeper deposits in the Creek channel. Samples collected for laboratory analysis were identified by the boring location and sample collection depth. Sample collection depths identified on deeper sediment sampling IDs were based on the barge deck surface measuring point (i.e., not depth below the top of sediment). Surface water elevations were measured throughout the sediment sampling process such that the elevation of each analytical sample could be calculated. Sediment sample analysis for shallow and deeper samples is identified in Section 3.12 and analytical results are discussed in Section 4. Sediment boring logs are provided in Appendix B.3. The location of each sediment boring was marked in the field and surveyed by Fisher Associates in March 2022.

3.5 Monitoring Well Installations and Development

A 2-inch diameter PVC monitoring well was constructed in each soil boring described in Section 3.4, consistent with procedures outlined in the FSAP of the SC Work Plan. Monitoring wells MW-LSC-1, MW-LSC-4, MW-LSC-5 and MW-BGL-1 were completed as flush-mount wells; MW-LSC-2, MW-LSC3 and MW-BGL-3 were completed with stick-up surface completions.

Table 1A summarizes monitoring well screen intervals and geologic material screened. Monitoring well construction logs are included as Appendix B.2. Each newly installed monitoring well was developed no sooner than 24 hours following installation, and consistent with procedures detailed in the FSAP of the SC Work Plan. Monitoring well development parameters are provided in Table 2. Monitoring well development water was collected and containerized as IDW and transported to the 1660 Niagara Street property for temporary storage. The location, ground surface elevation and top of casing elevation of each monitoring well was surveyed by Fisher Associates, Inc.

3.6 DNAPL Mobility Assessment and DNAPL Sampling

An assessment of DNAPL presence and mobility was performed on monitoring wells at the 1660 Niagara Street property (MW-5R, MW-7 and MW-8) and at MW-100 along the City of Buffalo Bike Path on October 26, 2021. At each location, DNAPL presence was evaluated using a weighted white PVC rod to measure NAPL thickness (DNAPL readily stains the surface of the white PVC clearly indicating DNAPL thickness). Where sufficient NAPL was present, accumulated DNAPL was removed using a peristaltic pump with down-hole tubing. NAPL was collected in a 5-gallon bucket and containerized as IDW and transported to 1660 Niagara Street for temporary storage. DNAPL recovery in each of the above wells was evaluated on November 12, 2021, again using a weighted PVC rod. At each location, DNAPL had recovered to pre-removal levels indicating a potentially high degree of mobility at each location. Table 3 identifies DNAPL presence in monitoring wells and measured thickness.

A representative DNAPL sample was collected from each monitoring well and submitted for forensic analysis by META Environmental (see Section 3.12). Additionally, DNAPL samples were collected from the North and South DNAPL collection vaults at the Scajaquada Riparian Site.

3.7 Groundwater Sampling and Water Level Gauging

Following well development, each newly installed SC monitoring well was sampled using low flow sampling methods (peristaltic pump and downhole tubing) in accordance with the FSAP contained in the SC Work Plan. Groundwater samples were collected for laboratory analyses as described in Section 3.12.

Groundwater measurements were collected from each newly installed monitoring well, existing wells on nearby properties, and at two Scajaquada Creek surface water measurement points during the investigation. Measurements were made jointly with Groundwater and Environmental Services, Inc. (GES), a contractor to the NYSDEC, who were previously collecting groundwater elevation measurements from existing wells in the Lower Scajaquada Creek area. Table 3 summarizes groundwater and surface water elevations collected prior to and during the investigation.

3.8 Creek Bank and Outfall Survey

Fisher Associates completed a survey of the creek bank from the northern end of the P-Site and the Inlet area of the creek to the West Avenue bridge (the Riparian Site). The survey was completed on March 15 and 16, 2022 and delineated accessible edges of the creek bank (edge of surface water), the presence of outfalls and headwalls, and structures associated with the City of Buffalo Pump Station. These features are indicated on Figure 2 and further discussed in Section 4.2.3. On March 15, 2022, only the City of Buffalo SPDES outfall near West Avenue was observed to be flowing. The outfall was flowing on all occasions when viewed, which included several dry weather conditions.

3.9 Pump Station and Vault

During field activities, sheens were observed on Scajaquada Creek during discharge from the City of Buffalo Pump Station located adjacent to the 1660 Niagara Street property. Subsequently, the NYSDEC spills group collected samples of water and sediment/debris from the subterranean vault below the Pump Station and a storm sewer manhole leading to the vault on November 17, 2021. Concurrent with NYSDEC sampling, GEI collected a sediment/debris sample from the Pump Station vault and a water sample from stormwater Manhole #23 directly upstream of the vault for laboratory analyses. Samples of debris containing tar-like material (TLM) were also collected from the Pump Station vault and Manhole #23 for forensic analysis by Meta Environmental. Analytical result for sediment/debris and water samples collected from the Pump Station are discussed in Section 4.4.

3.10 Investigation-Derived Waste Management

Investigation-Derived Waste (IDW) generated during the project included soil cuttings, sediment, groundwater, NAPL, decontamination water, decontamination materials and personal protective equipment. IDW was placed in 55-gallon DOT-approved drums and labeled with the contents (PPE, water, soil, etc.), quantity, date of generation, and generator name. An IDW staging area was established at the existing NYSDEC drum staging in the fenced portion of the property at 1660 Niagara Street following an agreement with the property owner.

3.11 Community Air Monitoring

Community air monitoring was performed using PIDs to provide real-time measurements of total VOCs and a Dust Track monitor to measure particulate (airborne dust) concentrations in air at the upwind (background) and downwind perimeter of each designated work area when intrusive investigation activities were performed. Action levels were not exceeded during intrusive investigation activities. CAMP data are provided in Appendix C.

3.12 Analytical Program

Samples of groundwater, shallow sediment, deeper sediment, soil, and debris from a manhole and pump house vault were collected during the investigation. Analyses of these media is described below.

3.12.1 SC Sample Collection

The laboratory samples collected, and the chemical analyses performed during the investigation are identified in Table 4. Constituents analyses were consistent with the work plan and DER-10 and included additional analyses for possible future assessment of toxicological impacts (i.e., NOAA 34 PAHs).

3.12.2 Laboratory Analysis and Data Usability Report

Investigation samples and associated QA/QC samples were collected and analyzed in accordance with the FSAP and QAPP of the SC Work Plan. Soil, sediment, groundwater, and debris samples were analyzed by Alpha Analytical. Forensic samples of suspected TLM-impacted soil and sediment and DNAPL in wells were analyzed by ESS Laboratories and the data were evaluated by META Environmental. The Alpha Analytical Category B ASP data package is provided in Appendix D.1 and the META Environmental/ESS Laboratories forensic report is provided in Appendix D.2. NOAA 34 PAHs results for shallow sediments and included in the Alpha Analytical data package are tabulated in Appendix D.3. A Data Usability Summary Report (DUSR) was prepared for soil, sediment, debris and groundwater samples in accordance with the QAPP of the SC Work Plan and is provided in Appendix E.

3.12.3 EIMS Data Submittal

Validated laboratory data were uploaded to a NYSDEC EQuIS EDD file and submitted through the EarthSoft NYSDEC EDP EDD Format Files Website.

3.13 Deviations from the Work Plan

Deviations from the work plan are summarized below:

- The EM geophysical survey performed on the P-Site did not identify any subsurface anomalies suggestive of relic structures or infrastructure (tanks, piping, etc.). As a result, the ground penetrating radar survey was not performed and subsequently no test pits were excavated.
- As discussed in Section 3.2, the staff from Buffalo Sewer Authority (BSA) identified the location of the BSA Interceptor Sanitary Sewer which in the subsurface, predominantly occupies the area between the I-190 northbound lane and the entrance ramp to the I-190 outbound entrance ramp from I-198 shown on Figure 2. Based on this information, proposed monitoring well MW-BGL-2 had to be omitted and proposed locations MW-

BGL-1 and SED-BGL-1 were modified to avoid this critical infrastructure. These revisions to the SC Work Plan field investigation were communicated in a letter to NYSDEC on November 10, 2021, and approved by NYSDEC in an e-mail that day. The revised locations of MW-BGL-1 and SED-BGL-1 were subsequently approved in the field by BSA personnel and cleared in the field by Dig Safely New York.

- The DNAPL presence and mobility assessment included gauging and removal of NAPL from monitoring wells on the 1660 Niagara Street property (MW-5R, MW-7 and MW-8) and at MW-100 along the City of Buffalo Bike Path. Following removal, NAPL levels in each well recovered to pre-removal levels following the initial two-week recovery period proposed in the SC Work Plan. DNAPL at upgradient and downgradient locations all exhibited similar high-mobility properties and were consistent with DNAPL observations in soil borings and monitoring wells installed during the SC. Subsequently, additional DNAPL mobility assessment was not performed on monitoring wells installed during the SC.
- The SC Work Plan proposed that two synoptic rounds of groundwater elevations would be collected following installation of the SC monitoring wells. A synoptic round of groundwater elevations in the Lower Scajaquada Creek well network was performed on November 23, 2021, by GES and GEI and is provided on Table 3. This included gauging accessible wells installed prior to the SC and wells installed-to-date as part of the SC which including MW-BGL-1, MW-BGL-3, MW-LSC-1, MW-LSC-2, MW-LSC-3 and MW-LSC-4. Well MW-LSC-5 was installed in January 2022 and was therefore not included in the November event. Subsequent gauging events by GES in January and March 2022 are considered substantially complete but some wells could not be accessed due to snow cover or locked access gates.
- The Work Plan called for analysis of shallow creek sediment samples for Extractable Petroleum Hydrocarbons (EPH) using Massachusetts-specified methods. The laboratory inadvertently utilized New Jersey EPH methods. Because the EPH analyses were for forensic purposes, are not associated with DER-10 or New York procedures, and the New Jersey method provides comparable data for the intended purposes, reanalysis by Massachusetts methods was not performed.
- All work activity associated with observations and sampling described in Section 3.9 related to the City of Buffalo Pump Station was outside the Work Plan scope.

4. Site Characterization Findings and Assessment

This section describes site characterization findings including geologic and hydrogeologic conditions and field observations during data collection. These field observations include discharges to Scajaquada Creek (one generating sheens), TLM in soil samples collected from borings, and DNAPL observed in wells. This section also describes constituents detected in media and provides a preliminary conceptual site model.

4.1 Geology and Hydrogeology

Intense development and industrialization of properties in the area of Scajaquada Creek has altered the natural setting of the P-Site and Lower Scajaquada Creek Area. As a result, the shallow geology has been highly disturbed. The ground surface elevation of the P-Site is approximately 573 feet North American Vertical Datum of 1988 (NAVD88) with higher elevations occurring closest to the northbound and southbound highways as engineered fill was placed during construction of the highway. The surrounding properties in the area of the Lower Scajaquada Creek are generally flat lying with ground surface elevations approximately 10 to 15 feet higher than the typical surface water elevation of Scajaquada Creek (typical elevations 570 to 573 ft NAVD88).

The general geology of the P-Site and the Lower Scajaquada Area consists of industrial fill material overlying an aerially extensive red-brown clay of lacustrine origin. Near Scajaquada Creek, alluvial deposits associated with historic or recently deposited sediments lie above the clay. Bedrock below the P-Site was identified in NYSDOT core logs as the Onondaga Limestone. A stratigraphic summary of soil and sediment encountered in soil and sediment borings is included in Table 1A and 1B, respectively. Geologic cross-sections showing strata encountered are provided on Figures 4A through 4F.

4.1.1 P-Site Geology

P-Site geology was characterized using five soil/rock boring logs completed by the NYSDOT in 1956 NYS Thruway I-190 on ramp/off ramp design for Route 198 (Scajaquada Expressway) (boring locations and logs are presented in Appendix B) as well as soil/sediment borings completed for the SC at MW-BGL-1, MW-BGL-3, and SED-BGL-1 (see Figure 2 and boring logs included in Appendix B). The boring logs indicate 8 to 20 feet of heterogenous fill material composed of either reworked red silty clay mixed with construction material (concrete and gravel) or black sand and gravel and mixed with fines and cinders and construction rubble. The fill at the P-Site directly overlies the lacustrine red-brown clay. The red-brown clay is greater than 50 feet thick below the P-Site. NYSDOT boring logs for Scajaquada Expressway design indicate limestone bedrock occurs at a depth of approximately 70 to 75 feet below the current Scajaquada Creek outlet. In some borings, a thin layer of sandy till (approximately 5 feet thick) was reportedly observed immediately above the bedrock (between the red-brown clay and

bedrock). Because the current Scajaquada Creek outlet was constructed during the mid-1950s by excavating through the fill into the upper lacustrine red-brown clay, and because this location likely has a higher scour velocity than the nearby Inlet area, less than 0.5 feet of recent fluvial fine-grain sediment was identified above the red-brown clay in sediment boring SED-BGL-1 in the creek channel at the P-Site.

4.1.2 Lower Scajaquada Creek Area Geology

The geology of Lower Scajaquada Creek area was characterized using five SC soil borings sampled for monitoring well installations (MW-LSC-1 through MW-LSC-5), and eight SC sediment borings (SED-LSC-1 through SED-LSC-8). Additionally, over a dozen soil borings/well installations completed by others to characterize the former industrial properties bordering Scajaquada Creek supported geologic interpretation (boring logs are included in Appendix B for boring/well locations shown on Figure 2). The SC boring logs identified a fill thickness ranging from 10 to 19.5 feet. The fill thickness was interpreted to decrease with distance from the current Scajaquada Creek channel as shown on cross-sections 4A-F based on soil boring data at nearby Brownfield sites. For example, wells completed at the former Fedders Corp Site at 57-71 Tonawanda Street report fill thickness of about 5 feet or less. Historic fill materials were variable in composition consisting of reworked red-brown clay soils and black silty sand with little gravel. The fill material contained anthropogenic materials including brown foundry sands, concrete, cinders, and wood debris.

At nearly all borings completed in the Lower Scajaquada Creek area, alluvial deposits were encountered below fill. The alluvial deposits became increasingly coarse grained with depth. These deposits were typically composed of dark gray to black silt or sand with occasional decayed woody materials. With depth, the alluvium transitioned to black silty sand and fine gravel with occasional wood pieces and shells from gastropods. The alluvium deposits are associated with historic deposition of creek sediments and are typically void of anthropogenic construction materials. The alluvium thickness ranged between 1 and 22.5 feet as encountered in borings. The thickest layer of alluvium was identified in borings completed at 1660, 1654, and 1626 Niagara Street as the historic creek channel scoured the red-brown clay during deposition of early alluvial sediments. The alluvium thickness diminishes with distance from the current creek channel as shown on geologic cross-sections (Figures 4A through 4F).

The bottom of the alluvium was observed as a sharp contact with the area-wide red-brown clay layer. With the exception of MW-LSC-1, all SC borings were advanced at least 5 feet into clay for verification. The composition of the red-brown clay was consistent at each of the borings. The surface elevation of the aerially extensive red-brown clay slopes toward the creek as shown in cross-sections and contour mapping shown on Figure 5. Figure 5 suggests the ancestral channel of Scajaquada Creek was situated farther north in comparison to its current channel (see additional discussion in 4.2.1).

Two soil borings were advanced to bedrock by GeoTrans during the RI at the Westwood Former MGP Terrestrial Site (1995) and encountered bedrock at elevations of 506 and 520 ft msl indicating the thickness of the red-brown clay is at least 50 feet thick throughout the Lower Scajaquada Creek Area.

Within the Scajaquada Creek channel, sediment borings identified a layer of recent fluvial deposits directly overlying red-brown clay or historic alluvial deposits. The recent fluvial deposits were described as black, soft silt with mixtures of sand and gravel containing anthropogenic materials such as glass, foundry sand, and wood with occasional zebra mussels. At sediment borings SED-LSC-1, -2,- 6, -7, and -8, historic alluvial deposits were identified having a similar composition to the alluvium described in SC soil borings below fill material when present. At sediment boring SED-BGL-1, less than six-inches of recent fluvial deposits were identified. The recent fluvial deposits occurred directly over the red-brown clay.

4.1.3 Area Hydrogeology

Groundwater in the area occurs under unconfined (water table) conditions in saturated fill and in alluvium. Groundwater and Scajaquada Creek surface water elevation data were recorded in monitoring wells by GES (contractor to the NYSDEC) prior to the SC and during the SC with GEI; gauging data are summarized in Table 3. Water level data indicate a direct correlation between surface elevations in the creek and groundwater elevations in monitoring wells as groundwater elevations respond to changes in surface water elevation. Figure 6 plots groundwater and creek surface water elevation data for the March 29, 2022, event which includes the SC monitoring wells and existing wells located on nearby Brownfield sites. The groundwater contour map on Figure 6 shows a convergent groundwater flow pattern toward the creek. A depressed area of groundwater elevations is observed in the area of Niagara Street with groundwater lows occurring near the City of Buffalo Pump Station. A possible cause of the depressed groundwater elevations is described in Section 4.4.2.

4.2 Field Observations

4.2.1 Scajaquada Creek Conditions, Bank Mapping, Historic Channel and Known Discharges

The outlet of the creek was modified during the mid-1950s during the construction of the NYS Thruway I-190 and the Scajaquada Creek Expressway (NY Route 198). The historic outlet of the creek was filled, and a new outlet created about 450 feet south by land excavation (directly across the P-Site). This created a shorter path for the creek to discharge to the Black Rock Channel and left a cut-off segment of the creek with no flow through it (currently identified as the Inlet).

There are no active stream gauges on Scajaquada Creek. Stream flow at the entrance to the tunnel was recorded by the USGS from March 1, 1957, to September 30, 1994 (gauging station

04216200). Flow rates ranges from a low of just under 0.2 cubic feet per second (cfs) to a high of nearly 900 cfs, with typical flows in the 2 to 70 cfs range. Data from 1958 to 1994 showed a maximum peak flow of about 2800 cfs.

A gauge that measures the water level is located in a storm sewer near the mouth of the creek, 50 feet east of Niagara Street and 115 feet south of the creek. The reported data show a relatively stable water level, with occasional brief rises of up to 2 feet, most likely due to minor seiche influences. Flow reversals in the lower portion of the creek occur due to seiches in Lake Erie, driven by sustained winds from the west that elevate the lake level at Buffalo and in the inlet to the Niagara River. Flow can also be slightly affected by the operations of the Black Rock Canal Lock, which is located 2,000 feet downstream of the mouth of the creek.

The edge of the water was mapped during the SC survey on March 16 and 17, 2022 for comparison to historic mapping of the Scajaquada Creek channel and is shown on Figure 2. Historic mapping of the Black Rock area shows the creek wider than its current configuration, as shown in the 1901 USGS topographic map shown below. This is consistent with extensive observations of fill in the area.

Figure above from USGS Topographic Map dated 1901.

Figure 7 is a more detailed historic map G.M. Hopkins Atlas Map from 1894 that is an overlay on the current aerial of the Lower Scajaquada Creek Area. Similar to the 1901 USGS topographic map, the creek is wider to the east and appears to have a structure to control flow at the original creek outlet. As described in Section 4.1.2, boring data indicate thicker alluvium on the west side of Niagara Street near the rail tracks crossing the creek (see Figure 5) and property at 1660 Niagara Street. These data indicate the historic outlet area of the creek (currently identified as the Inlet after 1950s construction of the NYS I-190 Thruway) was closer to the present-day location of Niagara Street.

4.2.2 TLM and DNAPL Observation

TLM[1] was identified in some coarser grain alluvial soil and sediment samples and DNAPL[2] was observed in some monitoring wells installed during NYSDEC site investigations and three monitoring wells installed during the Site Characterization. The following wells contained DNAPL: MW-LSC-2, MW-LSC-4, MW-LSC-5, 1660-MW-3R, 1660-MW-5R, 1660-MW-7, 1660-MW-8, and MW-100. TLM was described in many soil and sediment samples collected from deeper coarser-grain alluvial soils. Wells containing DNAPL with borings completed in soil/sediment having TLM are shown on Figure 8. As shown in Figure 8, the occurrence of DNAPL in wells and observation of TLM is confined to an area below the creek and land in close proximity to the current creek channel. It is recognized that some wells, particularly those at the north end of property at 1660 Niagara Street, were installed to monitor water quality in saturated fill and are of limited use for DNAPL monitoring. TLM and/or DNAPL potentially present within the underlying alluvial soils at these locations would not accumulate in wells penetrating only the fill materials. DNAPL may have been identified if those well locations had well screens installed to the top of clay in the alluvial soils.

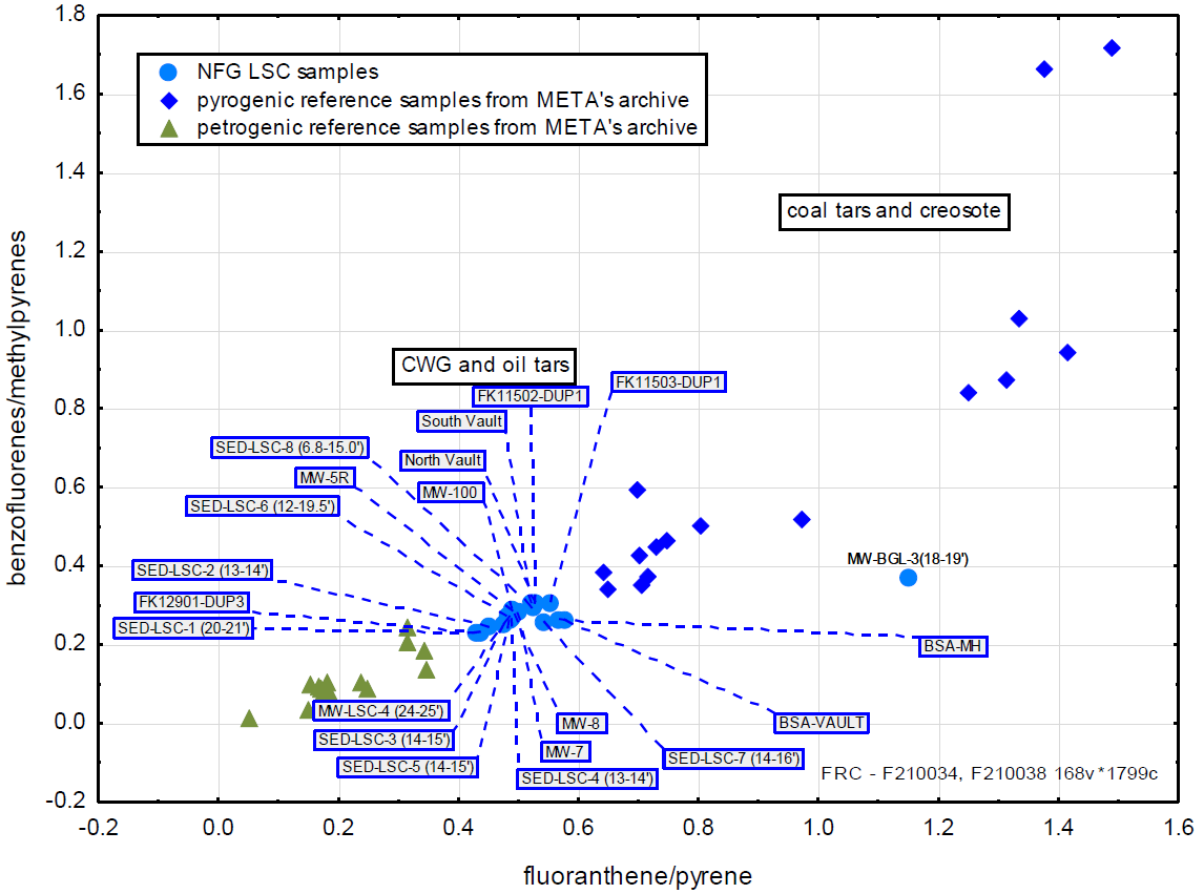
TLM and DNAPL, where present, were observed only in the deeper soils. Based on the absence of shallow TLM and DNAPL in any of the borings, it is apparent that TLM migrated laterally to areas where it was identified. The chemical composition of the TLM/DNAPL was evaluated and a preliminary assessment of potential mobility was performed.

4.2.2.1 Chemical Composition

As described in Section 3.12.2, sediment and soil samples that were visibly impacted with TLM were submitted for forensic analysis. In addition, samples of DNAPL in monitoring wells and the DNAPL recovery systems (South Vault and North Vault) at the Westwood Former MGP Riparian Site were also analyzed. Forensic analyses identified monocyclic aromatic hydrocarbons (MAHs), including benzene, toluene, ethylbenzene, and xylenes, polycyclic aromatic hydrocarbons (PAHs), and saturated hydrocarbons in each of the samples. The forensic report is included in Appendix D.2 and key findings are summarized below.

The graphic below from the forensic report is a double ratio plot that identifies META's pyrogenic reference samples for coal tars and creosote as well as carbureted water gas (CWG) and oil tars from known MGP source samples. The ratio plot also includes petrogenic (non-MGP related) reference samples for comparison of PAH ratios. Iroquois Gas/Westwood Pharmaceutical Former MGP source material samples collected are identified as North Vault and South Vault.

[1] TLM refers to tar-like material present in soil or sediment as observed in sample cores or excavation
[2] DNAPL refers to dense non-aqueous phase liquid that is observed on the bottom of a monitoring well that can accumulate if TLM is sufficiently mobile.



Forensic analysis of MAH and PAH chemical patterns of DNAPL samples analyzed from the North Vault and South Vault were reported to be similar in chemical composition and likely produced by a CWG or oil gas pyrogenic process. Similar chemical patterns were found in DNAPL samples collected from monitoring wells. TLM in soil and sediment samples contained mixtures of pyrogenic and petroleum-related compounds as shown by the wider range of Fl/Py ratios with SED-LSC-1 and BSA-MH as mixing end members (see plot above). This is evident by some sediment samples containing elevated concentrations of TPH and extractable petroleum hydrocarbons. Several soil samples analyzed from sediment borings were identified as being weathered and included SED-LSC-1, SED-LSC-7, and SED-LSC-8. Weathered tar refers to samples of DNAPL or soil/sediment containing TLM that shows evidence of environmental weathering (e.g., loss of compounds by evaporation, dissolution in water, or biodegradation). A "hump" on the chromatogram for sample SED-LSC-8 (see Appendix D.2 Forensic Report) suggests the presence of long chain and cyclic hydrocarbons associated with petroleum.

Soil sample MW-BGL-3 (18-19') in the plot above was characterized as being different and did not contain tar-like material from a pyrogenic-source of PAHs (i.e., sourced from combustion of coal or coal/oil mixture). That sample was collected from the P-Site and contained petrogenic PAHs derived from No. 2 fuel oil or diesel product (i.e., sourced from a petroleum distilled product).

4.2.2.2 Preliminary Mobility Assessment

TLM in soil can migrate via advective forces caused by hydraulic gradients and its own gravity if the specific gravity is greater than water. TLM migration in sediment can also occur by the same mechanisms as well as by ebullition and scour. Monitoring well bail tests showing full DNAPL recovery to pre-bail levels indicate TLM in soil at tested well locations has potential mobility (as DNAPL) via advection and gravity.

TLM encountered in some sediment boring locations in the historic alluvial deposits above the clay layer would have potential similar mobility as TLM encountered in soil borings. Ebullition-facilitated transport is a mechanism which occurs when gas bubbles (commonly methane but also CO2) form in sediments from degrading organic materials, pass through NAPL-impacted material and transport sheen and/or NAPL to the water column. Ebullition facilitated transport of sheen and NAPL was observed in the near-shore sediments in the creek near the City of Buffalo Pump Station. Scour is possible where surface water discharges to the creek and agitates areas where TLM occurs in shallow sediments. Based on the high MAH and PAH concentration in shallow sediments at sediment boring SED-LSC-3 with reported sheen, TLM is suspected in the area. During discharge from the City of Buffalo Pump Station, the high-volume of water discharging to the creek has the potential to scour the sediment surface and place TLM into suspension, thereby transporting MAHs and PAHs visible as a sheen on the surface water. This mechanism is discussed in Section 4.4.

4.2.3 Discharges to the Lower Scajaquada Creek and Sheen Observations

Section 2.4 identifies several combined sewer overflow discharges to the creek. As described in Section 3.8, outfalls to the creek in the Lower Scajaquada Creek Area were mapped during the investigation. Mapped concrete headwall and discharge pipes are shown on Figure 9A identified below:

16” RCP (reinforced concrete pipe) with gabion baskets for erosion control upstream from the West Avenue bridge with discharge to the surface water in the Riparian site.	Buffalo Sewer Authority (BSA) CSO SPDES Outfall #059
6” VCP (vitreous clay pipe)	BSA CSO SPDES Outfall #058 (unconfirmed continuously flowing VCP)
Headwall	Unknown concrete headwall south of City of Buffalo SPDES Outfall #58 (no pipe observed)
16” RCP	Unknown outfall along south bank of Creek, approx. 70’ south of West Ave.
30” RCP	BSA CSO SPDES Outfall #057 Stormwater outfall on south bank of Creek, east of Niagara St.
16” Steel outfall	Outfall associated with City of Buffalo Pump Station.

6" HDPE outfall/ 4" CPP (concrete)	Adjacent outfall along south bank of the Scajaquada Creek Outlet (presumed French drains/groundwater drainage for I-190 overpass berm)

Stormwater runoff from nearby streets and downspouts from the Scajaquada Expressway contribute to the mapped point source discharges to the creek in the Lower Scajaquada Creek. During the investigation, only the 6" VCP outfall was observed to be continuously flowing. This was true during longer periods of dry weather. Intermittent flow (cycled discharge) was observed at the 16-inch pipe associated with the City of Buffalo Pump Station. The flow rate during cycled pumping station discharge was visually estimated at more than 400 gpm. Since 2014, NYSDEC records reported petroleum-type sheens in the area of the pump house discharge on surface water in Scajaquada Creek. The City of Buffalo Pump Station, its function, and sheen generation is discussed in Section 4.4.

4.3 Soil, Groundwater, Sediment Quality

The SC Work Plan included collection and analysis of various soil, groundwater, and sediment samples to evaluate the P-Site and Lower Scajaquada Creek Area. Field sampling activities, laboratory analyses, and data usability assessments were discussed in Section 3. Laboratory analytical data included in Appendix D and Data Usability Summary Reports (DUSRs) are included in Appendix E. The resulting data are summarized in the following tables:

- Table 5A – Land Subsurface Soil Borings
- Table 5B – Groundwater
- Table 5C – Shallow Creek Sediment
- Table 5D – Deeper Creek Sediment

In addition to summarizing the data for each medium, Tables 5A through 5D identify various NYSDEC screening criteria and use color coding to indicate results that exceed the various screening criteria. The screening criteria are not intended to imply applicability to the site at this time, but only an indication of whether and to what extent the analytical results exceed various screening levels. The media-specific screening levels to which data are compared are summarized below:

Medium	Screening Criteria
Soil (land-based borings)	• Unrestricted Soil Cleanup Objectives (SCOs) from 6 NYCRR Part 375, Table 375-6.8(a) • Restricted Residential SCOs from 6 NYCRR Part 375, Table 375-6.8(b)* • Protection of Ecological Resources SCOs from 6 NYCRR Part 375, Table 375-6.8(b)* • Ecological Supplemental Soil Cleanup Objectives (SSCOs) from Table 1 of NYSDEC Policy CP-51 dated October 21, 2010*

Medium	Screening Criteria

Groundwater	• New York State Ambient Water Quality Standards (AWQSs) and Guidance Values (TOGS 1.1.1, June 1998)
	• NYSDEC interim guidance for 1,4-dioxane and PFAS as follows:
	  • For PFAS - Sampling, Analysis, and Assessment of PFAS under NYSDEC's Part 375 Remedial Programs (June 2021)
	  • For 1,4-Dioxane – Sampling for 1,4-Dioxane and PFAS under DEC's Part 375 Remedial Programs (February 2019)

Shallow Sediment	• The upper range of Class B Freshwater Sediment Guidance Values from Table 5 of NYSDEC's "Screening and Assessment of Contaminated Sediment" (June 14, 2014)
Deeper Creek Soil/ "Sediment"	• The upper range of Class B Freshwater Sediment Guidance Values from Table 5 of NYSDEC's "Screening and Assessment of Contaminated Sediment" (June 14, 2014) • Unrestricted Soil Cleanup Objectives (SCOs) from 6 NYCRR Part 375, Table 375-6.8(a) • Protection of Ecological Resources SCOs from 6 NYCRR Part 375, Table 375-6.8(b)* • Ecological Supplemental Soil Cleanup Objectives (SSCOs) from Table 1 of NYSDEC Policy CP-51 dated October 21, 2010*

* - Screening levels from Table 375-6.8(b) and CP-51 apply to a depth of 15 feet; soil samples collected entirely below 15 feet bgs were not considered when comparing to these values

It should be noted that deeper creek sediment samples were compared to both sediment screening values from NYSDEC's "Screening and Assessment of Contaminated Sediment" (June 14, 2014) and soil SCOs similar to land-based soil samples. This is based on the fact that – as indicated on the sediment core logs in Appendix B.3 – the deeper "sediment" samples were collected at various intervals including some within the fluvial deposits, some entirely below the fluvial deposits in the historical alluvial deposits, and some straddling the interface of the two materials. Material entirely within the depositional zone is consistent with the description of "sediment" in NYSDEC's guidance because this material "is comprised of all detrital, inorganic, or organic particles eventually settling on the bottom of a body of water." The deeper samples collected from historical alluvial and/or lacustrine silty clay deposits (see logs in Appendix B.3) are area-wide and geologically pre-date the formation of the Scajaquada Creek channel. Those materials are not representative of sediment that has deposited in Scajaquada Creek but are consistent with the land-based soil samples (Table 5A). Because the deeper sediment samples are comprised of both soil and sediment material, they are comparatively screened relative to both sediment- and soil-based standards in Table 5D. However, unlike the land-based soil samples, they are not screened relative to Restricted Residential SCOs because their in-water location precludes residential use at that specific location.

The following subsections discuss key media-specific observations based on the analytical results and screening comparisons provided in Table 5A through 5D. The first subsection discusses data for samples collected within the limits of the P-Site, as shown on Figure 2. The

second subsection discusses data for samples collected from other locations within the Lower Scajaquada Creek area. Note also that Section 4.4 discusses two additional media samples (water and debris) collected from a stormwater drainage vault that discharges to Lower Scajaquada Creek.

4.3.1 P-Site Area

As shown on Figure 2, two monitoring wells (MW-BGL-1 and MW-BGL-3) and one sediment boring (SED-BGL-1) were advanced within the limits of the P-Site. Media samples from these locations included two soil samples (plus a duplicate), two groundwater samples, one shallow sediment sample, and one deeper sediment sample. The primary objective of the sampling was to initially assess the potential presence and extent of contaminants within the P-Site and relative to the LSC Area. The following subsections summarize the key findings and observations relative to each medium.

4.3.1.1 P-Site Soil Samples

Two soil samples plus one blind duplicate were collected within the P-Site. One sample (plus the duplicate) was collected from a depth of 16-20 feet below grade (ftbg) at location MW-BGL-1. The other was collected from a depth of 17-19 ftbg at location MW-BGL-3. These samples were collected from lacustrine clay and fill material, respectively. As indicated on Table 5A:

- no VOCs, SVOCs, PCBs, herbicides, or cyanide were detected at concentrations exceeding any screening criteria.
- One pesticide (4-4'-DDT) was detected in the sample from location MW-BGL-3 at a concentration exceeding the Unrestricted SCO.
- Various metals were also detected, but only lead and zinc were detected above applicable screening levels. Both the lead and zinc exceedances were detected in the blind duplicate from location MW-BGL-1 but did not exceed in the corresponding parent sample.

As noted in Section 3.12 and discussed in Section 4.2.2, a forensic soil sample was analyzed from soil at a depth of 18-19 ftbg at MW-BGL-3. The sample was collected from the lower fill based on observation of sheen and petroleum-type odor. The Forensic Report (Appendix D.2) identified a non-pyrogenic source (diesel fuel) of PAH constituents detected in the sample.

4.3.1.2 P-Site Groundwater Samples

A monitoring well was constructed at each soil boring completed at the P-Site (MW-BGL-1 and MW-BGL-3). One groundwater sample was collected from each location and analyzed as discussed in Section 3.5. As indicated on Table 5B:

- no VOCs, pesticides, herbicides, PCBs, or PFAS were detected at concentrations exceeding any screening criteria.
- Selected SVOCs (and more specifically PAHs) were detected above AWQS screening levels in one or both samples, including benzo(a)anthracene, benzo(k)fluoranthene, benzo(a)pyrene, chrysene, indeno(1,2,3-cd)pyrene, and naphthalene.
- For metals, iron, magnesium, manganese, and sodium were detected at concentrations exceeding the AWQS screening criteria in one or both samples.

As noted in Section 4.3.1.1, the PAHs detected in the soil sample analyzed from the saturated zone at MW-BGL-3 are derived from a non-pyrogenic source indicating the PAHs detected in the groundwater sample from MW-BGL-3 are similarly sourced.

4.3.1.3 P-Site Sediment

One shallow and one deep sample were collected from the sediment boring location SED-BGL-1. The shallow sample was collected from the uppermost 6-inches of material at the bottom of the creek. The deeper sample was collected from a depth of approximately 1 to 3 feet below the sediment interface. As indicated on Table 5C, the shallow sediment sample did not exceed specified screening levels for any target analytes. As shown on Table 5D, the deeper sample did not exceed screening criteria for any analyte with the exception of calcium.

4.3.2 Lower Scajaquada Creek Area

As shown on Figure 2, five monitoring wells (MW-LSC-1 through -5) were installed and eight sediment borings (SED-LSC-1 through -8) were advanced to assess conditions within the Lower Scajaquada Creek area. Media samples from these locations included nine soil samples, five groundwater samples (plus two duplicates3), eight shallow sediment samples (plus one duplicate), and eight deeper sediment samples. The following subsections summarize the key findings and observations relative to each medium.

4.3.2.1 Lower Scajaquada Creek Area Soil Samples

Nine subsurface soil samples were collected during drilling of the five boreholes for monitoring well installation. This included two samples per boring with the exception of MW-LSC-2, where only one sample was collected for analysis. As indicated on Table 5A:

- Pesticides, PCBs, herbicides, and cyanide were not detected in any of the Lower Scajaquada Creek soil samples at concentrations exceeding specified screening levels.
- No VOCs were detected above screening levels in any of the samples collected from the MW-LSC-1 or MW-LSC-2 locations.

[3] One duplicate was analyzed for PFAS compounds, and the other was analyzed for other groundwater analytes

- Certain VOCs, including acetone, benzene, ethylbenzene, toluene, and xylenes were detected above Unrestricted SCOs in one or more samples from locations MW-LSC-3 through -5. Xylene concentrations are also noted in Table 5A to exceed the protection of ecological resources SCO due to the fact that the criterion is the same as the Unrestricted SCO. The acetone concentrations are generally low, and the exceedances are all estimated values below the laboratory Reporting Limit. The detections of benzene, ethylbenzene, toluene, and xylenes (BTEX) are likely related to the TLM observed in the soil at those locations.
- Only two of the nine samples (the 11-12' sample from MW-LSC-3 and the 24-28' sample from MW-LSC-4) indicated SVOC concentrations above screening levels. Several SVOCs exceed screening levels in each sample, with exceedances primarily PAH compounds associated with TLM observed in the lower alluvial soils above the red-brown clay. In most cases, the detected concentrations exceed only the most stringent Unrestricted SCO and exceedances of other screening levels are noted because those standards are equal to the Unrestricted SCO value. The two exceptions where a detected SVOC concentration exceeds a less stringent (i.e., numerically higher) screening level are benzo(a)pyrene and hexachlorobenzene; both occur in the 11-12' sample collected from fill having a suspected solvent-type odor from location MW-LSC-3. TLM was not observed at that location.
- Various metals were detected in all of the Lower Scajaquada Creek soil samples, but only three samples [MW-LSC-1(13-15), MW-LSC-3(11-12), and MW-LSC-4(22-24)] indicated exceedances of screening level values for one or more metals. The metals exceeding screening levels in one or more samples include arsenic (one sample), copper (two samples), lead (one sample), mercury (two samples), and zinc (one sample). The exceedances were all relative to the most stringent Unrestricted SCO. Table 5A also indicates exceedances of other screening levels, but in each case those values are equivalent to the Unrestricted SCO.

4.3.2.2 Lower Scajaquada Creek Area Groundwater Samples

Five monitoring wells were constructed within the Lower Scajaquada Creek area, and one sample was collected from each well as part of the site characterization. The groundwater samples were analyzed for the parameters indicated in Section 3.12 and the results are included in Table 5B. As indicated by the screening-level evaluations on that table:

- With the exception of metals (discussed below), no target analytes were detected in the groundwater sample collected from well MW-LSC-1.
- No pesticides, herbicides, or PCBs were detected in any of the groundwater samples
- Total cyanide concentrations, when detected, were well below AWQSs.
- BTEX compounds were detected in samples from four of the wells (excluding MW-LSC-1), in most cases at concentrations above AWQSs.

- Other VOCs detected above AWQSs in one or more samples included isopropylbenzene, styrene, and vinyl chloride.
- Several SVOCs, primarily PAHs, were detected above AWQSs in the samples collected from locations MW-LSC-2 through -5.
- 1,4-Dioxane was detected in four groundwater samples, with one (MW-LSC-5) exceeding the screening level of 0.35 ug/l.
- Other SVOCs detected above AWQSs included in at least one sample included biphenyl and 2-methylphenol.
- Various metals were detected in groundwater samples from each of the wells. Metals detected above AWQSs in one or more samples included iron, magnesium, manganese, and sodium.
- Various PFAS compounds were detected in all samples except the one from MW-LSC-1. Only the detected concentration of PFOS in MW-LSC-4 was above the screening level.

PAHs detected in groundwater samples in the LSC wells are primarily related to TLM observed in soils at these locations. 1,4-Dioxane detection in MW-LSC-5 is likely associated with the chlorinated VOC impacts detected in groundwater at 31 Tonawanda Street. PFAS detected in groundwater samples are likely related to storm water/CSO discharges to Scajaquada Creek.

4.3.2.3 Lower Scajaquada Creek Sediment Samples

Eight shallow and eight deeper samples were collected from the sediment boring locations at SED-LSC-1 through -8. The shallow sample was collected from the uppermost 6-inches of material at the bottom of the creek. The depths of the deeper samples below the sediment interface varied based on core recovery and field observations; typically, sediment/soil intervals that had visually suspected impacts were sampled. Elevations and the approximate depths below the sediment surface for each deep sediment sample are included on Table 5D.

Analytical data for the shallow sediment samples are summarized on Table 5C and screened relative to NYSDEC's freshwater sediment guidance values representative of the upper range of Class B sediment. As indicated on that table:

- Only one VOC (ethylbenzene) at one location (SED-LSC-3) exceeded its screening criterion.
- Several SVOCs, primarily PAHs, were detected in all of the shallow sediment samples. None of the individual SVOCs for which sediment screening values are available exceeded their respective screening levels. However, the total concentrations for 17 PAHs compounds summed by NYSDEC exceeded the sediment screening level (35,000 ug/kg) in four of the eight locations (SED-LSC-1, -2, -3, and -8). SED-LSC-1 through -3 are the samples collected from the Inlet area (backwater "slip" portion of the channel) and SED-LSC-8 represents the most upstream location. SED-LSC-8 forensic data

identified branched and cyclic alkanes and petroleum mixtures characteristic of accumulations of weathered fuel oils and heavy lubricating oils commonly found in urban sediment background. PAH concentrations at SED-LSC-3 were nearly 500 times higher than other shallow sediment samples. While not discussed, NOAA 34 PAH analytical results are summarized in Appendix D.2 for each of the shallow sediment samples and is presented for future use, if needed, to assess potential toxicologic effects to the benthic community.

- PCB Aroclors were detected in all of the samples, with three locations indicating total PCB concentrations above the screen criterion (1 mg/kg). The maximum detected concentration among the three samples was 1.23 mg/kg at SED-LSC-4.
- No herbicides or cyanide were detected in any of the samples.
- Three pesticides (DDT, DDE, and DDD) were detected at estimated concentration in the sample from location SED-LSC-1; none exceeded available screening criteria.
- Various metals were detected in all of the samples. Metals exceeding screening levels in one or more samples included copper, lead, nickel, and zinc. The highest levels of lead, mercury, nickel, and zinc were detected at SED-LSC-3.
- The samples were analyzed for petroleum hydrocarbon content. Volatile petroleum hydrocarbons were mostly not detected, with the exception of C_9-C_{12} aliphatics of SED-LSC-3. Extractable petroleum hydrocarbons were more frequently detected, with the highest concentrations detected at location SED-LSC-3. No NYSDEC standards are available for these hydrocarbon data, which were collected for forensic purposes.
- The highest concentrations of ammonia and sulfide were detected in samples collected from the Inlet area. Those constituents influence benthos toxicity.
- Total organic carbon content among the samples ranged from 4.67 to 13.5%.

Analytical data for the deeper sediment samples are summarized on Table 5D and screened relative to the various noted criteria. As indicated on that table:

- The deep sediment sample from location SED-LSC-4 (14-16') did not exceed any screening values for VOCs, SVOCs, total cyanide, pesticides, herbicides, or PCBs.
- BTEX constituents were detected in all of the remaining deep sediment samples, with highest BTEX concentrations detected in samples collected from the Inlet area. Benzene, ethylbenzene, and xylene concentrations exceeding one or more screening values in most samples. Benzene, ethylbenzene, and xylene exceeded freshwater sediment guidance values for Class B in at least two samples each.
- Isopropylbenzene was detected in four samples at concentrations exceeding the freshwater sediment screening value for this compound.
- Other VOCs detected in at least sample at concentrations exceeding one or more screening levels included cis-1,2-dichloroethene, trans-1,2-dichloroethene, and methyl ethyl ketone.
- Several SVOCs, primarily PAHs, were detected in all of the deeper sediment samples and several exceeded one or more screening values for individual compounds. The total

concentrations for 17 PAHs compounds summed for NYSDEC sediment criteria comparison exceeded the sediment screening level (35,000 ug/kg) in five deeper sediment samples (SED-LSC-1, -2, -3, -6, and -8) with sample SED-LSC-3 having the highest PAH concentrations. This is consistent with the shallow sediment samples, plus the addition of SED-LSC-6. TLM and/or sheen was identified at most of these locations.

- Target herbicides were not detected in any of the samples.
- Total cyanides were not detected in any samples at concentrations above screening levels.
- Two pesticides (DDT and DDD) were detected at concentrations above the unrestricted and protection of ecological resources SCOs. DDT exceeded the screening levels in the deep sediment sample from SED-LSC-6. DDD exceeded the screening value in the deep sediment samples from locations SED-LSC-3 and SED-LSC-6.
- Detected total PCB concentrations in two samples (SED-LSC-3 and SED-LSC-8) exceeded the Unrestricted SCO of 0.1 mg/kg.
- Various metals were detected in all of the samples. Metals exceeding screening levels in one or more samples included aluminum, cadmium, calcium, chromium, copper, lead, manganese, mercury, nickel, and zinc.
- Total organic carbon content among the samples ranged from 3.53 to 8.00%.

As a further evaluation of the shallow sediment data, Figure 10 identifies locations of shallow sediment samples collected in 2021 as part of this site characterization along with samples collected by NYSDEC and USACE in 2017 (NYSDEC/Empire GEO Services, June 2017) (see Section 2.4). It also plots distances along the approximate centerline of the creek beginning at the northwest end of the Inlet (Station 0+00), continuing toward the outlet to Black Rock Canal (Station 5+22), and extending upstream to the downstream end of the West Avenue bridge (Station 15+88). Using this stationing system, Figure 10 also includes four embedded charts that present surficial sediment concentrations versus distance along the stationing line. The charts present data for 2017 sample locations (orange) and 2021 sample locations (blue) by projecting the sample locations to their corresponding location along the centerline of the creek. The x-axis on each chart is the distance along the centerline measurement of the creek and the bar height is an indication of the measured total concentration at that point. The four charts compare data for mercury, lead, total PCBs, and total PAHs (based on the 16 PAHs summed in the 2017 dataset). These four COCs were selected for comparison because they have been identified as COCs for the Buffalo River, which is part of the broader Niagara River Area of Concern.

All of the 2021 samples plotted on Figure 10 were collected from a depth interval of 0-6" whereas the 2017 samples were collected as various intervals beginning at the sediment surface and extending up to 3.17 feet in thickness. Therefore, the samples intervals are not directly comparable between the two datasets. Regardless, the 2017 data almost always indicate higher concentrations than the 2021 results within a given reach of the creek. The one exception is a 2021 total 16 PAH concentration associated with sample location SED-LSC-3 (5,649 ppm). The generally lower concentrations measured in 2021 samples suggest either an overall improvement in conditions over the four years between sampling events or that the deeper sediment increments

included with the 2017 samples have higher concentrations relative to the shallower material. The latter would be an indication of recent cleaner sediment deposits. Note also that concentrations in the Inlet portion of the creek are generally much higher than the segment between the outlet and West Avenue, likely indicative of the depositional nature of this area since the natural outlet was modified during highway construction in the 1950s.

4.4 Pump Station Vault Debris and Discharged Water

4.4.1 Debris and Water Testing

As described in Section 3.9, a sheen was observed on Scajaquada Creek during discharge from the City of Buffalo Pump Station (see Figure 9A for location). The pumping station discharges groundwater and storm water from the topographically depressed Niagara Street beneath the at-grade rail crossing. A general schematic profile of the pumping station is provided on Figure 9B. A water sample was collected from Manhole #23 directly upstream of the pump station (BSA-MW-WATER) and a sediment/debris sample from the pump station vault (BSA-VAULT-DEBRIS). Analytical data for the water sample, which is representative of stormwater and groundwater flowing in the manhole, is summarized in Table 5B. Constituents detected above regulatory comparison criteria are identified below.

- The manhole water sample exhibited elevated concentrations of 1,4-dioxane (12,200 ug/L), a stabilizer used in chlorinated VOC products, when compared to the NYSDEC Interim Guidance Criteria of 0.35 ug/L.
- Concentrations of CVOCs, including chloroethane (60 J ug/L), 1,1- dichloroethane (140 ug/L), 1,1-dichloroethene (8.8 ug/L), cis-1,2-dichloroethene (1,700 ug/L), 1,1,1-trichloroethane (53 ug/L) and vinyl chloride (280 ug/L) were detected above AWQS.
- BTEX compounds and several PAHs were detected above AWQS. Naphthalene was detected below NYS guidance.
- Metals that exceeded AWQS included antimony, iron, lead and sodium as presented on Table 5B.
- PCBs were detected at an estimated (J-qualified) concentration of 0.066 ug/L.
- While PFAS was detected in the sample at concentrations similar to other groundwater samples, individual compounds were below NYS guidance values.

A sediment/debris sample was collected from the bottom of the pump station vault (BSA-VAULT-DEBRIS) laboratory analysis. A sample was not obtained from MH#23 as the material was gravel size and insufficient volume could be obtained for laboratory analysis by Alpha Analytical; however, sufficient volume was present for forensic analysis. Sediment/debris sample results for material at the bottom of the pump station vault are summarized in Table 5E. Comparison criteria are not provided on the table as the sample was not natural sediment or soil. Testing identified the following:

- The sediment/debris sample from the vault contained concentrations of CVOCs, including chloroethane (160 ug/kg), 1,1-dichloroethane (280 ug/kg), 1,1-dichloroethene (23 ug/kg), cis-1,2-dichloroethene (2,200 ug/kg), trans-1,2-dichloroethene (14 ug/kg), 1,1,1-trichloroethane (440 ug/kg), and vinyl chloride (450 ug/kg).
- BTEX compounds were detected in the vault sediment debris sample including benzene (1,800 ug/kg), toluene (2,900 ug/kg), ethylbenzene (62,000 ug/kg) and total xylenes (33,000 ug/kg).
- Total PAH 17 was less than 5 ppm.
- Total PCBs in the sediment/debris sample from the vault were detected at an estimated (J-qualified) concentration of 135 ug/kg.

Sufficient volume of sediment/debris was available from Manhole #23 for forensic analysis. Additionally, a sample of the debris within the pump station vault was submitted for forensic analyses. Both samples contained suspected TLM with a strong coal tar-like odor. The forensic testing results for these two samples are presented in Appendix D.2. Forensic analyses identified MAHs, including benzene, toluene, ethylbenzene, and xylenes, PAHs, and saturated hydrocarbons in each of the samples. Forensic laboratory detected compound concentrations are summarized below:

- BSA-MH (MH#23) – Total PAH 16[4] – 1,850 mg/kg; TPH – 9,680 mg/kg
- BSA-VAULT - Total PAH 16 – 1,880 mg/kg; TPH – 9,500 mg/kg
- Both samples contained weathered pyrogenic MAH and PAH tar-like patterns consistent with TLM in soil and DNAPL in wells. Low-level petrogenic character identified as Fluorine/Pyrene ratios were slightly higher than soil and DNAPL samples from wells.

4.4.2 Pump Station Operation and Suspected Influence

GEI submitted a FOIL request to the City of Buffalo and obtained design drawings for the local storm sewer network servicing the pump station. These drawings indicate storm sewer piping that is present below Niagara Street and that transmits water to the pump station was installed to approximate elevations ranging between 570 ft and 568 ft. Piping leads to Manhole #23 at elevation 565.9 ft and to the pump station vault with a bottom elevation of approximately 558 ft. Based on the observed piping configuration, it appears that the water level in the vault is maintained at an elevation no higher than approximately 567 ft to 568 ft (see Figure 9B). GEI observed a pump cycle discharge during sampling where the water level in the vault wet well was drawn down several feet, likely approaching an elevation of 560 ft.

Section 4.1.3 identified a depressed area of groundwater in the area of Niagara Street and the City of Buffalo Pump Station. Given that the storm sewer piping beneath Niagara Street is approximately 3 to 4 feet lower than the typical surface water elevation of Scajaquada Creek and approximately 1 to 2 feet lower than groundwater elevations in nearby wells, it is suspected that

[4] Total PAH 16 excludes 2-methylnapthalene where PAH 17 includes the compound in the sum.

the pump station cycling is affecting the elevation of groundwater in the area of the pump station. The observation of water flow in the storm sewer piping during a dry weather event suggests groundwater is collected and conveyed by the storm sewer effectively maintaining a depressed groundwater elevation beneath Niagara Street. Hydraulic testing and monitoring of the water table in the area of the pump station is required to validate this presumption.

Field observations and laboratory data described in Section 4.4.1 indicate TLM presence in the pump station vault and Manhole #23. This observation was somewhat unexpected because TLM and DNAPL in wells closest to the pump station was much deeper (i.e., more than 10 feet deeper). The mechanism for TLM entry into the vault and Manhole #23 is not fully understood at this time and requires additional assessment. Surcharge of the pump station vault and piping can occur when creek levels are five to six feet higher during seiche conditions. Backflow from the creek to the storm sewer is a plausible condition for TLM entry.

During pump station discharge cycles, stormwater and groundwater that has accumulated in the vault (Figure 9B) discharges to surface water in Scajaquada Creek by way of a 16-inch diameter cast-iron pipe installed in the concrete headwall on the creek side of the pump station at an estimated flow rate of approximately 2500 gpm. Limestone riprap blocks designed to mitigate erosion of the bank during discharge events are present directly below the pump station outfall as part of the original design specifications for the pump station. Observations made during the SC field investigation indicated the riprap below the outfall exhibited heavy staining on riprap material with suspected TLM, a sheen on pooled surface water within the riprap, and a strong coal tar-like odor.

The high rate of discharge from the pump station outfall causes turbulent flow at the creek edge directly downgradient of the outfall. GEI field staff observed the generation of a heavy petroleum-like sheen on the surface water of the creek during discharge cycles. The discharge events are suspected to scour near shore sediments. It is unknown if the observed sheen results from TLM presence in the pump station discharge or if nearshore sediment contains TLM and the discharge causes scour and resuspension of TLM in shallow sediment. Ebullition processes were also observed in the near-shore sediments in this area. The specific contributions of these factors have not been determined and further assessment would be necessary to characterize these pathways.

Shallow sediment sample SED-LSC-3 (0-0.5) (Figure 2) was collected from Scajaquada Creek sediments directly adjacent to the pump station outfall. Analytical results from this sample exhibited the highest concentrations of total PAHs (6,849 mg/kg) and EPH fractions (67,000 mg/kg) in shallow sediments analyzed during the SC investigation. The data suggest that stormwater discharge from the City of Buffalo pumping station may potentially influence shallow sediment quality in the Inlet area of Scajaquada Creek. Further assessment is required to understand if a correlation exists between pump station discharge and elevated constituent concentrations detected in this area.

4.5 Preliminary Conceptual Site Model

A preliminary conceptual site model was developed and is presented to understand the potential sources of contamination, constituents of potential concern, potentially affected media (soil, sediment, and groundwater), and preliminarily identify transport and exposure pathways that could impact human or ecological receptors. The conceptual site model synthesizes data collected by the NYSDEC, investigations at nearby BCP sites, and this investigation.

4.5.1 P-Site

A major source of contamination was not identified at the P-Site. Some minor contamination of deeper soil and groundwater related to diesel fuel was identified. Incidental releases from highway traffic are the probable cause of the petroleum-related impacts identified. Constituent concentrations in shallow sediment, and soil below the recent fluvial deposits (red-brown clay) did not exceed regulatory comparison criteria. Site investigation data indicate the P-Site is not a source of contamination and does not pose a significant threat to human health or the environment.

4.5.2 Lower Scajaquada Creek Area

VOCs and PAHs related to coal tar and petroleum-related compounds, PCBs, and several metals were detected in soil and creek sediments above regulatory comparison criteria in the Lower Scajaquada Creek area. In groundwater, BTEX and several PAH compounds as well as isopropylbenzene, styrene, vinyl chloride, 1,4-dioxane, and PFAS were detected above AWQS. TLM was identified in some soil and sediment borings and DNAPL was observed in several monitoring wells (see Figure 8). Based on the forensic identification of coal tar in soil and sediment, a discussion of historic transport follows.

Discussion of Historic and Possible Active Coal Tar Transport: As described in Section 4.2.2.1, the forensic evaluation of MAH and PAH chemical patterns from the analysis of soil and sediment samples containing TLM identified forensic chemical patterns similar to the DNAPL samples derived from historic coal gas manufacturing at the Iroquois Gas/Westwood Pharmaceutical Former MGP Terrestrial and Riparian Sites. It was also apparent that many of the sediment and soil samples contained varying degrees of mixtures with petroleum. It is presumed that releases of coal tar to the ground surface between MGP start-up (early 1900s) and termination of operations in 1955 caused contamination of soil, groundwater, and creek sediments in addition to historic releases of petroleum from former industrial sites that operated in the Black Rock Area and CSO discharges. The conditions at the Former MGP were mitigated following implementation of remedial actions in 2000. Prior to remediation, transport of coal tar occurred in soil and sediment. Physical conditions conducive for coal tar transport from the Former MGP toward the creek and migration toward the present-day creek Inlet include:

1. a convergent groundwater flow pattern with hydraulic gradients toward the creek,

2. coarse grain alluvial soils that have effective porosity and hydraulic conductivity suitable for coal tar migration,
3. the material density of the coal tar being slightly heavier than water, and
4. vertical confinement of the alluvial soils at its base by the area-wide red-brown clay layer with a topographic surface that slopes in a channel-like manner in a southwest and north direction.

Currently, the Terrestrial Site remediation was designed and constructed to mitigate coal tar migration through surface capping, a physical barrier (sheet pile wall), and a hydraulic barrier through operation of the groundwater collection trench on the land side of the site. On the Riparian side of the Site, (the NYSDEC specified the area of sediment cleanup to be between historic boring B-1 upstream and the West Avenue bridge downstream), contaminated sediments were excavated, a sediment cap was installed, and a physical barrier was installed immediately upstream from the West Avenue bridge (i.e. vertical sheet pile wall). Active DNAPL removal occurs upstream of the vertical barrier to remove remaining coal tar residuals upstream of the West Avenue bridge. These remedial measures were put in place to contain coal tar presence identified at the former MGP and adjacent creek sediments following historic site investigations.

Coal tar residuals in alluvial soil identified in two of the three NYSDEC wells installed along the bike path next to the Riparian Site, with DNAPL presence in well MW-100, is expected to be contained by current remedial controls and site geologic condition. The rapid rise in the clay surface elevation along the northwest side of the current creek channel (Figure 5) and a southeastward groundwater flow direction toward the creek would prevent coal tar migration beyond the bounds of the Riparian Site to areas beneath the 51-71 Tonawanda Street BCP Site. Migration of DNAPL at well MW-100 in a southern direction is expected to be contained by DNAPL removal by the South Vault, the vertical sheet pile barrier, and the concrete abutment of the West Avenue bridge.

Historic coal tar residuals remaining in alluvial sediments downstream from the West Avenue bridge has the potential to migrate laterally in a downstream direction within the lower portion of historic alluvial creek channel above the scoured surface of the red-brown clay. As shown in Figures 5 and 8, the deepest areas of the former creek channel are somewhat offset to the north side of the current creek edge, especially near the intersection of Niagara and Tonawanda Streets and northward paralleling Niagara Street (well locations MW-LSC-4, MW-LSC-5, 1660-MW-7, and 1660-MW-8). In this area, the clay surface elevation is deeper, and a groundwater depression occurs as groundwater beneath Niagara Street removed by the City of Buffalo Pump Station. These features can cause advective transport of non-residual coal tar toward this area. However, with remedial controls in place at the primary source area controlled, advective transport is expected to be diminished over time. Other mechanisms of coal tar transport considered viable in Scajaquada Creek is the scour and re-suspension of existing coal tar residual or direct discharge of coal tar blebs to the creek causing sheen. As described in Section 4.5, further assessment is needed to understand if these mechanisms are significant.

Non-MGP Related Constituent Loadings: In addition to PAHs and BTEX associated with TLM, several constituents of concern are non-MGP sourced chemicals that affect groundwater, soil, and sediment. These include chlorinated VOCs and 1,4-dioxane, PAHs and BTEX from non-MGP sources, PCBs, heavy metals, PFAS, and organic loading that affects nutrient levels in the shallow sediment. Sources of these constituents are BCP sites and point source and non-point source discharges. Mechanisms of transport include advective transport in groundwater, direct discharges from three BSA combined sewer overflow discharge pipes, roadway runoff from nearby streets and downspouts for the Scajaquada Expressway, and upstream sources beyond the Riparian Site.

The City of Buffalo Pump Station on Niagara Street discharges storm water and groundwater to the creek. Active transport of TLM with petroleum was identified in sediment/debris in the vault and a manhole (see Section 4.5). Water discharge contained elevated concentrations of chlorinated VOCs, 1,4-dioxane, PCBs and metals. This discharge affects shallow sediment quality in the Inlet portion of the creek.

Human Exposure Pathways: Scajaquada Creek is a Class B designated stream. Primary and secondary contact recreation and fishing exposure are pathways that could potentially impact human receptors. Coal tar impacted soils are typically deeper than 15 feet below the ground surface so human exposure would be limited to construction workers involved in deep excavation work such as major utility infrastructure development or building construction with deep foundations. Under these scenarios, human exposure routes for chemicals include:

- inhalation of VOCs volatized from groundwater (particularly CVOCs in the area of Niagara Street);
- dermal contact inhalation and incidental ingestion of soil/sediment particulates; and
- dermal contact with ground water/surface water.

Groundwater in the area is not a source of drinking water.

The following ecological exposure routes for chemicals include:

- inhalation of VOCs volatized from soil gas in burrow air (particularly CVOCs in the area of Niagara Street);
- incidental ingestion and dermal contact of creek sediment particulates; and
- ingestion and contact with contaminated surface water.

Additional evaluation of exposure pathways is required to determine if exposure pathways are complete.

5. Conclusions and Recommendations

5.1 P-Site

Site historical information indicate the P-Site contained a gas holder between 1896 and 1928. Historical information indicates coal gas was not manufactured on the property. During the mid-1950s, the property was developed for construction of the I-190 Thruway corridor and the on/off ramps for the I-198 Scajaquada Expressway. Construction involved soil removal and importation of construction quality fill suitable for highway road base and support structures. Substantial soil excavation of the pre-existing fill as well as native red-brown clay was required to construct the re-routed outlet of Scajaquada Creek during the highway construction project.

Site investigation did not identify buried structures and soil, groundwater, and sediment sampling results did not detect concentrations of chemical constituents which would pose a significant threat to the human health or the environment. The PAHs detected in suspected petroleum impacted soil at one monitoring well location were derived from a petrogenic source with a chemical fingerprint suggesting impact from diesel fuel. This is consistent with the property's current use supporting highway infrastructure. These findings suggest the P-Site does not pose a significant threat to human health or the environment. No further assessment is recommended.

5.2 Lower Scajaquada Creek

TLM was identified in many soil and sediment borings and DNAPL accumulation was observed in monitoring wells situated closest to Scajaquada Creek. Forensic analysis of MAH and PAH patterns indicate a pyrogenic source with influences from petroleum-related constituents. The surface geometry of the red-brown clay and hydraulic gradients confine the distribution of coal tar impacts to deeper alluvial soil in the area of the current creek channel. Coal tar residuals in soil and DNAPL accumulation was identified in wells where the historic creek channel was wider. This was the case in the subsurface near the intersection of Niagara and Tonawanda Streets and the area immediately west of Niagara Street closest to the City of Buffalo Pump Station.

BTEX and PAH compounds were detected in groundwater samples collected from wells near the creek between the Riparian Site and properties upgradient of Niagara Street at concentrations above AWQS where coal tar impacts were identified in soil. Similarly, BTEX and PAHs were detected in groundwater at well locations closer to the creek Inlet where TLM was present in soil. However, groundwater also contained several chlorinated VOCs at concentrations above AWQS, as well as 1,4-dioxane and PFAS at levels above screening criteria. This included water being discharged from the City of Buffalo Pump Station to Scajaquada Creek also was found to contain these chemicals above AWQS.

In creek sediments, shallow (upper six inches) and deeper sediments were characterized. Shallow sediments exhibited more impacts from coal tar in the Inlet area of the creek near the Pump Station compared to samples collected from the main creek channel. Sediment samples containing TLM typically exceeded sediment screening levels for PAHs as well as other constituents. The concentration of total 17 PAH compounds exceeded NYSDEC sediment screening levels in four of the eight shallow sediment locations sampled. PAH concentrations at SED-LSC-3 were nearly 500 times higher than other shallow sediment samples and influence from City of Buffalo Pump Station discharge is suspected. That particular sample also contained elevated levels volatile petroleum hydrocarbons and extractable petroleum hydrocarbons. Total PCB concentrations exceeded 1 mg/kg at three locations with a direct correlation of outfall pipes and the pump station. Metals that include mercury, copper, lead, nickel, and zinc exceeded screening criteria with the highest detected at SED-LSC-3. The highest concentrations of ammonia and sulfide were detected in samples collected from the Inlet area. Those constituents influence benthos toxicity.

In deeper sediments, coal tar impacted materials or sheen was identified in six of eight sediment borings. The highest concentrations of BTEX and PAH compounds in deeper sediments were detected in borings completed in the creek Inlet (borings SED-LSC-1 through SED-LSC-3) and sediment boring SED-LSC-6, which is situated near well MW-LSC-4 (which contained coal tar impacts with DNAPL accumulation). Concentrations of total 17 PAHs exceeding NYSDEC screening levels occurred in each of the deeper sediment samples collected from the Inlet area (concentrations were approximately 4 to 5 times higher than other detections) including and the samples collected from locations at SED-LSC-6 and SED-LSC-8 which also exceeded screening criteria. Pesticide compounds were detected above SCOs in samples analyzed from SED-LSC-3 and SED-LSC-6 and total PCB concentrations exceeded SCOs samples collected from SED-LSC-3 and SED-LSC-8. Metals exceeding screening levels in one or more samples included aluminum, cadmium, calcium, chromium, copper, lead, manganese, mercury, nickel, and zinc.

Site characterization findings indicate further evaluation of the Lower Scajaquada Creek Area is warranted to fully assess the overall threat to human health and the environment from the constituents detected in groundwater, soil, and sediment. Therefore, the completion of a Remedial Investigation and Feasibility Study (RI and FS) is recommended for the Lower Scajaquada Area.

An objective of the RI would be to fill key data gaps that include: further characterization of the overall extent of TLM in soil and creek sediments including fate and transport, identification of major infrastructure in the Lower Scajaquada Creek Area to assess possible migration pathways of utilities deep enough to be in contact with coal tar impacted soil, and investigation of the influence of discharge from the City of Buffalo Pump Station including evaluation of potential DNAPL mobilization during operation and other impacts of the discharge on creek sediment quality.

6. References

BE3 Corp, 2019. Draft Remedial Investigation/Alternatives Analysis Report, 57-71 Tonawanda Street Site, Buffalo, New York. NYSDEC Site # C915351. May 2019.

Brown's Directory of American Gas Companies. 1918. Press of 'Progressive Age'.

CBCA, 2010. Historic Resources of the Black Rock Planning Neighborhood, City of Buffalo, Erie, County, New York. State and National Registers of Historic Places Nomination Multiple Property Documentation Form, November 2010.

Ecology & Environment, 1992. Phase II Investigations – Fedders Automotive Components, City of Buffalo, New York, Site No. 915024, February 1992.

EDR Historic Aerial photographs 1938-2017, Inquiry number 6562420.8 July 2, 2021, 1654 Niagara Street, Buffalo, NY 14207.

GEI Consultants, Inc. P.C. August 2021. Site Characterization Report Former Buffalo Gaslight Iroquois Gas Corp. Site Buffalo, New York NYSDEC Site #915351

GeoTrans, Inc. February 1994. Final Remedial Investigation/Feasibility Study – Iroquois Gas/Westwood Pharmaceuticals Site #915141

Labella Associates, P.C., 2016. Phase II Environmental Site Assessment, 1660 Niagara Street, Buffalo, New York BCP C915311.

New York State Thruway Authority, 1956. Plans for Constructing a Portion of the New York State Thruway, Niagara Section – Subdivision N-6 (Porter Avenue to Ontario Street- Part 1). NTC-56-03, March 1956.

NYSDEC, 1993. Scajaquada Creek Sediment Sampling Grant Street Weir to the Black Rock Canal, Division of Hazardous Waste Remediation. August 1993 Authored by Kevin N. Glaser.

NYSDEC, 1999. Division of Water Technical and Operation Guidance Series (TOGS 1.1.1),

NYSDEC, 2017. Summary Report for Sediment Screening Assessment Within the Lower Scajaquada Creek Corridor and Block Rock Canal, Buffalo, New York. Prepared by Empire Geo Services, Inc. Project Number BEV-17-001.

NYSDEC, 2010. DER-10 – Technical Guidance for Site Investigation and Remediation, May 2010.

NYSDEC, 2014. Screening and Assessment of Contaminated Sediment, June 2014.

NYSDEC, 2021. 6 NYCRR Part 375, Environmental Remediation Programs, June 2021.

NYSDEC, 2021. Report. HW.915351.2019-06-17. Property Ownership Mapping (NYSDEC provided).

NYSDEC/Empire GEO Services, 2015. Letter report, Sediment Sampling - Scajaquada Creek Slip behind 1654 Niagara Street, Buffalo, New York NYSDEC Spill Number 1406307; PIN 06511, February 2, 2015.

NYSDEC/Empire GEO Services, 2017. Letter report, Summary Report for Sediment Screening Assessment Within the Lower Scajaquada Creek Corridor and Black Rock Canal Buffalo, New York NYSDEC Spill Number 1406307; PIN 06511 Great Lakes Program Call Out GL-2017-01, June 27, 2017.

NYSDEC, 2021. Order on Consent and Administrative Settlement, Former Buffalo Gaslight Iroquois Gas Corp., NYSDEC Site No 915351. Signed June 23, 2021.

NYSDOT, 2017. Final Design Report/Final Environmental Impact Statement, Final 4(f) Evaluation PIN 5470.22 NYS Route 198 (Scajaquada Expressway) Corridor Grant Street Interchange to Parkside Avenue Intersection City of Buffalo Erie County, November 2017.

NYSDOT, 1956. Subsurface Exploration Logs for the Scajaquada Creek Expressway, Delaware Park (provided via e-mail as supplement to FOIL request)

Panamerican Environmental, Inc., 2014. Phase II Environmental Site Assessment – 31 Tonawanda Street, Buffalo, New York - Site No. C915299. September 2014.

Panamerican Environmental, Inc., 2017. Phase II Environmental Site Assessment – 68 Tonawanda Street, Buffalo, New York - Site No. C915316. February 2017.

Photographic Log

FIELD OBSERVATION PHOTOGRAPHS

Project :	Former Buffalo Gaslight Iroquois Gas Corp. Site Characterization	**Page:**	1 of 10
Client :	National Fuel Gas Distribution Corp.	**GEI Proj. No.**	2101620

Photo 1. Geoprobe (Nothnagle Drilling) set up at MW-BGL-3 location beneath I-190.

Photo 2. Geoprobe (Nothnagle Drilling) set up at MW-LSC-2 location adjacent to Scajaquada Creek.

Photo 3. Barge and skid-mounted drill rig (SJB Enterprises) set up at SED-LSC-6 location.

Photo 4. Barge and skid-mounted drill rig (Nothnagle Drilling) within Scajaquada Creek Slip.

Photo 5. Shallow sediment sample collection in Scajaquada Creek main channel.

Photo 6. Geoprobe (Nothnagle Drilling) set up at MW-LSC-3 location.

Photo **7. Representative photo of native reddish-brown lacustrine clay unit. (MW-BGL-1 16-20' sample shown.)**

Photo **8. Representative photo of recent fluvial deposits in Scajaquada Creek. Sample depth is upper 5.5 feet of sediment column. (SED-LSC-4 9.5-14 sample shown, top is to the right.)**

Photo **9. Stormwater discharge from the City of Buffalo pumping station located at 1654 Niagara Street.**

Photo **10. Flooding along Niagara Street at the CSX railroad underpass, following Lake Erie seiche event on December 11, 2021.**

Tables

Table 1A. Stratigraphic Summary and Monitoring Well Screened Interval
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

Location	Ground Surface Elevation (fasl)	Total Depth	Well Screen Length	Well Screen elevation	Sump Present	Unit Thickness (ft)		Top of Alluvium Elevation (fasl)	Depth to Top of Lacustrine Clay (ft)	Top of Lacustrine Clay Elevation (fasl)	DNAPL Thickness Measured in Well [1] (inches)	Notes/Observations
						Fill	Alluvium					
Site Characterization Monitoring Wells:												
MW-LSC-1	585.76	24.0	10.0	561.76-571.76	Yes	17.0	5	568.76	22.0	563.76	0	--
MW-LSC-2	582.46	40.0	10.0	549.96-559.96	Yes	16.0	12	566.46	28.0	554.46	1	Coal tar-like odor base of Alluvium/DNAPL accumulated in sump
MW-LSC-3	584.19	32.0	17.0	558.19-575.19	Yes	19.5	4.5	564.69	24.0	560.19	0	--
MW-LSC-4	582.37	32.0	10.0	554.37-564.37	Yes	12.0	15	570.37	27.0	555.37	24	TLM at bottom of Alluvium/DNAPL accumulated in sump
MW-LSC-5	582.72	42.0	10.0	549.72-559.72	Yes	10.0	20	572.72	30.0	552.72	1	TLM at bottom of Alluvium/DNAPL accumulated in sump
MW-BGL-1	582.17	28.0	10.0	557.17-567.17	Yes	8.0	0	574.17	8.0	574.17	0	--
MW-BGL-3	576.88	28.0	10.0	557.88-567.88	Yes	19.0	0	557.88	19.0	557.88	0	--
Existing Brownfield Site Monitoring Wells:												
1660-MW-3R	579.01	28.0	15.0	550.26-565.26	No	5.0	22.5	574.01	27.5	551.51	0	--
1660-MW-5R	578.02	28.0	15.0	550.27-565.27	No	4.5	21.5	573.52	26.0	552.02	18	DNAPL accumulated in screen set 2-ft into clay
1660-MW-7	577.64	25.3	10.0	552.34-562.34	No	6.0	--	--	>25.3	<552.34	1	DNAPL accumulated on bottom cap
1660-MW-8	578.27	32.0	15.0	547.02-562.02	No	10.0	22	568.27	32.0	546.27	32	DNAPL accumulated in screen set above clay
MW-100	578.82	29.0	15.0	550.18-565.18	No	16.8	6.7	562.02	23.5	555.32	29	DNAPL accumulated in screen set 5-ft into clay
MW-103	578.81	24.0	15.0	555.18-570.18	No	12.8	9.7	566.01	22.5	556.31	0	--
MW-106	579.84	24.0	15.0	556.26-571.26	No	9.3	10.7	570.54	20.0	559.84	0	--
1675-MW-1	578.10	32.0	15.0	546.60-561.60	No	4.0	14	574.10	18.0	560.10	0	--
1675-MW-2	580.58	15.0	10.0	567.08-577.08	No	5.0	7	575.58	12.0	568.58	0	--
57-71-MW-1	577.70	15.0	10.0	562.2-572.2	No	6.2	1	571.50	7.2	570.50	0	--
57-71-MW-2	578.33	15.0	10.0	563.8-573.8	No	3.3	1.5	575.03	4.8	573.50	0	--
57-71-MW-3	579.15	15.0	9.0	564.8-573.8	No	3.3	4.3	575.85	7.6	571.50	0	--
57-71-MW-4	582.75	30.0	20.0	553.0-573.0	No	1.8	0	580.95	1.8	581.00	0	--
57-71-MW-5	584.00	30.0	20.0	554.0-574.0	No	2.0	0	582.00	2.0	582.00	0	--
31-MW-1	581.20	20.2	10.0	561.20-571.20	No						0	*
31-MW-2	582.30	30.2	10.0	552.1-562.1	No						0	**
31-MW-3R	579.30	18.0	10.0	561.3-571.3	No	Geologic information not provided on monitoring well construction logs					0	--
31-MW-4R	580.90	20.0	10.0	560.90-570.90	No						0	--
31-MW-5	581.40	20.5	20.0	555.90-575.90	No						0	--

Notes:
1. measurements recorded on October 26, 2021
* 31-MW-1 -top of lacustrine clay from soil boring 31 -BH-2 = 19 ft bgs; ~562 fasl
** 31-MW-2 - Top of lacustrine clay from soil boring 31 -BH-4 = >16 ft bgs; estimated ~ 566 fasl

Table 1B. Stratigraphic Summary - Sediment Borings
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

Location	Top of Sediment Elevation (fasl)	Total Boring Depth below Sediment Surface (ft)	Unit Thickness (ft)		Top of Alluvium Elevation (fmsl)	Depth to Top of Lacustrine Clay (ft)	Top of Lacustrine Clay Elevation (fasl)	Notes/Observations
			Recent Fluvial Deposits	Historic Alluvial Deposits				
Sediment Borings								
SED-LSC-1	567.56	22.5	8.5	6.3	559.06	14.8	552.76	Petroleum-like odor/sheen/Suspect TLM
SED-LSC-2	568.06	20	8.2	5.3	559.86	13.5	554.56	Petroleum-like odor/sheen/Suspect TLM
SED-LSC-3	566.30	21.5	11.5	0.0	--	11.5	554.80	Petroleum-like and Coal Tar-like odors/sheen
SED-LSC-4	564.66	16	5.0	0.0	--	5.0	559.66	Coal tar-like odor
SED-LSC-5	565.16	17	6.0	0.0	--	6.0	559.16	Coal tar-like odor/sheen/Suspect TLM
SED-LSC-6	564.71	16	8.0	3.5	556.71	11.5	553.21	Coal tar-like odor/sheen/Suspect TLM
SED-LSC-7	565.81	16	6.0	5.0	559.81	11.0	554.81	Petroleum-like and Coal Tar-like odors/sheen/Suspect TLM
SED-LSC-8	566.56	12.2	9.0	1.0	557.56	10.0	556.56	Petroleum-like odor
SED-BGL-1	563.16	11.5	0.2	0.0	--	0.2	562.96	Septic-like odor

Notes:
-- unit not encountered

Table 2. Well Development Field Measured Parameters
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

Well ID	Development Date	Sampling Time	Total Volume Purged (g)	pH (standard units)	Specific Conductance (mS/cm)	Temperature (°C)	Turbidity (ntu)	Oxidation Reduction Potential (mV)	Dissolved Oxygen (ppm)	Comments
Groundwater Monitoring Wells										
MW-LSC-1	11/15/2021	8:38	17	15.8	6.75	2.14	137.8	1.61	37.1	Light brown color.
MW-LSC-2	11/15/2021	10:45	30	NRT	NRT	NRT	NRT	NRT	NRT	No Readings Take (NRT) due to presence of coal tar, sheens and odors. Light to medium sheen with light to moderate coal tar odor.
MW-LSC-3	11/15/2021	11:32	15	13.5	6.74	2.72	-1.6	1.54	Out of Range	Light gray. Dry at 15 gal. Let recharge 15 min before continuing.
MW-LSC-4	11/16/2021	8:08	10	NRT	NRT	NRT	NRT	NRT	NRT	NRT due to presence of coal tar, sheens and odors. Strong odor and heavy sheen, bailer stained heavily. Free product blebs in bailer.
MW-LSC-5	1/10/2022	9:40	20	7.22	2.19	15.7	25	115.6	0.82	clear, slight coal tar-like odor.
MW-BGL-1	11/16/2021	8:35	2	NRT	NRT	NRT	NRT	NRT	NRT	NRT due to very slow recovery (<0.1' in 30min) and purged dry at 2 gal.
MW-BGL-3	11/16/20201	9:50	17	10.6	7.46	9.78	131.6	7.39	Out of Range	Very silty and turbid, no sheens or odors. Dry at 17 gal.

Notes:
Values only represent the last data reading before termination of development.

Table 3. Groundwater and Surface Water Elevation Summary
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

Well ID	Top of Riser Elevation (fasl)	6/1/2017		8/24/2017		8/30/2017		1/16/2019		9/16/2020		9/17/2020		9/18/2020		9/21/2020	
		DTW (ft)	GW Elevation (ft)	DTW (ft)	GW Elevation (ft)	DTW (ft)	GW Elevation (ft)	DTW (ft)	GW Elevation (ft)	DTW (ft)	GW Elevation (ft)	DTW (ft)	GW Elevation (ft)	DTW (ft)	GW Elevation (ft)	DTW (ft)	GW Elevation (ft)
Ground Water Monitoring Locations																	
TPMW1	582.20		-		-	NA	-	10.03	571.14		-		-		-		-
1660-MW-1	578.79	7.90	570.89		-	7.50	571.29	7.70	571.09		-		-		-		-
1660-MW-2	577.65	8.30	569.35		-	6.30	571.35	5.90	571.75		-		-		-		-
1660-MW-3	577.32	5.60	571.72		-	6.10	571.22	6.15	571.17		-		-		-		-
1660-MW-3R	578.63		-		-		-		-		-		-		-		-
1660-MW-4	575.55	5.10	570.45		-	4.40	571.15	3.85	571.70		-		-		-		-
1660-MW-5	577.25	10.80	566.45		-	5.40	571.85	5.69	571.56		-		-		-		-
1660-MW-5R	577.61		-		-		-		-		-		-		-	5.89	571.72
1660-MW-6	577.20	6.10	571.10	6.40	570.80	6.50	570.70	6.12	571.08		-		-		-	8.68	568.52
1660-MW-7	578.34		-	7.00	571.34	7.10	571.24	6.56	571.78		-		-		-		-
1660-MW-8	580.80		-		-		-		-		-		-		-	9.94	570.86
1675-MW-1	577.51		-		-		-		-	3.21	574.30	4.63	572.88	5.29	572.22	5.37	572.14
1675-MW-2	580.29		-		-		-		-	3.95	576.34	3.95	576.34	3.95	576.34	3.95	576.34
MW-100	578.56		-		-		-		-		-		-		-		-
MW-103	578.37		-		-		-		-		-		-		-		-
MW-106	579.84		-		-		-		-		-		-		-		-
68-MW-2	585.55		-		-		-		-		-		-		-		-
68-MW-3	584.26		-		-		-		-		-		-		-		-
68-MW-4	579.22		-		-		-		-		-		-		-		-
31-MW-1	580.66		-		-		-		-		-		-		-		-
31-MW-2	581.59		-		-		-		-		-		-		-		-
31-MW-3	578.93		-		-		-		-		-		-		-		-
31-MW-4	580.60		-		-		-		-		-		-		-		-
57-MW-1	577.70		-		-		-		-		-		-		-		-
57-MW-2	578.33		-		-		-		-		-		-		-		-
57-MW-3	579.15		-		-		-		-		-		-		-		-
57-MW-4	582.75		-		-		-		-		-		-		-		-
57-MW-5	584.00		-		-		-		-		-		-		-		-
MW-BGL-1	581.74		-		-		-		-		-		-		-		-
MW-BGL-3	579.82		-		-		-		-		-		-		-		-
MW-LSC-1	585.69		-		-		-		-		-		-		-		-
MW-LSC-2	585.35		-		-		-		-		-		-		-		-
MW-LSC-3	586.95		-		-		-		-		-		-		-		-
MW-LSC-4	582.13		-		-		-		-		-		-		-		-
MW-LSC-5	582.48		-		-		-		-		-		-		-		-
Scajaquada Creek Surface Water Monitoring Locations																	
SC-West Ave[1]	583.61		-		-		-		-		-		-		-		-
SC-Bike Path[1]	578.96		-		-		-		-		-		-		-		-
SC-1660 [1]	577.09		-		-		-		-		-		-		-		-
Black Rock Channel	578.96		-		573.05 [4]		572.54		572.79		573.55		573.43		573.17		573.04

Notes:

[1] Surface Water Elevation in Scajaquada Creek

[2] NAPL thickness measured 11/12/21

[3] NAPL on inside of PVC casing prevents accurate measurment.

(4) Black Rock Channel surface water elevation data from https://waterdata.usgs.gov/ny/nwis/uv?site_no=04216218

Blank cells indicate no measurement was taken or no data are available.

-Monitoring well not installed or data not available.

NA- Measurement not available

Table 3. Groundwater and Surface Water Elevation Summary
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

Well ID	Top of Riser Elevation (fasl)	9/24/2020		10/27-28/2020		11/4/2020		1/14/2021		3/5/2021		4/30/2021		10/26/2021		
		DTW (ft)	GW Elevation (ft)	DTW (ft)	GW Elevation (ft)	DTW (ft)	GW Elevation (ft)	DTW (ft)	GW Elevation (ft)	DTW (ft)	GW Elevation (ft)	DTW (ft)	GW Elevation (ft)	DTW (ft)	GW Elevation (ft)	NAPL Thickness (inches)
Ground Water Monitoring Locations																
TPMW1	582.20		-		-		-	9.92	572.28	10.56	571.64	10.20	572.00	10.43	571.77	
1660-MW-1	578.79	8.46	570.33		-		-	8.23	570.56	8.19	570.60	7.83	570.96	7.50	571.29	
1660-MW-2	577.65	7.43	570.22		-		-	7.13	570.52	7.55	570.10	6.08	571.57	6.40	571.25	
1660-MW-3	577.32		-		-		-		-		-		-		-	
1660-MW-3R	578.63	6.26	572.37	6.42	572.21	6.32	572.31	6.33	572.30	6.70	571.93	6.32	572.31	6.60	572.03	
1660-MW-4	575.55		-		-		-		-		-		-		-	
1660-MW-5	577.25		-		-		-		-		-		-		-	
1660-MW-5R	577.61	5.55	572.06	5.90	571.71	5.81	571.80	5.56	572.05	5.93	571.68	5.61	572.00	5.95	571.66	18.00
1660-MW-6	577.20	8.48	568.72		-		-	8.51	568.69	8.55	568.65	8.37	568.83	8.75	568.45	
1660-MW-7	578.34	7.94	570.40		-		-	8.02	570.32	8.16	570.18	7.89	570.45	8.20	570.14	1.00
1660-MW-8	580.80	9.80	571.00	9.90	570.90	9.92	570.88	9.85	570.95	9.77	571.03	9.70	571.10	10.02	570.78	31.50
1675-MW-1	577.51	5.12	572.39	4.84	572.67	5.11	572.40	5.22	572.29	5.56	571.95	5.20	572.31	5.51	572.00	
1675-MW-2	580.29	4.00	576.29	3.68	576.61	3.61	576.68	3.61	576.68	3.43	576.86	3.44	576.85	3.35	576.94	
MW-100	578.56		-		-		-		-		-		-	5.46	573.10	28.80
MW-103	578.37		-		-		-		-		-		-	4.93	573.44	
MW-106	579.84		-		-		-		-		-		-	6.75	573.09	
68-MW-2	585.55		-		-		-		-		-		-		-	
68-MW-3	584.26		-		-		-		-		-		-	10.04	574.22	
68-MW-4	579.22		-		-		-		-		-		-	8.15	571.07	
31-MW-1	580.66		-		-		-		-		-		-		-	
31-MW-2	581.59		-		-		-		-		-		-	4.97	576.62	
31-MW-3	578.93		-		-		-		-		-		-		-	
31-MW-4	580.60		-		-		-		-		-		-	6.83	573.77	
57-MW-1	577.70		-		-		-		-		-		-	4.75	572.95	
57-MW-2	578.33		-		-		-		-		-		-		-	
57-MW-3	579.15		-		-		-		-		-		-	2.35	576.80	
57-MW-4	582.75		-		-		-		-		-		-	3.17	579.58	
57-MW-5	584.00		-		-		-		-		-		-		-	
MW-BGL-1	581.74		-		-		-		-		-		-		-	
MW-BGL-3	579.82		-		-		-		-		-		-		-	
MW-LSC-1	585.69		-		-		-		-		-		-		-	
MW-LSC-2	585.35		-		-		-		-		-		-		-	
MW-LSC-3	586.95		-		-		-		-		-		-		-	
MW-LSC-4	582.13		-		-		-		-		-		-		-	
MW-LSC-5	582.48		-		-		-		-		-		-		-	
Scajaquada Creek Surface Water Monitoring Locations																
SC-West Ave[(1)]	583.61		-		-		-	11.00	572.61	10.75	572.86	9.52	574.09	10.80	572.81	
SC-Bike Path[(1)]	578.96		-		-		-	6.06	572.90	6.22	572.74	5.00	573.96	6.20	572.76	
SC-1660 [(1)]	577.09		-		-		-	4.34	572.75		-	3.79	573.30		-	
Black Rock Channel	578.96		573.45		573.21		572.83		572.68		572.73		573.61		-	

Notes:

[(1)] Surface Water Elevation in Scajaquada Creek

[(2)] NAPL thickness measured 11/12/21

[(3)] NAPL on inside of PVC casing prevents accurate measurment.

(4) Black Rock Channel surface water elevation data from https://waterdata.usgs.gov/ny/nwis/uv?site_no=04216218

Blank cells indicate no measurement was taken or no data are available.

-Monitoring well not installed or data not available.

NA- Measurement not available

Table 3. Groundwater and Surface Water Elevation Summary
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

Well ID	Top of Riser Elevation (fasl)	11/23/2021			1/25/2022			3/29/2022		
		DTW (ft)	GW Elevation (ft)	NAPL Thickness (inches)[2]	DTW (ft)	GW Elevation (ft)	NAPL Thickness (inches)	DTW (ft)	GW Elevation (ft)	NAPL Thickness (inches)
Ground Water Monitoring Locations										
TPMW1	582.20	10.02	572.18		NA	-		10.37	571.83	
1660-MW-1	578.79	7.73	571.06		NA	-		NA	-	
1660-MW-2	577.65	6.19	571.46		NA	-		NA	-	
1660-MW-3	577.32		-			-			-	
1660-MW-3R	578.63	6.34	572.29		NA	-		NA	-	
1660-MW-4	575.55		-			-			-	
1660-MW-5	577.25		-			-			-	
1660-MW-5R	577.61	5.48	572.13	(3)	NA	-		NA	-	
1660-MW-6	577.20	8.43	568.77		8.71	568.49		8.76	568.44	
1660-MW-7	578.34	7.95	570.39	6"	8.26	570.08		8.31	570.03	
1660-MW-8	580.80	9.69	571.11	31"	10.12	570.68		10.06	570.74	
1675-MW-1	577.51	5.13	572.38		5.65	571.86		5.57	571.94	
1675-MW-2	580.29	3.43	576.86		3.51	576.78		3.39	576.90	
MW-100	578.56	5.43	573.13	28"	NA	-		5.62	572.94	
MW-103	578.37	4.69	573.68		NA	-		4.35	574.02	
MW-106	579.84	6.52	573.32		NA	-		6.77	573.07	
68-MW-2	585.55	9.61	575.94		9.69	575.86		9.98	575.57	
68-MW-3	584.26	10.26	574.00		NA	-		10.18	574.08	
68-MW-4	579.22	7.94	571.28		NA	-		8.92	570.30	
31-MW-1	580.66		-		NA	-		NA	-	
31-MW-2	581.59	5.10	576.49		NA	-		NA	-	
31-MW-3	578.93		-		NA	-		6.75	572.18	
31-MW-4	580.60	6.51	574.09		NA	-		NA	-	
57-MW-1	577.70	4.61	573.09		NA	-		NA	-	
57-MW-2	578.33		-		NA	-		NA	-	
57-MW-3	579.15	1.94	577.21		NA	-		NA	-	
57-MW-4	582.75	3.00	579.75		NA	-		4.53	578.22	
57-MW-5	584.00	2.91	581.09		NA	-		3.94	580.06	
MW-BGL-1	581.74	23.21	558.53		16.80	564.94		6.19	575.55	
MW-BGL-3	579.82	6.62	573.20		6.92	572.90		7.21	572.61	
MW-LSC-1	585.69	11.89	573.80		12.59	573.10		12.37	573.32	
MW-LSC-2	585.35	12.48	572.87	<1"	12.89	572.46		12.77	572.58	
MW-LSC-3	586.95	13.81	573.14		14.35	572.60		14.40	572.55	
MW-LSC-4	582.13	9.47	572.66	12"	9.91	572.22		9.82	572.31	
MW-LSC-5	582.48		-		10.33	572.15		NA	-	
Scajaquada Creek Surface Water Monitoring Locations										
SC-West Ave[1]	583.61	10.77	572.84		NA	-		10.95	572.66	
SC-Bike Path[1]	578.96	6.01	572.95		6.80	572.16		6.49	572.47	
SC-1660 [1]	577.09		-			-			-	
Black Rock Channel	578.96		-			-			-	

Notes:

[1] Surface Water Elevation in Scajaquada Creek

[2] NAPL thickness measured 11/12/21

[3] NAPL on inside of PVC casing prevents accurate measurment.

(4) Black Rock Channel surface water elevation data from https://waterdata.usgs.gov/ny/nwis/uv?site_no=04216218

Blank cells indicate no measurement was taken or no data are available.

-Monitoring well not installed or data not available.

NA- Measurement not available

Table 4. Sample Summary
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

Sample ID	Sample Type	Parameters	Date Collected
MW-LSC-1	Groundwater	1,2,3,5,6,7,13	1/18/2022
MW-LSC-2		1,2,3,5,6,7,13	1/18/2022
MW-LSC-3		1,2,3,5,6,7,13	1/18/2022
MW-LSC-4		1,2,3,5,6,7,13	1/19/2022
MW-LSC-5		1,2,3,5,6,7,13	1/19/2022
MW-BGL-1		1,2,3,5,6,7,13	1/19/2022
MW-BGL-3		1,2,3,5,6,7,13	1/19/2022
MW-LSC-1/13-15'	Soil Boring Samples	1,2,3,5,6,7	11/3/2021
MW-LSC-1/22-24'		1,2,3,5,6,7	11/3/2021
MW-LSC-2/18-20'		1,2,3,5,6,7	11/4/2021
MW-LSC-3/11-12'		1,2,3,5,6,7	11/8/2021
MW-LSC-3/22-24'		1,2,3,5,6,7	11/8/2021
MW-LSC-4/20-22'		1,2,3,5,6,7	11/5/2021
MW-LSC-4/24-28'		1,2,3,4,5,6,7	11/5/2021
MW-LSC-4 24-25'		4	11/5/2021
MW-LSC-5/18-22		1,2,3,5,6,7	1/4/2022
MW-LSC-5/26-30		1,2,3,5,6,7	1/4/2022
MW-BGL-1/16-20'		1,2,3,5,6,7	11/11/2021
MW-BGL-3/17-19'		1,2,3,4,5,6,7	11/9/2021
MW-BGL-318-19'		4	11/9/2021
SED-LSC-1/0-0.5'	Surface Sediment Samples	1,3,5,6,7,8,9,10,11,12	11/11/2021
SED-LSC-2/0-0.5'		1,3,5,6,7,8,9,10,11,12	11/11/2021
SED-LSC-3/0-0.5'		1,3,5,6,7,8,9,10,11,12	11/11/2021
SED-LSC-4/0-0.5'		1,3,5,6,7,8,9,10,11,12	11/11/2021
SED-LSC-5/0-0.5'		1,3,5,6,7,8,9,10,11,12	11/11/2021
SED-LSC-6/0-0.5'		1,3,5,6,7,8,9,10,11,12	11/3/2021
SED-LSC-7/0-0.5'		1,3,5,6,7,8,9,10,11,12	11/3/2021
SED-LSC-8/0-0.5'		1,3,5,6,7,8,9,10,11,12	11/3/2021
SED-BGL-1/0-0.5'		1,3,5,6,7,8,9,10,11,12	11/11/2021
SED-LSC-1/11-15'	Deeper Sediment Samples	1,2,3,5,6,7,12	11/9/2021
SED-LSC-1 20-21'		4	11/9/2021
SED-LSC-2/14-16'		1,2,3,5,6,7,12	11/9/2021
SED-LSC-2 13-14'		4	11/9/2021
SED-LSC-3/13-16'		1,2,3,5,6,7,12	11/9/2021
SED-LSC-3 14-15'		4	11/9/2021
SED-LSC-4/14-16'		1,2,3,5,6,7,12	11/10/2021
SED-LSC-4 13-14'		4	11/10/2021
SED-LSC-5/14-16'		1,2,3,5,6,7,12	11/10/2021
SED-LSC-5 14-15'		4	11/10/2021
SED-LSC-6/12-19.5'		1,2,3,5,6,7,12	11/3/2021
SED-LSC-6 12-19.5'		4	11/3/2021
SED-LSC-7/12-18'		1,2,3,5,6,7,12	11/2/2021
SED-LSC-7 14-16'		4	11/2/2021
SED-LSC-8/6.8-15'		1,2,3,5,6,7,12	11/1/2021
SED-LSC-8 6.8-15.0'		4	11/1/2021
SED-BGL-1/12-14'		1,2,3,5,6,7,12	11/10/2021

Table 4. Sample Summary
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

Sample ID	Sample Type	Parameters	Date Collected
MW-100	DNAPL Samples	4	10/27/2021
1660-MW-5R		4	10/27/2021
1660-MW-7		4	10/27/2021
1660-MW-8		4	10/27/2021
North Vault		4	10/15/2021
South Vault		4	10/15/2021
SED-EB-1	QA/QC Samples	1,2,3,5,6,7	11/15/2021
SB-EB-1		1,2,3,5,6,7	11/15/2021
MW-BGL-1/16-20'/MS/MSD		1,2,3,5,6,7	11/11/2021
MW-BGL-DUP		1,2,3,5,6,7	11/11/2021
SED-LSC-3/0-0.5'/MS/MSD		1,3,5,6,7,8,9,10,11,12	11/11/2021
SED-LSC-DUP-0-0.5'		1,3,5,6,7,8,9,10,11,12	11/11/2021
BSA-MH-WATER	Outfall samples	1,2,3,4,5,6,7,13	11/18/2021
BSA-VAULT DEBRIS		1,3,4,5,6,7,8,9,10,11,12	11/18/2021

*See sediment boring logs for analytical sample elevations.

Analytical Notes:

1- TCL VOCs EPA SW8260C/5035
2- TCL SVOCs EPA SW8270D
3- Total Cyanide EPA SW9012A
4- Forensic Analysis EPA SW8270M/8015M
5- Pesticides and Herbicides EPA SW8081B/8151A
6- PCBs EPA SW8082A
7- TAL Metals EPA SW6010B/6020/7470
8- TCL SVOCs / NOAA34 PAHs EPA SW8270D SIM
9- VPH MADEP/EPH NJ EPH R3
10- Ammonia EPA SWSM4500
11- Sulfide EPA SW9030B
12- Total Organic Carbon EPA SW9060A
13- PFAS EPA SW8327

Abbreviations

VOCs - Volatile Organic Compounds
SVOCs - Semi-volatile Organic Compounds
PCBs - Polychlorinated biphenyls
PAHs - Polycyclic Aromatic Hydrocarbons
VPH -Volatile Petroleum Hydrocarbons
EPH -Extractable Petroleum Hydrocarbons
PFAS - Per- and Polyfluoroalkyl Substances
TCL - Target Compound List
TAL - Target Analyte List

Table 5A. Land Subsurface Soil Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

							Task Description	Former Buffalo Gas Light Area						Lower Scajaquada Creek Area																			
							Sample Name	MW-BGL-1/16-20		MW-BGL-DUP		MW-BGL-3/17-19		MW-LSC-1/13-15		MW-LSC-1/22-24		MW-LSC-2/18-20		MW-LSC-3/11-12		MW-LSC-3/22-24		MW-LSC-4/20-22		MW-LSC-4/24-28		MW-LSC-5 (18-22)		MW-LSC-5 (26-30)	
							Start Depth	16		16		17		13		22		18		11		22		20		24		18		26	
							End Depth	20		20		19		15		24		20		12		24		22		28		22		30	
							Depth Unit	ft		ft		ft		ft		ft		ft		ft		ft		ft		ft		ft		ft	
							Sample Date	11/11/2021		11/11/2021		11/9/2021		11/3/2021		11/3/2021		11/4/2021		11/8/2021		11/8/2021		11/5/2021		11/5/2021		1/4/2022		1/4/2022	
							Parent Sample			MW-BGL-1/16-20																					
Analyte		Units	CAS No.	375-6.8(a) Unrestricted SCO	375-6.8(b) Restricted-Residential SCO	375-6.8(b) Protection of Eco Resources SCO	NY CP-51 RESTRICTED USE ECO SSCO																								
VOCs (SW 8260C)		ug/kg																													
	Acetone		67-64-1	50	100000	2200	NE	8.8	UJ	10	UJ	11	UJ	11	UJ	11	UJ	**31**		770	UJ	**81**	J	**47**		**28**	J	**130**	J	1,200	UJ
	Benzene		71-43-2	60	4800	70000	NE	0.44	U	0.5	U	0.55	U	0.56	UJ	0.54	UJ	0.54	U	**540**		0.61	U	**21**		**1,400**		**1.1**	J	**200**	
	Bromochloromethane		74-97-5	NE	NE	NE	NE	1.8	UJ	2	U	2.2	U	2.2	UJ	2.2	UJ	2.2	U	150	U	2.4	U	3.2	U	2	UJ	5.4	U	240	U
	Bromodichloromethane		75-27-4	NE	NE	NE	NE	0.44	U	0.5	U	0.55	U	0.56	UJ	0.54	UJ	0.54	U	38	U	0.61	U	0.81	U	0.51	UJ	1.4	U	61	U
	Bromoform		75-25-2	NE	NE	NE	NE	3.5	UJ	4	U	4.4	UJ	4.5	UJ	4.3	UJ	4.3	UJ	310	U	4.9	U	6.4	U	4.1	UJ	11	U	490	U
	Bromomethane		74-83-9	NE	NE	NE	NE	1.8	U	2	U	2.2	U	2.2	UJ	2.2	UJ	2.2	UJ	150	UJ	2.4	UJ	3.2	U	2	UJ	5.4	U	240	U
	Carbon disulfide		75-15-0	NE	NE	NE	NE	8.8	U	10	U	**9.2**	J	11	UJ	11	UJ	11	U	770	UJ	12	UJ	**24**		**16**	J	**24**	J	1,200	UJ
	Carbon tetrachloride		56-23-5	760	2400	NE	NE	0.88	U	1	U	1.1	U	1.1	UJ	1.1	UJ	1.1	U	77	U	1.2	U	1.6	U	1	UJ	2.7	U	120	U
	Chlorobenzene		108-90-7	1100	100000	40000	NE	0.44	UJ	0.5	U	0.55	U	0.56	UJ	0.54	UJ	0.54	U	38	U	0.61	U	0.81	U	0.51	UJ	1.4	U	61	U
	Chloroethane		75-00-3	NE	NE	NE	NE	1.8	U	2	U	2.2	U	2.2	UJ	2.2	UJ	2.2	UJ	150	UJ	2.4	UJ	3.2	U	2	UJ	5.4	U	240	U
	Chloroform (Trichloromethane)		67-66-3	370	49000	12000	NE	1.3	U	1.5	U	1.6	U	1.7	UJ	1.6	UJ	1.6	U	120	U	1.8	U	2.4	U	1.5	UJ	4	U	180	U
	Chloromethane		74-87-3	NE	NE	NE	NE	3.5	U	4	U	4.4	U	4.5	UJ	4.3	UJ	4.3	U	310	U	4.9	U	6.4	U	4.1	UJ	11	U	490	U
	Cyclohexane		110-82-7	NE	NE	NE	NE	8.8	U	10	U	11	U	11	UJ	11	UJ	11	U	770	U	12	U	16	U	**1.4**	J	27	U	1,200	U
	1,2-Dibromo-3-chloropropane		96-12-8	NE	NE	NE	NE	2.6	UJ	3	U	3.3	UJ	3.4	UJ	3.2	UJ	3.2	U	230	U	3.7	U	4.8	U	3.1	UJ	8.1	UJ	360	UJ
	Dibromochloromethane		124-48-1	NE	NE	NE	10000	0.88	U	1	U	1.1	U	1.1	UJ	1.1	UJ	1.1	U	77	U	1.2	U	1.6	U	1	UJ	2.7	U	120	U
	1,2-Dibromoethane (EDB)		106-93-4	NE	NE	NE	NE	0.88	UJ	1	U	1.1	U	1.1	UJ	1.1	UJ	1.1	UJ	77	U	1.2	U	1.6	U	1	UJ	2.7	U	120	U
	1,2-Dichlorobenzene (o-DCB)		95-50-1	1100	100000	NE	NE	1.8	UJ	2	U	2.2	U	2.2	UJ	2.2	UJ	2.2	U	150	U	2.4	U	3.2	U	2	UJ	5.4	U	240	U
	1,3-Dichlorobenzene (m-DCB)		541-73-1	2400	49000	NE	NE	1.8	UJ	2	U	2.2	U	2.2	UJ	2.2	UJ	2.2	U	150	U	2.4	U	3.2	U	2	UJ	5.4	U	240	U
	1,4-Dichlorobenzene (p-DCB)		106-46-7	1800	13000	20000	NE	1.8	UJ	2	U	2.2	U	2.2	UJ	2.2	UJ	2.2	U	150	U	2.4	U	3.2	U	**0.32**	J	5.4	U	240	U
	Dichlorodifluoromethane (Freon 12)		75-71-8	NE	NE	NE	NE	8.8	U	10	U	11	UJ	11	UJ	11	UJ	11	U	770	UJ	12	UJ	16	U	10	UJ	27	UJ	1,200	UJ
	1,1-Dichloroethane		75-34-3	270	26000	NE	NE	0.88	U	1	U	1.1	U	1.1	UJ	1.1	UJ	1.1	U	77	U	1.2	U	1.6	U	1	UJ	2.7	U	120	U
	1,2-Dichloroethane		107-06-2	20	3100	10000	NE	0.88	U	1	U	1.1	U	1.1	UJ	1.1	UJ	1.1	U	77	U	1.2	U	1.6	U	1	UJ	2.7	U	120	U
	1,1-Dichloroethene		75-35-4	330	100000	NE	NE	0.88	U	1	U	1.1	U	1.1	UJ	1.1	UJ	1.1	U	77	U	1.2	U	1.6	U	1	UJ	2.7	U	120	U
	cis-1,2-Dichloroethene		156-59-2	250	100000	NE	NE	0.88	U	1	U	1.1	U	1.1	UJ	1.1	UJ	1.1	U	77	U	1.2	U	**0.5**	J	**0.31**	J	2.7	U	120	U
	trans-1,2-Dichloroethene		156-60-5	190	100000	NE	NE	1.3	U	1.5	U	1.6	U	1.7	UJ	1.6	UJ	1.6	U	120	U	1.8	U	2.4	U	1.5	UJ	4	U	180	U
	1,2-Dichloropropane		78-87-5	NE	NE	NE	700000	0.88	U	1	U	1.1	U	1.1	UJ	1.1	UJ	1.1	U	77	U	1.2	U	1.6	U	1	UJ	2.7	U	120	U
	cis-1,3-Dichloropropene		10061-01-5	NE	NE	NE	NE	0.44	U	0.5	U	0.55	U	0.56	UJ	0.54	UJ	0.54	U	38	U	0.61	U	0.81	U	0.51	UJ	1.4	U	61	U
	trans-1,3-Dichloropropene		10061-02-6	NE	NE	NE	NE	0.88	UJ	1	U	1.1	U	1.1	UJ	1.1	UJ	1.1	U	77	U	1.2	U	1.6	U	1	UJ	2.7	U	120	U
	1,4-Dioxane		123-91-1	100	13000	100	NE		R		R		R		R		R		R		R		R		R		R		R	
	Ethylbenzene		100-41-4	1000	41000	NE	NE	0.88	U	1	U	**0.51**	J	1.1	UJ	1.1	UJ	1.1	U	**12,000**		1.2	U	**0.5**	J	**37,000**		2.7	U	**5,200**	
	2-Hexanone		591-78-6	NE	NE	NE	NE	8.8	UJ	10	U	11	U	11	UJ	11	UJ	11	UJ	770	U	12	U	16	U	10	UJ	27	U	1,200	U
	Isopropylbenzene		98-82-8	NE	NE	NE	NE	0.88	U	1	U	**1**	J	1.1	UJ	1.1	UJ	1.1	U	**950**		1.2	U	1.6	U	**1,900**		2.7	U	**1,600**	
	Methyl acetate		79-20-9	NE	NE	NE	NE	3.5	U	4	U	4.4	U	4.5	UJ	4.3	UJ	4.3	U	**280**	J	4.9	U	6.4	UJ	4.1	UJ	11	U	490	U
	Methyl ethyl ketone (2-Butanone)		78-93-3	120	100000	100000	NE	8.8	UJ	10	U	11	U	11	UJ	11	UJ	11	U	770	U	**15**		**8.7**	J	10	UJ	**28**	J	1,200	UJ
	Methyl tert-butyl ether (MTBE)		1634-04-4	930	100000	NE	NE	1.8	U	2	U	2.2	U	2.2	UJ	2.2	UJ	2.2	UJ	150	UJ	2.4	UJ	3.2	U	2	UJ	5.4	U	240	U
	4-Methyl-2-pentanone (MIBK)		108-10-1	NE	NE	NE	NE	8.8	UJ	10	U	11	U	11	UJ	11	UJ	11	U	770	U	12	U	16	U	10	UJ	27	U	1,200	U
	Methylcyclohexane		108-87-2	NE	NE	NE	NE	3.5	U	4	U	**1.4**	J	4.5	UJ	4.3	UJ	4.3	U	**78**	J	4.9	U	6.4	U	**10**	J	11	U	490	U
	Methylene chloride		75-09-2	50	100000	12000	NE	4.4	U	5	U	5.5	UJ	5.6	UJ	5.4	UJ	5.4	U	380	UJ	6.1	UJ	8.1	U	5.1	UJ	14	U	610	U
	Styrene		100-42-5	NE	NE	NE	300000	0.88	UJ	1	U	1.1	U	1.1	UJ	1.1	UJ	1.1	U	**150**		1.2	U	1.6	U	**420**		2.7	U	120	U
	1,1,2,2-Tetrachloroethane		79-34-5	NE	NE	NE	NE	0.44	UJ	0.5	U	0.55	U	0.56	UJ	0.54	UJ	0.54	U	38	UJ	0.61	UJ	0.81	U	0.51	UJ	1.4	U	61	U
	Tetrachloroethene (PCE)		127-18-4	1300	19000	2000	NE	0.44	U	0.5	U	0.55	U	0.56	UJ	0.54	UJ	0.54	U	**78**		0.61	U	0.81	U	0.51	UJ	1.4	U	61	U
	Toluene		108-88-3	700	100000	36000	NE	0.88	U	1	U	1.1	U	1.1	UJ	1.1	UJ	1.1	U	**1,600**	J	1.2	UJ	1.6	U	**4,100**		2.7	U	120	U
	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)		76-13-1	NE	NE	NE	NE	3.5	U	4	U	4.4	U	4.5	UJ	4.3	UJ	4.3	U	310	U	4.9	U	6.4	U	4.1	UJ	11	U	490	U
	1,2,3-Trichlorobenzene		87-61-6	NE	NE	NE	20000	1.8	UJ	2	U	2.2	U	2.2	UJ	2.2	UJ	2.2	U	150	U	2.4	U	3.2	U	2	UJ	5.4	UJ	240	UJ
	1,2,4-Trichlorobenzene		120-82-1	NE	NE	NE	20000	1.8	UJ	2	U	2.2	U	2.2	UJ	2.2	UJ	2.2	U	150	U	2.4	U	3.2	U	2	UJ	5.4	U	240	U
	1,1,1-Trichloroethane (TCA)		71-55-6	680	100000	NE	NE	0.44	U	0.5	U	0.55	U	0.56	UJ	0.54	UJ	0.54	U	38	U	0.61	U	0.81	U	0.51	UJ	1.4	U	61	U
	1,1,2-Trichloroethane		79-00-5	NE	NE	NE	NE	0.88	UJ	1	U	1.1	U	1.1	UJ	1.1	UJ	1.1	U	77	U	1.2	U	1.6	U	1	UJ	2.7	U	120	U
	Trichloroethene (TCE)		79-01-6	470	21000	2000	NE	0.44	U	0.5	U	0.55	U	0.56	UJ	0.54	UJ	0.54	U	38	U	0.61	U	0.81	U	0.51	UJ	1.4	U	61	U
	Trichlorofluoromethane (Freon 11)		75-69-4	NE	NE	NE	NE	3.5	U	4	U	4.4	U	4.5	UJ	4.3	UJ	4.3	U	310	U	4.9	U	6.4	U	4.1	UJ	11	U	490	U

Table 5A. Land Subsurface Soil Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

							Task Description	Former Buffalo Gas Light Area						Lower Scajaquada Creek Area																					
							Sample Name	MW-BGL-1/16-20		MW-BGL-DUP		MW-BGL-3/17-19		MW-LSC-1/13-15		MW-LSC-1/22-24		MW-LSC-2/18-20		MW-LSC-3/11-12		MW-LSC-3/22-24		MW-LSC-4/20-22		MW-LSC-4/24-28		MW-LSC-5 (18-22)		MW-LSC-5 (26-30)					
							Start Depth	16		16		17		13		22		18		11		22		20		24		18		26					
							End Depth	20		20		19		15		24		20		12		24		22		28		22		30					
							Depth Unit	ft		ft		ft		ft		ft		ft		ft		ft		ft		ft		ft		ft					
							Sample Date	11/11/2021		11/11/2021		11/9/2021		11/3/2021		11/3/2021		11/4/2021		11/8/2021		11/8/2021		11/5/2021		11/5/2021		1/4/2022		1/4/2022					
							Parent Sample			MW-BGL-1/16-20																									
Analyte		Units	CAS No.	375-6.8(a) Unrestricted SCO	375-6.8(b) Restricted-Residential SCO	375-6.8(b) Protection of Eco Resources SCO	NY CP-51 RESTRICTED USE ECO SSCO																												
	Vinyl chloride		75-01-4	20	900	NE	NE	0.88	U	1	U	1.1	U	1.1	UJ	1.1	UJ	1.1	U	77	U	1.2	U	1.6	U	1	UJ	2.7	U	120	U				
	o-Xylene		95-47-6	260	100000	260	NE	0.88	U	1	U	**0.74**	**J**	1.1	UJ	1.1	UJ	1.1	U	**3,100**		1.2	U	1.6	U	**10,000**		2.7	U	**1,800**					
	m/p-Xylene		179601-23-1	260	100000	260	NE	1.8	U	2	U	**1.6**	**J**	2.2	UJ	2.2	UJ	2.2	U	**7,100**		2.4	U	3.2	U	**20,000**		5.4	U	**1,200**					
SVOCs (SW 8270D)		ug/kg																															
	Acenaphthene		83-32-9	20000	100000	20000	NE	160	U	170	U	150	U	520	U	160	U	190	U	**11,000**		**260**		**320**		**31,000**		240	U	**2,300**					
	Acenaphthylene		208-96-8	100000	100000	NE	NE	160	U	170	U	150	U	520	U	160	U	190	U	**3,700**		**55**	**J**	230	U	**91,000**		240	U	210	U				
	Acetophenone		98-86-2	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	Anthracene		120-12-7	100000	100000	NE	NE	120	U	120	U	110	U	390	U	120	U	140	U	**7,000**		**340**		**830**		**56,000**		180	U	**140**	**J**				
	Atrazine		1912-24-9	NE	NE	NE	NE	160	U	170	U	150	U	520	U	160	U	190	U	1,700	U	210	U	230	U	3,600	U	240	U	210	U				
	Benzaldehyde		100-52-7	NE	NE	NE	NE	260	U	270	U	250	U	850	U	260	U	310	U	2,900	U	350	U	380	U	6,000	U	390	U	340	U				
	Benzo(a)anthracene		56-55-3	1000	1000	NE	NE	120	U	120	U	110	U	390	U	120	U	140	U	**3,700**		**480**		**160**	**J**	**29,000**		180	U	160	U				
	Benzo(b)fluoranthene		205-99-2	1000	1000	NE	NE	120	U	120	U	110	U	390	U	120	U	140	U	**2,300**		**390**		**120**	**J**	**18,000**		180	U	160	U				
	Benzo(k)fluoranthene		207-08-9	800	3900	NE	NE	120	U	120	U	110	U	390	U	120	U	140	U	**820**	**J**	**180**		170	U	**5,300**		180	U	160	U				
	Benzo(g,h,i)perylene		191-24-2	100000	100000	NE	NE	160	U	170	U	150	U	520	U	160	U	190	U	**1,400**	**J**	**160**	**J**	**160**	**J**	**9,800**		240	U	210	U				
	Benzo(a)pyrene		50-32-8	1000	1000	2600	NE	160	U	170	U	150	U	520	U	160	U	190	U	**3,200**		**340**		**130**	**J**	**26,000**		240	U	210	U				
	Biphenyl (1,1-Biphenyl)		92-52-4	NE	NE	NE	60000	450	U	470	U	430	U	1,500	U	450	U	540	U	**4,000**	**J**	600	U	**100**	**J**	**37,000**		680	U	**260**	**J**				
	Bis(2-chloroethoxy)methane		111-91-1	NE	NE	NE	NE	220	U	220	U	200	U	700	U	210	U	260	U	2,300	U	280	U	310	U	4,900	U	320	U	280	U				
	Bis(2-chloroethyl)ether		111-44-4	NE	NE	NE	NE	180	U	190	U	170	U	580	U	180	U	210	U	2,000	U	240	U	260	U	4,100	U	270	U	230	U				
	2,2-oxybis(1-Chloropropane)		108-60-1	NE	NE	NE	NE	240	UJ	250	UJ	230	UJ	780	UJ	240	UJ	280	U	2,600	UJ	320	UJ	340	UJ	5,500	UJ	360	U	310	U				
	Bis(2-ethylhexyl)phthalate		117-81-7	NE	NE	NE	239000	200	UJ	210	UJ	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	4-Bromophenyl phenyl ether		101-55-3	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	Butyl benzyl phthalate		85-68-7	NE	NE	NE	NE	200	UJ	210	UJ	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	Caprolactam		105-60-2	NE	NE	NE	NE	200	UJ	210	UJ	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	UJ	300	U	260	U				
	Carbazole		86-74-8	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	**540**	**J**	300	U	**38**	**J**				
	4-Chloro-3-methylphenol		59-50-7	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	4-Chloroaniline		106-47-8	NE	NE	NE	NE	200	UJ	210	UJ	190	U	650	UJ	200	UJ	240	U	2,200	U	260	U	280	U	4,600	U	300	UJ	260	UJ				
	2-Chloronaphthalene		91-58-7	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	2-Chlorophenol		95-57-8	NE	NE	NE	800	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	4-Chlorophenyl phenyl ether		7005-72-3	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	Chrysene		218-01-9	1000	3900	NE	NE	120	U	120	U	110	U	390	U	120	U	140	U	**3,300**		**400**		**210**		**22,000**		180	U	160	U				
	Dibenz(a,h)anthracene		53-70-3	330	330	NE	NE	120	U	120	U	110	U	390	U	120	U	140	U	**350**	**J**	**54**	**J**	170	U	**2,000**	**J**	180	U	160	U				
	Dibenzofuran		132-64-9	7000	59000	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	**1,400**	**J**	**130**	**J**	280	U	**8,900**		300	U	**140**	**J**				
	3,3-Dichlorobenzidine		91-94-1	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	UJ	260	UJ				
	2,4-Dichlorophenol		120-83-2	NE	NE	NE	20000	180	U	190	U	170	U	580	U	180	U	210	U	2,000	U	240	U	260	U	4,100	U	270	U	230	U				
	Diethyl phthalate		84-66-2	NE	NE	NE	100000	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	Dimethyl phthalate		131-11-3	NE	NE	NE	200000	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	2,4-Dimethylphenol		105-67-9	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	Di-n-butyl phthalate		84-74-2	NE	NE	NE	14	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	4,6-Dinitro-2-methylphenol		534-52-1	NE	NE	NE	NE	520	UJ	540	UJ	490	R	1,700	U	510	U	620	UJ	5,600	U	680	U	740	U	12,000	U	780	UJ	680	UJ				
	2,4-Dinitrophenol		51-28-5	NE	NE	NE	20000	960	UJ	1000	UJ	910	U	3,100	U	940	U	1,100	UJ	10,000	R	1,300	R	1,400	UJ	22,000	U	1,400	UJ	1,200	UJ				
	2,4-Dinitrotoluene		121-14-2	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	2,6-Dinitrotoluene		606-20-2	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	Di-n-octyl phthalate		117-84-0	NE	NE	NE	NE	200	UJ	210	UJ	190	UJ	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	Fluoranthene		206-44-0	100000	100000	NE	NE	120	U	120	U	110	U	390	U	120	U	140	U	**6,600**		**1000**		**490**		**52,000**		180	U	**33**	**J**				
	Fluorene		86-73-7	30000	100000	30000	NE	200	U	210	U	190	U	650	U	200	U	240	U	**5,800**		**300**		**810**		**53,000**		300	U	**500**					
	Hexachlorobenzene		118-74-1	330	1200	NE	NE	120	U	120	U	110	U	390	U	120	U	140	U	1,300	U	160	U	170	U	2,700	U	180	U	160	U				
	1,3-Hexachlorobutadiene (C-46)		87-68-3	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	Hexachlorocyclopentadiene		77-47-4	NE	NE	NE	10000	570	U	590	U	540	UJ	1,800	U	560	U	680	U	6,200	U	750	U	820	U	13,000	U	850	U	740	U				
	Hexachloroethane		67-72-1	NE	NE	NE	NE	160	U	170	U	150	U	520	U	160	U	190	U	1,700	U	210	U	230	U	3,600	U	240	U	210	U				
	Indeno(1,2,3-cd)pyrene		193-39-5	500	500	NE	NE	160	U	170	U	150	U	520	U	160	U	190	U	**1,300**	**J**	**190**	**J**	**78**	**J**	**9,000**		240	U	210	U				

Table 5A. Land Subsurface Soil Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

							Task Description	Former Buffalo Gas Light Area						Lower Scajaquada Creek Area																					
							Sample Name	MW-BGL-1/16-20		MW-BGL-DUP		MW-BGL-3/17-19		MW-LSC-1/13-15		MW-LSC-1/22-24		MW-LSC-2/18-20		MW-LSC-3/11-12		MW-LSC-3/22-24		MW-LSC-4/20-22		MW-LSC-4/24-28		MW-LSC-5 (18-22)		MW-LSC-5 (26-30)					
							Start Depth	16		16		17		13		22		18		11		22		20		24		18		26					
							End Depth	20		20		19		15		24		20		12		24		22		28		22		30					
							Depth Unit	ft		ft		ft		ft		ft		ft		ft		ft		ft		ft		ft		ft					
							Sample Date	11/11/2021		11/11/2021		11/9/2021		11/3/2021		11/3/2021		11/4/2021		11/8/2021		11/8/2021		11/5/2021		11/5/2021		1/4/2022		1/4/2022					
							Parent Sample			MW-BGL-1/16-20																									
	Analyte	Units	CAS No.	375-6.8(a) Unrestricted SCO	375-6.8(b) Restricted-Residential SCO	375-6.8(b) Protection of Eco Resources SCO	NY CP-51 RESTRICTED USE ECO SSCO																												
	Isophorone		78-59-1	NE	NE	NE	NE	180	U	190	U	170	U	580	U	180	U	210	U	2,000	U	240	U	260	U	4,100	U	270	U	230	U				
	2-Methylnaphthalene		91-57-6	NE	NE	NE	NE	240	U	250	U	**64**	**J**	780	U	240	U	280	U	**31,000**		**140**	**J**	**970**		**300,000**		360	U	**5,200**					
	2-Methylphenol (o-Cresol)		95-48-7	330	100000	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	3,4-Methylphenol (m,p-Cresol)		108394/106445	NE	NE	NE	NE	290	U	300	U	270	U	930	U	280	U	340	U	3,100	U	**86**	**J**	410	U	6,600	U	430	U	380	U				
	Naphthalene		91-20-3	12000	100000	NE	NE	200	U	210	U	**25**	**J**	650	U	200	U	240	U	**43,000**		**200**	**J**	**490**		**510,000**		300	U	**2,600**					
	2-Nitroaniline		88-74-4	NE	NE	NE	NE	200	UJ	210	UJ	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	3-Nitroaniline		99-09-2	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	4-Nitroaniline		100-01-6	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	Nitrobenzene		98-95-3	NE	NE	NE	40000	180	U	190	U	170	U	580	U	180	U	210	U	2,000	U	240	U	260	U	4,100	U	270	U	230	U				
	2-Nitrophenol		88-75-5	NE	NE	NE	7000	430	UJ	450	UJ	410	U	1,400	U	420	U	510	U	4,700	U	570	U	620	U	9,800	U	640	UJ	560	UJ				
	4-Nitrophenol		100-02-7	NE	NE	NE	7000	280	U	290	U	260	U	900	U	270	U	330	U	3,000	U	370	U	400	U	6,400	U	420	U	360	U				
	N-Nitrosodiphenylamine (NDFA)		86-30-6	NE	NE	NE	20000	160	U	170	U	150	U	520	U	160	U	190	U	1,700	U	210	U	230	U	3,600	U	240	U	210	U				
	N-Nitrosodi-n-propylamine (NDPA)		621-64-7	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	Pentachlorophenol		87-86-5	800	6700	800	NE	160	U	170	U	150	U	520	U	160	U	190	U	1,700	U	210	U	230	U	3,600	U	240	U	210	U				
	Phenanthrene		85-01-8	100000	100000	NE	NE	120	U	120	U	110	U	390	U	120	U	140	U	**22,000**		**920**		**2,400**		**200,000**		180	U	**720**					
	Phenol		108-95-2	330	100000	30000	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	Pyrene		129-00-0	100000	100000	NE	NE	120	U	120	U	110	U	390	U	120	U	140	U	**12,000**		**750**		**1,800**		**110,000**		180	U	**52**	**J**				
	1,2,4,5-Tetrachlorobenzene		95-94-3	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	2,3,4,6-Tetrachlorophenol		58-90-2	NE	NE	NE	NE	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	2,4,5-Trichlorophenol		95-95-4	NE	NE	NE	4000	200	U	210	U	190	U	650	U	200	U	240	U	2,200	U	260	U	280	U	4,600	U	300	U	260	U				
	2,4,6-Trichlorophenol		88-06-2	NE	NE	NE	NE	120	U	120	U	110	U	390	U	120	U	140	U	1,300	U	160	U	170	U	2,700	U	180	U	160	U				
	Total PAH (17) (ND=0)		TPAH17_ND0	NE	NE	NE	NE	ND		ND		**89**		ND		ND		ND		**158,470**		**6,159**		**8,968**		**1,524,100**		ND		**11,545**					
PESTICIDES (SW 8081B)		ug/kg																																	
	Aldrin		309-00-2	5	97	140	NE	1.89	U	1.9	U	1.8	R	2.15	U	1.86	U	2.24	U	10	UJ	24.1	UJ	13.6	U	10.8	U	2.92	U	2.43	U				
	alpha-BHC (Hexachlorocyclohexane)		319-84-6	20	480	40	NE	0.788	U	0.792	U	0.749	R	0.896	U	0.777	U	0.932	U	4.19	UJ	10	UJ	5.67	U	4.49	U	1.22	U	1.01	U				
	beta-BHC (beta-Hexachlorocyclohexane)		319-85-7	36	360	600	NE	1.89	U	1.9	U	1.8	R	2.15	U	1.86	U	2.24	U	10	UJ	24.1	UJ	13.6	U	10.8	U	2.92	U	2.43	U				
	gamma-BHC (gamma-Hexachlorocyclohexane) (		58-89-9	100	1300	6000	NE	0.788	U	0.792	U	0.749	R	0.896	U	0.777	U	0.932	U	4.19	UJ	10	UJ	5.67	U	4.49	U	1.22	U	1.01	U				
	delta-BHC (delta-Hexachlorocyclohexane)		319-86-8	40	100000	40	NE	1.89	U	1.9	U	1.8	R	2.15	U	1.86	U	2.24	U	10	UJ	24.1	UJ	13.6	U	10.8	U	2.92	U	2.43	U				
	Chlordane (Technical)		12789-03-6	NE	NE	NE	NE	15.8	U	15.8	U	15	R	17.9	U	15.5	U	18.6	U	83.8	UJ	200	UJ	113	U	89.9	U	24.3	U	20.2	U				
	alpha-chlordane		5103-71-9	94	4200	1300	NE	2.36	U	2.38	U	2.25	R	2.69	U	2.33	U	2.8	U	12.6	UJ	30.1	UJ	17	U	13.5	U	3.65	U	3.04	U				
	gamma-Chlordane		5103-74-2	NE	NE	NE	NE	2.36	U	2.38	U	2.25	R	2.69	U	2.33	U	2.8	U	12.6	UJ	30.1	UJ	17	U	13.5	U	3.65	U	3.04	U				
	4,4'-DDT (p,p'-DDT)		50-29-3	3.3	7900	3.3	NE	3.55	U	3.56	U	**15.3**	**J**	4.03	U	3.5	U	4.19	U	18.8	UJ	45.1	UJ	25.5	U	20.2	U	5.47	U	4.56	U				
	4,4'-DDE (p,p'-DDE)		72-55-9	3.3	8900	3.3	NE	1.89	U	1.9	U	1.8	R	2.15	U	1.86	U	2.24	U	10	UJ	24.1	UJ	13.6	U	10.8	U	2.92	U	2.43	U				
	4,4'-DDD (p,p-DDD)		72-54-8	3.3	13000	3.3	NE	1.89	U	1.9	U	1.8	R	2.15	U	1.86	U	2.24	U	10	UJ	24.1	UJ	13.6	U	10.8	U	2.92	U	2.43	U				
	Dieldrin		60-57-1	5	200	6	NE	1.18	U	1.19	U	1.12	R	1.34	U	1.16	U	1.4	U	6.28	UJ	15	UJ	8.5	U	6.74	U	1.82	U	1.52	U				
	alpha-Endosulfan (I)		959-98-8	2400	24000	NE	NE	1.89	U	1.9	U	1.8	R	2.15	U	1.86	U	2.24	U	10	UJ	24.1	UJ	13.6	U	10.8	U	2.92	U	2.43	U				
	beta-Endosulfan (II)		33213-65-9	2400	24000	NE	NE	1.89	U	1.9	U	1.8	R	2.15	U	1.86	U	2.24	U	10	UJ	24.1	UJ	13.6	U	10.8	U	2.92	U	2.43	U				
	Endosulfan sulfate		1031-07-8	2400	24000	NE	NE	0.788	U	0.792	U	0.749	R	0.896	U	0.777	U	0.932	U	4.19	UJ	10	UJ	**7.06**	**J**	4.49	U	1.22	U	1.01	U				
	Endrin		72-20-8	14	11000	14	NE	0.788	U	0.792	U	0.749	R	0.896	U	0.777	U	0.932	U	4.19	UJ	10	UJ	5.67	U	4.49	U	1.22	U	1.01	U				
	Endrin aldehyde		7421-93-4	NE	NE	NE	NE	2.36	U	2.38	U	2.25	R	2.69	U	2.33	U	2.8	U	12.6	UJ	30.1	UJ	17	U	13.5	U	3.65	U	3.04	U				
	Endrin ketone		53494-70-5	NE	NE	NE	NE	1.89	U	1.9	U	1.8	R	2.15	U	1.86	U	2.24	U	10	UJ	24.1	UJ	13.6	U	10.8	U	2.92	U	2.43	U				
	Heptachlor		76-44-8	42	2100	140	NE	0.946	U	0.95	U	0.899	R	1.07	U	0.932	U	1.12	U	5.03	UJ	12	UJ	6.8	U	5.39	U	1.46	U	1.22	U				
	Heptachlor epoxide		1024-57-3	NE	NE	NE	NE	3.55	U	3.56	U	3.37	R	4.03	U	3.5	U	4.19	U	18.8	UJ	45.1	UJ	25.5	U	20.2	U	5.47	U	4.56	U				
	Methoxychlor		72-43-5	NE	NE	NE	1200	3.55	UJ	3.56	UJ	3.37	R	4.03	U	3.5	U	4.19	UJ	18.8	UJ	45.1	UJ	25.5	U	20.2	U	5.47	U	4.56	U				
	Toxaphene		8001-35-2	NE	NE	NE	NE	35.5	U	35.6	U	33.7	R	40.3	U	35	U	41.9	U	188	UJ	451	UJ	255	U	202	U	54.7	U	45.6	U				
PCBs (SW 8082A)		ug/kg																																	
	Aroclor 1016		12674-11-2	NE	NE	NE	NE	39.9	U	40.6	U	38.8	UJ	43.3	UJ	38.7	UJ	47.3	U	41.6	UJ	52.9	U	57.3	U	44.5	U	59.4	U	51.3	U				
	Aroclor 1221		11104-28-2	NE	NE	NE	NE	39.9	U	40.6	U	38.8	UJ	43.3	UJ	38.7	UJ	47.3	U	41.6	UJ	52.9	U	57.3	U	44.5	U	59.4	U	51.3	U				
	Aroclor 1232		11141-16-5	NE	NE	NE	NE	39.9	U	40.6	U	38.8	UJ	43.3	UJ	38.7	UJ	47.3	U	41.6	UJ	52.9	U	57.3	U	44.5	U	59.4	U	51.3	U				
	Aroclor 1242		53469-21-9	NE	NE	NE	NE	39.9	U	40.6	U	38.8	UJ	43.3	UJ	38.7	UJ	47.3	U	41.6	UJ	52.9	U	57.3	U	44.5	U	59.4	U	51.3	U				

Table 5A. Land Subsurface Soil Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

							Task Description	Former Buffalo Gas Light Area						Lower Scajaquada Creek Area																			
							Sample Name	MW-BGL-1/16-20		MW-BGL-DUP		MW-BGL-3/17-19		MW-LSC-1/13-15		MW-LSC-1/22-24		MW-LSC-2/18-20		MW-LSC-3/11-12		MW-LSC-3/22-24		MW-LSC-4/20-22		MW-LSC-4/24-28		MW-LSC-5 (18-22)		MW-LSC-5 (26-30)	
							Start Depth	16		16		17		13		22		18		11		22		20		24		18		26	
							End Depth	20		20		19		15		24		20		12		24		22		28		22		30	
							Depth Unit	ft		ft		ft		ft		ft		ft		ft		ft		ft		ft		ft		ft	
							Sample Date	11/11/2021		11/11/2021		11/9/2021		11/3/2021		11/3/2021		11/4/2021		11/8/2021		11/8/2021		11/5/2021		11/5/2021		1/4/2022		1/4/2022	
							Parent Sample			MW-BGL-1/16-20																					
Analyte		Units	CAS No.	375-6.8(a) Unrestricted SCO	375-6.8(b) Restricted-Residential SCO	375-6.8(b) Protection of Eco Resources SCO	NY CP-51 RESTRICTED USE ECO SSCO																								
Aroclor 1248			12672-29-6	NE	NE	NE	NE	39.9	U	40.6	U	38.8	UJ	43.3	UJ	38.7	UJ	47.3	U	41.6	UJ	52.9	U	57.3	U	44.5	U	59.4	U	51.3	U
Aroclor 1254			11097-69-1	NE	NE	NE	NE	39.9	U	40.6	U	**65.9**	**J**	43.3	UJ	38.7	UJ	47.3	U	**7.38**	**J**	52.9	U	57.3	U	44.5	U	59.4	U	51.3	U
Aroclor 1260			11096-82-5	NE	NE	NE	NE	39.9	U	40.6	U	38.8	UJ	43.3	UJ	38.7	UJ	47.3	U	41.6	UJ	52.9	U	57.3	U	44.5	U	59.4	U	51.3	U
Aroclor 1262			37324-23-5	NE	NE	NE	NE	39.9	U	40.6	U	38.8	UJ	43.3	UJ	38.7	UJ	47.3	U	41.6	UJ	52.9	U	57.3	U	44.5	U	59.4	U	51.3	U
Aroclor 1268			11100-14-4	NE	NE	NE	NE	39.9	U	40.6	U	38.8	UJ	43.3	UJ	38.7	UJ	47.3	U	41.6	UJ	52.9	U	57.3	U	44.5	U	59.4	U	51.3	U
Total PCBs (Lab calculated)			1336-36-3	100	1000	1000	NE	39.9	U	40.6	U	**65.9**	**J**	43.3	UJ	38.7	UJ	47.3	U	**7.38**	**J**	52.9	U	57.3	U	44.5	U	59.4	U	51.3	U
METALS (SW 6010D/SW 7471B)		mg/kg																													
Aluminum			7429-90-5	NE	NE	NE	10000	**10,800**	**J**	**2,120**	**J**	**14,000**		**1,980**	**J**	**13,000**	**J**	**7,530**	**J**	**4,730**		**8,580**		**3,340**	**J**	**2,890**	**J**	**9,060**		**7,800**	
Antimony			7440-36-0	NE	NE	NE	12	4.73	UJ	**0.785**	**J**	22.7	U	5.18	U	4.57	U	5.55	U	4.92	U	6.39	U	6.46	U	5.28	U	7.08	UJ	5.94	UJ
Arsenic			7440-38-2	13	16	13	NE	**3.72**	**J**	**2.11**	**J**	**6.13**		**16.3**		**4.19**		**3.7**		**5.76**		**6.07**		**1.78**		**7.07**		**4.97**		**6.89**	
Barium			7440-39-3	350	400	433	NE	**106**	**J**	**60.9**	**J**	**126**		**19.3**		**58.4**		**38.7**		**55.5**		**65.2**		**40.6**		**18.3**		**38.3**		**82.2**	
Beryllium			7440-41-7	7.2	72	10	NE	**0.463**	**J**	**0.106**	**J**	**1.72**	**J**	**0.249**	**J**	**0.649**		**0.366**	**J**	**0.335**	**J**	**0.486**	**J**	**0.168**	**J**	**0.137**	**J**	**0.354**	**J**	**0.297**	**J**
Cadmium			7440-43-9	2.5	4.3	4	NE	**0.605**	**J**	**0.823**	**J**	4.54	UJ	**0.539**	**J**	**0.713**	**J**	**0.778**	**J**	**0.374**	**J**	**0.55**	**J**	**0.634**	**J**	**0.401**	**J**	1.42	UJ	1.19	UJ
Calcium			7440-70-2	NE	NE	NE	10000	**32,000**	**J**	**29,100**	**J**	**67,600**		**2,390**	**J**	**41,100**	**J**	**14,500**	**J**	**152,000**		**18,400**		**155,000**		**131,000**		**24,600**		**102,000**	
Chromium			7440-47-3	30	180	41	NE	**17.6**		**22.7**		**17.5**		**9.1**		**19.9**		**11.4**		**7.66**		**17.2**		**6.66**		**6.1**		**12.5**		**12.1**	
Cobalt			7440-48-4	NE	NE	NE	20	**9.7**	**J**	**1.91**	**J**	**8.54**	**J**	**10.3**		**11.2**		**6.86**		**4.13**		**8.86**		**3.26**		**3.05**		**8.33**		**7.23**	
Copper			7440-50-8	50	270	50	NE	**20**	**J**	**39.2**	**J**	**22.2**		**407**		**21.1**		**16.7**		**43**		**61.7**		**6.7**	**J**	**5.17**	**J**	**23.7**		**15.4**	
Iron			7439-89-6	NE	NE	NE	NE	**20,400**	**J**	**7,970**	**J**	**23,700**		**20,400**		**24,800**		**17,800**		**13,800**	**J**	**25,700**	**J**	**17,000**		**12,000**		**23,000**		**16,100**	
Lead			7439-92-1	63	400	63	NE	**9.53**	**J**	**1,920**	**J**	**20.8**	**J**	**15.1**		**10.1**		**8.71**		**43.9**		**126**		**5.04**	**J**	**6.45**	**J**	**10.4**		**11.6**	
Magnesium			7439-95-4	NE	NE	NE	NE	**13,200**	**J**	**5,910**	**J**	**17,300**		**1,240**	**J**	**13,400**	**J**	**6,280**	**J**	**56,900**		**9,040**		**10,400**	**J**	**45,800**	**J**	**8,940**		**34,200**	
Manganese			7439-96-5	1600	2000	1600	NE	**301**	**J**	**123**	**J**	**1,250**		**84.4**		**472**		**203**		**363**		**193**		**270**		**318**		**405**		**451**	
Mercury			7439-97-6	0.18	0.81	0.18	NE	0.079	U	0.079	U	0.073	U	0.086	U	0.077	U	**0.065**	**J**	**0.298**		**0.832**		0.108	U	0.087	U	0.118	U	0.099	U
Nickel			7440-02-0	30	310	30	NE	**23.6**	**J**	**7.01**	**J**	**17.9**		**23.3**		**23.7**		**17.2**		**9.21**		**21.3**		**6.5**		**5.46**		**21.2**		**15.8**	
Potassium			7440-09-7	NE	NE	NE	NE	**1,820**	**J**	**286**	**J**	**1,800**		**468**		**1,570**		**655**		**751**		**698**		**550**		**615**		**698**		**1,390**	
Selenium			7782-49-2	3.9	180	3.9	NE	1.89	U	1.94	U	**1.27**	**J**	2.07	U	**0.256**	**J**	**0.378**	**J**	**0.778**	**J**	**0.805**	**J**	**0.491**	**J**	2.11	U	2.83	U	2.38	U
Silver			7440-22-4	2	180	2	NE	0.946	U	0.969	U	4.54	U	1.04	U	0.915	U	1.11	U	0.984	U	1.28	U	1.29	U	1.06	U	1.42	U	1.19	U
Sodium			7440-23-5	NE	NE	NE	NE	**332**		**148**	**J**	908	U	**316**		**214**		**444**		**357**		**140**	**J**	**712**		**258**		**669**		**408**	
Thallium			7440-28-0	NE	NE	NE	5	1.89	UJ	1.94	UJ	9.08	UJ	2.07	U	**0.485**	**J**	2.22	U	**0.423**	**J**	**0.409**	**J**	**0.569**	**J**	**0.443**	**J**	2.83	U	2.38	U
Vanadium			7440-62-2	NE	NE	NE	39	**22.1**	**J**	**5.89**	**J**	**17.7**	**J**	**20.3**		**26**		**13.6**		**13.1**		**22.8**		**9.23**		**10.2**		**10.1**	**J**	**14.5**	**J**
Zinc			7440-66-6	109	10000	109	NE	**54.2**	**J**	**210**	**J**	**61**		**14.6**		**53.7**		**60.2**		**79.5**	**J**	**128**	**J**	**36.2**		**32.8**		**71.9**		**57**	
HERBICIDES (SW 8151A)		ug/kg																													
2,4-D (2,4-Dichlorophenoxyacetic acid)			94-75-7	NE	NE	NE	NE	202	UJ	203	UJ	190	U	220	U	198	U	234	U	212	U	262	U	282	U	227	U	299	U	261	U
2,4,5-T (2,4,5-Trichlorophenoxyacetic acid)			93-76-5	NE	NE	NE	NE	202	U	203	U	190	U	220	U	198	U	234	U	212	U	262	U	282	UJ	227	U	299	UJ	261	UJ
2,4,5-TP (Silvex)			93-72-1	3800	100000	NE	NE	202	U	203	U	190	U	220	U	198	U	234	U	212	U	262	U	282	U	227	U	299	U	261	U
GENERAL CHEMISTRY (SM 2540G)		%																													
Percent Solids			SOLIDS	NE	NE	NE	NE	**81.7**		**79.4**		**85.6**		**73.8**		**83.2**		**69.9**		**76.2**		**62.6**		**58.1**		**72.2**		**54.2**		**63.2**	
CYANIDES (SW 9012B)		mg/kg																													
Total Cyanide			57-12-5	27	27	NE	NE	1.1	UJ	1.2	UJ	**0.29**	**J**	**0.77**	**J**	1.1	UJ	1.3	UJ	**0.65**	**J**	1.6	UJ	1.7	UJ	1.4	UJ	1.8	UJ	1.5	UJ

Table 5A. Land Subsurface Soil Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

Notes:
Depths are measured from ground surface
mg/kg = milligrams/kilogram or parts per million (ppm)
% = Percent
ug/kg = micrograms per kilogram

PAH = Polycyclic Aromatic Hydrocarbon

PCB = Polychlorinated Biphenyl
SVOC = Semi-Volatile Organic Compound
VOC = Volatile Organic Compound

CAS No. = Chemical Abstracts Service Number
ND = Not Detected
NE = Not Established

6 NYCRR = New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York

Comparison of detected results are performed against one or more of the following NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCO)s: Unrestricted Use, Restricted-Residential, Protection of Ecological Resources

Bolding indicates a detected result concentration
Shading and bolding indicates that the detected concentration is above the NYSDOH guidance it was compared to.
Samples collected entirely below 15' bgs are not compared to Table 375-6.8(b) or CP-51 SSCOs.

Validation Qualifiers:
J = The result is an estimated value.
R = The result is rejected.
U = The result was not detected above the reporting limit.
UJ = The results was not detected at or above the reporting limit shown and the reporting limit is estimated.

1,4-Dioxane - Rejected Data

Due to the extremely low calibration response factor for 1,4-dioxane under Method 8260C, this compound is typically rejected during validation as it does not meet the minimal criteria set for accurate quantitation. Non-detect results for 1,4-dioxane are rejected as the presence or absence of the compound cannot be verified. Positive results are estimated due to the high variability inherent in the quantitation when using an extremely low response factor.

Table 5B. Groundwater Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

					Task Description	Former Buffalo Gas Light Area				Lower Scajaquada Creek Area					
					Sample Name	MW-BGL-1		MW-BGL-3		MW-LSC-1		MW-LSC-2		DUP	
					Sample Date	1/19/2022		1/19/2022		1/18/2022		1/18/2022		1/18/2022	
					Parent Sample									MW-LSC-2	
Analyte	**Units**	**CAS No. or Identifier**	**NY AWQS**	**NYSDEC Interim Guidance**											
VOCs (8260C)	ug/L														
Acetone		67-64-1	50*	NE	5	U	**3.2**	**J**	5	UJ	12	UJ	50	UJ
Benzene		71-43-2	1	NE	0.5	U	**0.73**		0.5	U	**540**	**J**	**990**	**J**
Bromochloromethane		74-97-5	5	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	U
Bromodichloromethane		75-27-4	50*	NE	0.5	U	0.5	U	0.5	U	1.2	U	5	U
Bromoform		75-25-2	50*	NE	2	U	2	U	2	UJ	5	UJ	20	UJ
Bromomethane		74-83-9	5	NE	2.5	UJ	2.5	UJ	2.5	UJ	6.2	UJ	25	UJ
Carbon disulfide		75-15-0	60*	NE	5	U	5	U	5	U	12	U	50	U
Carbon tetrachloride		56-23-5	5	NE	0.5	U	0.5	U	0.5	U	1.2	U	5	U
Chlorobenzene		108-90-7	5	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	U
Chloroethane		75-00-3	5	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	U
Chloroform (Trichloromethane)		67-66-3	7	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	U
Chloromethane		74-87-3	5	NE	2.5	U	2.5	U	2.5	UJ	6.2	UJ	25	U
Cyclohexane		110-82-7	NE	NE	10	U	**0.66**	**J**	10	U	25	U	100	U
1,2-Dibromo-3-chloropropane		96-12-8	0.04	NE	2.5	U	2.5	U	2.5	UJ	6.2	UJ	25	UJ
Dibromochloromethane		124-48-1	50*	NE	0.5	U	0.5	U	0.5	U	1.2	U	5	U
1,2-Dibromoethane (EDB)		106-93-4	0.0006	NE	2	U	2	U	2	U	5	U	20	U
1,2-Dichlorobenzene (o-DCB)		95-50-1	3	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	U
1,3-Dichlorobenzene (m-DCB)		541-73-1	3	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	U
1,4-Dichlorobenzene (p-DCB)		106-46-7	3	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	U
Dichlorodifluoromethane (Freon 12)		75-71-8	5	NE	5	UJ	5	UJ	5	UJ	12	UJ	50	UJ
1,1-Dichloroethane		75-34-3	5	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	U
1,2-Dichloroethane		107-06-2	0.6	NE	0.5	U	0.5	U	0.5	U	1.2	U	5	U
1,1-Dichloroethene		75-35-4	5	NE	0.5	U	0.5	U	0.5	U	1.2	U	5	U
cis-1,2-Dichloroethene		156-59-2	5	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	U
trans-1,2-Dichloroethene		156-60-5	5	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	U
1,2-Dichloropropane		78-87-5	1	NE	1	U	1	U	1	U	2.5	U	10	U
cis-1,3-Dichloropropene		10061-01-5	0.4	NE	0.5	U	0.5	U	0.5	U	1.2	U	5	U
trans-1,3-Dichloropropene		10061-02-6	0.4	NE	0.5	U	0.5	U	0.5	U	1.2	U	5	U
Ethylbenzene		100-41-4	5	NE	2.5	U	**2.1**	**J**	2.5	U	**1000**		**990**	
2-Hexanone		591-78-6	50*	NE	5	U	5	U	5	UJ	12	UJ	50	UJ
Isopropylbenzene		98-82-8	5	NE	2.5	U	2.5	U	2.5	U	**26**		**28**	
Methyl acetate		79-20-9	NE	NE	2	U	2	U	2	U	5	U	20	U
Methyl ethyl ketone (2-Butanone)		78-93-3	50*	NE	5	U	5	U	5	U	12	U	50	U
Methyl tert-butyl ether (MTBE)		1634-04-4	10*	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	U
4-Methyl-2-pentanone (MIBK)		108-10-1	NE	NE	5	U	5	U	5	U	12	U	50	UJ
Methylcyclohexane		108-87-2	NE	NE	10	U	10	U	10	UJ	25	UJ	100	UJ
Methylene chloride		75-09-2	5	NE	2.5	U	2.5	U	2.5	UJ	6.2	UJ	25	UJ

Table 5B. Groundwater Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

				Task Description	Former Buffalo Gas Light Area				Lower Scajaquada Creek Area					
				Sample Name	MW-BGL-1		MW-BGL-3		MW-LSC-1		MW-LSC-2		DUP	
				Sample Date	1/19/2022		1/19/2022		1/18/2022		1/18/2022		1/18/2022	
				Parent Sample									MW-LSC-2	
Analyte	**Units**	**CAS No. or Identifier**	**NY AWQS**	**NYSDEC Interim Guidance**										
Styrene		100-42-5	5	NE	2.5	U	2.5	U	2.5	U	**7.1**		25	U
1,1,2,2-Tetrachloroethane		79-34-5	5	NE	0.5	U	0.5	U	0.5	U	1.2	U	5	UJ
Tetrachloroethene (PCE)		127-18-4	5	NE	0.5	U	0.5	U	0.5	U	1.2	U	5	U
Toluene		108-88-3	5	NE	2.5	U	2.5	U	2.5	U	**560**	**J**	**420**	**J**
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)		76-13-1	5	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	U
1,2,3-Trichlorobenzene		87-61-6	5	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	UJ
1,2,4-Trichlorobenzene		120-82-1	5	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	UJ
1,1,1-Trichloroethane (TCA)		71-55-6	5	NE	2.5	U	2.5	U	2.5	U	6.2	U	25	U
1,1,2-Trichloroethane		79-00-5	1	NE	1.5	U	1.5	U	1.5	U	3.8	U	15	U
Trichloroethene (TCE)		79-01-6	5	NE	0.5	U	0.5	U	0.5	U	1.2	U	5	U
Trichlorofluoromethane (Freon 11)		75-69-4	5	NE	2.5	U	2.5	U	2.5	UJ	6.2	UJ	25	UJ
Vinyl chloride		75-01-4	2	NE	1	U	1	U	1	U	**0.52**	**J**	10	U
o-Xylene		95-47-6	5	NE	2.5	U	**0.74**	**J**	2.5	U	**240**		**220**	
m/p-Xylene		179601-23-1	5	NE	2.5	U	**1.2**	**J**	2.5	U	**500**		**420**	
SVOCs (8270D or DSIM)	ug/L													
Acenaphthene		83-32-9	20*	NE	0.1	U	**0.96**		0.1	U	**58**		**59**	
Acenaphthylene		208-96-8	NE	NE	**0.02**	**J**	**0.58**		0.1	U	**230**		**220**	
Acetophenone		98-86-2	NE	NE	5	U	5	U	5	U	**2.6**	**J**	**2.6**	**J**
Anthracene		120-12-7	50*	NE	0.1	U	**0.18**		0.1	U	**13**		**12**	
Atrazine		1912-24-9	7.5	NE	10	U	10	U	10	U	10	U	10	U
Benzaldehyde		100-52-7	NE	NE	5	U	5	U	5	U	5	U	5	U
Benzo(a)anthracene		56-55-3	0.002*	NE	**0.02**	**J**	**0.04**	**J**	0.1	U	**0.78**	**J**	**0.49**	**J**
Benzo(b)fluoranthene		205-99-2	0.002*	NE	**0.01**	**J**	**0.02**	**J**	0.1	U	**0.39**		**0.21**	
Benzo(k)fluoranthene		207-08-9	0.002*	NE	0.1	U	0.1	U	0.1	U	**0.1**		**0.06**	**J**
Benzo(g,h,i)perylene		191-24-2	NE	NE	0.1	U	**0.02**	**J**	0.1	U	**0.24**		**0.12**	
Benzo(a)pyrene		50-32-8	ND	NE	0.1	U	**0.02**	**J**	0.1	U	**0.59**	**J**	**0.29**	**J**
Biphenyl (1,1-Biphenyl)		92-52-4	5	NE	2	U	2	U	2	U	**54**		**53**	
Bis(2-chloroethoxy)methane		111-91-1	5	NE	5	U	5	U	5	U	5	U	5	U
Bis(2-chloroethyl)ether		111-44-4	1	NE	2	U	2	U	2	U	2	U	2	U
2,2-oxybis(1-Chloropropane)		108-60-1	5	NE	2	U	2	U	2	U	2	U	2	U
Bis(2-ethylhexyl)phthalate		117-81-7	5	NE	3	U	3	U	3	U	3	U	3	U
4-Bromophenyl phenyl ether		101-55-3	NE	NE	2	U	2	U	2	U	2	U	2	U
Butyl benzyl phthalate		85-68-7	50*	NE	5	U	5	U	5	UJ	5	UJ	5	UJ
Caprolactam		105-60-2	NE	NE	10	U	10	U	10	U	10	U	10	U
Carbazole		86-74-8	NE	NE	2	U	**0.76**	**J**	2	UJ	**2.4**	**J**	**2.5**	**J**
4-Chloro-3-methylphenol		59-50-7	NE	NE	2	U	2	U	2	U	2	U	2	U
4-Chloroaniline		106-47-8	5	NE	5	U	5	U	5	UJ	5	UJ	5	UJ
2-Chloronaphthalene		91-58-7	10*	NE	0.2	U	0.2	U	0.2	U	0.2	U	0.2	U

Table 5B. Groundwater Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

				Task Description	Former Buffalo Gas Light Area				Lower Scajaquada Creek Area					
				Sample Name	MW-BGL-1		MW-BGL-3		MW-LSC-1		MW-LSC-2		DUP	
				Sample Date	1/19/2022		1/19/2022		1/18/2022		1/18/2022		1/18/2022	
				Parent Sample									MW-LSC-2	
Analyte	Units	CAS No. or Identifier	NY AWQS	NYSDEC Interim Guidance										
2-Chlorophenol		95-57-8	NE	NE	2	U	2	U	2	U	2	U	2	U
4-Chlorophenyl phenyl ether		7005-72-3	NE	NE	2	U	2	U	2	U	2	U	2	U
Chrysene		218-01-9	0.002*	NE	0.1	U	**0.02**	**J**	0.1	U	**0.56**	**J**	**0.34**	**J**
Dibenz(a,h)anthracene		53-70-3	NE	NE	0.1	U	0.1	U	0.1	U	**0.05**	**J**	**0.03**	**J**
Dibenzofuran		132-64-9	NE	NE	2	U	2	U	2	U	**8.4**		**8.2**	
3,3-Dichlorobenzidine		91-94-1	5	NE	5	U	5	U	5	UJ	5	UJ	5	UJ
2,4-Dichlorophenol		120-83-2	5	NE	5	U	5	U	5	U	5	U	5	U
Diethyl phthalate		84-66-2	50*	NE	5	U	5	U	5	U	5	U	5	U
Dimethyl phthalate		131-11-3	50*	NE	5	U	5	U	5	U	5	U	5	U
2,4-Dimethylphenol		105-67-9	50*	NE	5	U	5	U	5	UJ	5	UJ	5	UJ
Di-n-butyl phthalate		84-74-2	50	NE	5	U	5	U	5	U	5	U	5	U
4,6-Dinitro-2-methylphenol		534-52-1	NE	NE	10	U	10	U	10	U	10	U	10	U
2,4-Dinitrophenol		51-28-5	10*	NE	20	U	20	U	20	U	20	U	20	U
2,4-Dinitrotoluene		121-14-2	5	NE	5	U	5	U	5	U	5	U	5	U
2,6-Dinitrotoluene		606-20-2	5	NE	5	U	5	U	5	U	5	U	5	U
Di-n-octyl phthalate		117-84-0	50*	NE	5	U	5	U	5	U	5	U	5	U
1,4-Dioxane		123-91-1	NE	0.35	0.144	U	**0.285**		0.15	U	**0.0798**	**J**	**0.065**	**J**
Fluoranthene		206-44-0	50*	NE	0.1	U	**0.18**	**J**	0.1	U	**4.5**		**4.1**	
Fluorene		86-73-7	50*	NE	0.1	U	**0.79**		0.1	U	**45**		**44**	
Hexachlorobenzene		118-74-1	0.04	NE	0.8	U	0.8	U	0.8	U	0.8	U	0.8	U
1,3-Hexachlorobutadiene (C-46)		87-68-3	0.5	NE	0.5	U	0.5	U	0.5	U	0.5	U	0.5	U
Hexachlorocyclopentadiene		77-47-4	5	NE	20	U	20	U	20	U	20	U	20	U
Hexachloroethane		67-72-1	5	NE	0.8	U	0.8	U	0.8	U	0.8	U	0.8	U
Indeno(1,2,3-cd)pyrene		193-39-5	0.002*	NE	0.1	U	**0.02**	**J**	0.1	U	**0.24**		**0.13**	
Isophorone		78-59-1	50*	NE	5	U	5	U	5	U	5	U	5	U
2-Methylnaphthalene		91-57-6	NE	NE	0.1	U	**4.9**		0.1	U	**960**		**960**	
2-Methylphenol (o-Cresol)		95-48-7	1	NE	5	U	**0.81**	**J**	5	U	**0.73**	**J**	**0.74**	**J**
3,4-Methylphenol (m,p-Cresol)		108394/106445	NE	NE	5	U	**1.4**	**J**	5	U	**1.2**	**J**	**1.1**	**J**
Naphthalene		91-20-3	10*	NE	0.1	U	**15**		0.1	U	**3,900**		**3,800**	
2-Nitroaniline		88-74-4	5	NE	5	U	5	U	5	U	5	U	5	U
3-Nitroaniline		99-09-2	5	NE	5	U	5	U	5	U	5	U	5	U
4-Nitroaniline		100-01-6	5	NE	5	U	5	U	5	U	5	U	5	U
Nitrobenzene		98-95-3	0.4	NE	2	U	2	U	2	UJ	2	UJ	2	UJ
2-Nitrophenol		88-75-5	NE	NE	10	U	10	U	10	UJ	10	UJ	10	UJ
4-Nitrophenol		100-02-7	NE	NE	10	U	10	U	10	U	10	U	10	U
N-Nitrosodiphenylamine (NDFA)		86-30-6	50*	NE	2	U	2	U	2	U	2	U	2	U
N-Nitrosodi-n-propylamine (NDPA)		621-64-7	NE	NE	5	U	5	U	5	U	5	U	5	U
Pentachlorophenol		87-86-5	1	NE	0.8	UJ	0.8	UJ	0.8	U	**0.17**	**J**	**0.16**	**J**

Table 5B. Groundwater Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

				Task Description	Former Buffalo Gas Light Area				Lower Scajaquada Creek Area					
				Sample Name	MW-BGL-1		MW-BGL-3		MW-LSC-1		MW-LSC-2		DUP	
				Sample Date	1/19/2022		1/19/2022		1/18/2022		1/18/2022		1/18/2022	
				Parent Sample									MW-LSC-2	
Analyte	**Units**	**CAS No. or Identifier**	**NY AWQS**	**NYSDEC Interim Guidance**										
Phenanthrene		85-01-8	50*	NE	0.1	U	**1**		0.1	U	**64**		**61**	
Phenol		108-95-2	1	NE	5	U	5	U	5	U	**3.3**	**J**	**4.4**	**J**
Pyrene		129-00-0	50*	NE	**0.03**	**J**	**0.23**		0.1	U	**8.2**		**7.3**	
1,2,4,5-Tetrachlorobenzene		95-94-3	5	NE	10	U	10	U	10	U	10	U	10	U
2,3,4,6-Tetrachlorophenol		58-90-2	NE	NE	5	U	5	U	5	U	5	U	5	U
2,4,5-Trichlorophenol		95-95-4	NE	NE	5	U	5	U	5	U	5	U	5	U
2,4,6-Trichlorophenol		88-06-2	NE	NE	5	U	5	U	5	U	5	U	5	U
Total PAH (17) (ND=0)		TPAH17_ND0	NE	NE	**0.08**		**24**		ND		**5,286**		**5,169**	
CYANIDES (9012B)	mg/L													
Total Cyanide		57-12-5	0.2	NE	0.005	UJ	**2.57**	**J**	0.005	U	0.005	U	**0.002**	**J**
METALS (6020B/7470A)	mg/L													
Aluminum		7429-90-5	NE	NE	**0.106**		**0.0736**		0.01	U	**0.0373**	**J**	**0.0262**	**J**
Antimony		7440-36-0	0.003	NE	0.004	U	**0.00107**	**J**	0.004	U	0.004	U	0.004	U
Arsenic		7440-38-2	0.025	NE	**0.00143**		**0.01161**		**0.00064**		**0.00074**		**0.00071**	
Barium		7440-39-3	1	NE	**0.03388**	**J**	**0.1054**	**J**	**0.03948**		**0.324**		**0.4215**	
Beryllium		7440-41-7	0.003*	NE	0.0005	U	0.0005	U	0.0005	U	0.0005	U	0.0005	U
Cadmium		7440-43-9	0.005	NE	0.0002	U	0.0002	U	0.0002	U	0.0002	U	0.0002	U
Calcium		7440-70-2	NE	NE	**306**		**989**		**106**		**164**		**159**	
Chromium		7440-47-3	0.05	NE	**0.00083**	**J**	**0.00093**	**J**	0.001	U	0.001	U	0.001	U
Cobalt		7440-48-4	NE	NE	**0.00335**	**J**	**0.00112**	**J**	**0.00046**	**J**	0.0005	U	0.0005	U
Copper		7440-50-8	0.2	NE	**0.00227**		**0.00238**		**0.00176**		0.001	U	0.001	U
Iron		7439-89-6	0.3	NE	**0.13**	**J**	**1.57**		**0.0648**	**J**	**3.97**		**3.62**	
Lead		7439-92-1	0.025	NE	**0.00036**	**J**	**0.00111**		0.001	U	0.001	U	0.001	U
Magnesium		7439-95-4	35*	NE	**1,070**		**41.3**		**58.1**		**37.8**		**37.5**	
Manganese		7439-96-5	0.3	NE	**0.1111**	**J**	**0.4066**		**0.3172**		**0.2505**		**0.2419**	
Mercury		7439-97-6	0.0007	NE	0.0002	U	0.0002	U	0.0002	U	0.0002	U	0.0002	U
Nickel		7440-02-0	0.1	NE	**0.00441**	**J**	**0.00159**	**J**	**0.00245**		0.002	U	0.002	U
Potassium		7440-09-7	NE	NE	**57.5**		**45.1**		**6.52**		**11.6**		**11**	
Selenium		7782-49-2	0.01	NE	0.005	U	0.005	U	**0.00199**	**J**	0.005	U	0.005	U
Silver		7440-22-4	0.05	NE	0.0004	U	0.0004	U	0.0004	U	0.0004	U	0.0004	U
Sodium		7440-23-5	20	NE	**465**		**1,860**		**216**		**968**		**969**	
Thallium		7440-28-0	0.0005*	NE	0.001	U	0.001	U	0.001	U	0.001	U	0.001	U
Vanadium		7440-62-2	NE	NE	**0.00299**	**J**	**0.00235**	**J**	**0.0017**	**J**	0.005	U	0.005	U
Zinc		7440-66-6	2*	NE	0.01032	U	0.01	U	0.01	U	0.01	U	0.01	U
PESTICIDES (8081B)	ug/L													
Aldrin		309-00-2	ND	NE	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ
alpha-BHC (Hexachlorocyclohexane)		319-84-6	0.01	NE	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ
beta-BHC (beta-Hexachlorocyclohexane)		319-85-7	0.04	NE	0.014	U	0.014	U	0.014	U	0.014	U	0.014	U

Table 5B. Groundwater Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

					Task Description	Former Buffalo Gas Light Area				Lower Scajaquada Creek Area					
					Sample Name	MW-BGL-1		MW-BGL-3		MW-LSC-1		MW-LSC-2		DUP	
					Sample Date	1/19/2022		1/19/2022		1/18/2022		1/18/2022		1/18/2022	
					Parent Sample									MW-LSC-2	
Analyte	**Units**	**CAS No. or Identifier**	**NY AWQS**	**NYSDEC Interim Guidance**											
gamma-BHC (gamma-Hexachlorocyclohexane) (Lindane)		58-89-9	0.05	NE	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ
delta-BHC (delta-Hexachlorocyclohexane)		319-86-8	0.04	NE	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ
Chlordane (Technical)		12789-03-6	NE	NE	0.143	UJ	0.143	UJ	0.143	UJ	0.143	UJ	0.143	UJ
alpha-chlordane		5103-71-9	NE	NE	0.014	UJ	0.014	UJ	0.014	U	0.014	U	0.014	U
gamma-Chlordane		5103-74-2	NE	NE	0.014	UJ	0.014	UJ	0.014	U	0.014	U	0.014	U
2,4-D (2,4-Dichlorophenoxyacetic acid)		94-75-7	50	NE										
4,4'-DDT (p,p'-DDT)		50-29-3	0.2	NE	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ
4,4'-DDE (p,p'-DDE)		72-55-9	0.2	NE	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ
4,4'-DDD (p,p-DDD)		72-54-8	0.3	NE	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ
Dieldrin		60-57-1	0.004	NE	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ
alpha-Endosulfan (I)		959-98-8	NE	NE	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ
beta-Endosulfan (II)		33213-65-9	NE	NE	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ
Endosulfan sulfate		1031-07-8	NE	NE	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ
Endrin		72-20-8	ND	NE	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ
Endrin aldehyde		7421-93-4	5	NE	0.029	U	0.029	U	0.029	U	0.029	U	0.029	U
Endrin ketone		53494-70-5	5	NE	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ	0.029	UJ
Heptachlor		76-44-8	0.04	NE	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ
Heptachlor epoxide		1024-57-3	0.03	NE	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ	0.014	UJ
Methoxychlor		72-43-5	35	NE	0.143	UJ	0.143	UJ	0.143	UJ	0.143	UJ	0.143	UJ
2,4,5-TP (Silvex)		93-72-1	0.26	NE										
Toxaphene		8001-35-2	0.06	NE	0.143	UJ	0.143	UJ	0.143	UJ	0.143	UJ	0.143	UJ
HERBICIDES (8151A)	ug/L													
2,4-D (2,4-Dichlorophenoxyacetic acid)		94-75-7	50	NE	10	U	10	U	10	U	10	U	10	U
2,4,5-T (2,4,5-Trichlorophenoxyacetic acid)		93-76-5	35	NE	2	U	2	U	2	UJ	2	UJ	2	UJ
2,4,5-TP (Silvex)		93-72-1	0.26	NE	2	U	2	U	2	U	2	U	2	U
PCBs (8082A)	ug/L													
Aroclor 1016		12674-11-2	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ
Aroclor 1221		11104-28-2	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ
Aroclor 1232		11141-16-5	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ
Aroclor 1242		53469-21-9	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ
Aroclor 1248		12672-29-6	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ
Aroclor 1254		11097-69-1	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ
Aroclor 1260		11096-82-5	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ
Aroclor 1262		37324-23-5	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ
Aroclor 1268		11100-14-4	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ
Total PCBs (Lab calculated)		1336-36-3	0.09	NE	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ	0.071	UJ
PFAS (LCMSMS-ID)	ng/L													

Table 5B. Groundwater Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

					Task Description	Former Buffalo Gas Light Area				Lower Scajaquada Creek Area					
					Sample Name	MW-BGL-1		MW-BGL-3		MW-LSC-1		MW-LSC-2		DUP	
					Sample Date	1/19/2022		1/19/2022		1/18/2022		1/18/2022		1/18/2022	
					Parent Sample									MW-LSC-2	
Analyte	Units	CAS No. or Identifier	NY AWQS	NYSDEC Interim Guidance											
N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)		2991-50-6	NE	NE	1.9	U	2.02	U	1.78	U	1.79	U		
N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)		2355-31-9	NE	NE	1.9	U	2.02	U	1.78	U	1.79	U		
Perfluorobutanesulfonic acid (PFBS)		375-73-5	NE	NE	1.9	U	**1.6**	**J**	1.78	U	**1.31**	**J**		
Perfluorobutanoic acid (PFBA)		375-22-4	NE	NE	1.9	U	2.02	U	1.78	U	**15.6**			
Perfluorodecanesulfonic acid (PFDS)		335-77-3	NE	NE	1.9	UJ	2.02	UJ	1.78	UJ	1.79	UJ		
Perfluorodecanoic acid (PFDA)		335-76-2	NE	NE	1.9	U	2.02	U	1.78	U	1.79	U		
Perfluorododecanoic acid (PFDoA)		307-55-1	NE	NE	1.9	U	2.02	U	1.78	U	1.79	U		
Perfluoroheptanesulfonic acid (PFHpS)		375-92-8	NE	NE	1.9	U	2.02	U	1.78	U	1.79	U		
Perfluoroheptanoic acid (PFHpA)		375-85-9	NE	NE	**0.668**	**J**	**5.66**	**J**	1.78	U	**9.74**			
Perfluorohexanoic acid (PFHxA)		307-24-4	NE	NE	**1.69**	**J**	**3.39**		1.78	U	**17.9**			
Perfluorooctanesulfonamide (FOSA)		754-91-6	NE	NE	1.9	U	2.02	U	1.78	U	1.79	U		
Perfluoropentanoic Acid (PFPeA)		2706-90-3	NE	NE	**1.97**	**J**	**31.9**		1.78	U	**34.8**			
Perfluorotetradecanoic acid (PFTA/PFTeDA)		376-06-7	NE	NE	1.9	U	2.02	U	1.78	U	1.79	U		
Perfluorotridecanoic acid (PFTriA/PFTrDA)		72629-94-8	NE	NE	1.9	U	**0.391**	**J**	1.78	U	1.79	U		
Perfluoroundecanoic acid (PFUnA)		2058-94-8	NE	NE	1.9	U	2.02	U	1.78	U	1.79	U		
Sodium 1H,1H,2H,2H-Perfluorodecane Sulfonate (8:2)		39108-34-4	NE	NE	1.9	UJ	2.02	UJ	1.78	UJ	1.79	UJ		
Sodium 1H,1H,2H,2H-Perfluorooctane Sulfonate (6:2)		27619-97-2	NE	NE	1.9	U	2.02	U	1.78	U	**1.44**	**J**		
Perfluorohexanesulfonic acid (PFHxS)		355-46-4	NE	NE	1.9	U	2.02	U	1.78	U	**2.1**			
Perfluorononanoic Acid (PFNA)		375-95-1	NE	NE	1.9	U	2.02	U	1.78	U	1.79	U		
Perfluorooctanesulfonic acid (PFOS)		1763-23-1	NE	10	1.9	U	2.02	U	1.78	U	**1.56**	**J**		
Perfluorooctanoic Acid (PFOA)		335-67-1	NE	10	**1.4**	**J**	**2.1**		1.78	U	**6.65**			
Total PFAS (ND=0)		TPFAS_ND0	NE	NE	**5.728**		**45.041**		ND		**91.1**			

Table 5B. Groundwater Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

					Task Description		Lower Scajaquada Creek Area							
					Sample Name	MW-LSC-3		MW-LSC-4		MW-LSC-5		DUP		BSA-MH-WATER	
					Sample Date	1/18/2022		1/19/2022		1/19/2022		1/19/2022		11/18/2021	
					Parent Sample							MW-LSC-5			
Analyte	**Units**	**CAS No. or Identifier**	**NY AWQS**	**NYSDEC Interim Guidance**											
VOCs (8260C)	ug/L														
Acetone		67-64-1	50*	NE	5	UJ	120	U	25	U			50	U
Benzene		71-43-2	1	NE	**4.5**		**560**		**420**				**130**	
Bromochloromethane		74-97-5	5	NE	2.5	U	62	U	12	U			25	U
Bromodichloromethane		75-27-4	50*	NE	0.5	U	12	U	2.5	U			5	U
Bromoform		75-25-2	50*	NE	2	UJ	50	U	10	U			20	U
Bromomethane		74-83-9	5	NE	2.5	UJ	62	UJ	12	UJ			25	UJ
Carbon disulfide		75-15-0	60*	NE	5	U	120	U	25	U			50	U
Carbon tetrachloride		56-23-5	5	NE	0.5	U	12	U	2.5	U			5	U
Chlorobenzene		108-90-7	5	NE	2.5	U	62	U	12	U			25	U
Chloroethane		75-00-3	5	NE	2.5	U	62	U	12	U			**60**	**J**
Chloroform (Trichloromethane)		67-66-3	7	NE	2.5	U	62	U	12	U			25	U
Chloromethane		74-87-3	5	NE	2.5	UJ	62	U	12	U			25	U
Cyclohexane		110-82-7	NE	NE	10	U	250	U	50	U			100	U
1,2-Dibromo-3-chloropropane		96-12-8	0.04	NE	2.5	UJ	62	U	12	U			25	U
Dibromochloromethane		124-48-1	50*	NE	0.5	U	12	U	2.5	U			5	U
1,2-Dibromoethane (EDB)		106-93-4	0.0006	NE	2	U	50	U	10	U			20	U
1,2-Dichlorobenzene (o-DCB)		95-50-1	3	NE	2.5	U	62	U	12	U			25	U
1,3-Dichlorobenzene (m-DCB)		541-73-1	3	NE	2.5	U	62	U	12	U			25	U
1,4-Dichlorobenzene (p-DCB)		106-46-7	3	NE	2.5	U	62	U	12	U			25	U
Dichlorodifluoromethane (Freon 12)		75-71-8	5	NE	5	UJ	120	UJ	25	UJ			50	U
1,1-Dichloroethane		75-34-3	5	NE	2.5	U	62	U	12	U			**140**	
1,2-Dichloroethane		107-06-2	0.6	NE	0.5	U	12	U	2.5	U			5	U
1,1-Dichloroethene		75-35-4	5	NE	0.5	U	12	U	2.5	U			**8.8**	
cis-1,2-Dichloroethene		156-59-2	5	NE	2.5	U	62	U	12	U			**1,700**	
trans-1,2-Dichloroethene		156-60-5	5	NE	2.5	U	62	U	12	U			25	U
1,2-Dichloropropane		78-87-5	1	NE	1	U	25	U	5	U			10	U
cis-1,3-Dichloropropene		10061-01-5	0.4	NE	0.5	U	12	U	2.5	U			5	U
trans-1,3-Dichloropropene		10061-02-6	0.4	NE	0.5	U	12	U	2.5	U			5	U
Ethylbenzene		100-41-4	5	NE	**13**		**3,300**		**400**				**180**	
2-Hexanone		591-78-6	50*	NE	5	UJ	120	U	25	U			50	UJ
Isopropylbenzene		98-82-8	5	NE	**0.76**	**J**	**64**		**64**				25	U
Methyl acetate		79-20-9	NE	NE	2	U	50	U	10	U			20	UJ
Methyl ethyl ketone (2-Butanone)		78-93-3	50*	NE	5	U	120	U	25	U			50	U
Methyl tert-butyl ether (MTBE)		1634-04-4	10*	NE	2.5	U	62	U	12	U			25	U
4-Methyl-2-pentanone (MIBK)		108-10-1	NE	NE	5	U	120	U	25	U			50	U
Methylcyclohexane		108-87-2	NE	NE	10	UJ	250	U	50	U			100	U
Methylene chloride		75-09-2	5	NE	2.5	UJ	62	U	12	U			25	U

Table 5B. Groundwater Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

				Task Description	Lower Scajaquada Creek Area									
				Sample Name	MW-LSC-3		MW-LSC-4		MW-LSC-5		DUP		BSA-MH-WATER	
				Sample Date	1/18/2022		1/19/2022		1/19/2022		1/19/2022		11/18/2021	
				Parent Sample							MW-LSC-5			
Analyte	**Units**	**CAS No. or Identifier**	**NY AWQS**	**NYSDEC Interim Guidance**										
Styrene		100-42-5	5	NE	2.5	U	**30**	**J**	12	U			25	U
1,1,2,2-Tetrachloroethane		79-34-5	5	NE	0.5	U	12	U	2.5	U			5	U
Tetrachloroethene (PCE)		127-18-4	5	NE	0.5	U	12	U	2.5	U			5	U
Toluene		108-88-3	5	NE	**2.2**	**J**	**700**		**5.7**	**J**			**66**	
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)		76-13-1	5	NE	2.5	U	62	U	12	U			25	U
1,2,3-Trichlorobenzene		87-61-6	5	NE	2.5	U	62	U	12	U			25	UJ
1,2,4-Trichlorobenzene		120-82-1	5	NE	2.5	U	62	U	12	U			25	U
1,1,1-Trichloroethane (TCA)		71-55-6	5	NE	2.5	U	62	U	12	U			**53**	
1,1,2-Trichloroethane		79-00-5	1	NE	1.5	U	38	U	7.5	U			15	U
Trichloroethene (TCE)		79-01-6	5	NE	0.5	U	12	U	2.5	U			5	U
Trichlorofluoromethane (Freon 11)		75-69-4	5	NE	2.5	UJ	62	U	12	U			25	U
Vinyl chloride		75-01-4	2	NE	1	U	**3.8**	**J**	**0.81**	**J**			**280**	
o-Xylene		95-47-6	5	NE	**6.2**		**740**		**140**				**47**	
m/p-Xylene		179601-23-1	5	NE	**7.8**		**1,400**		**72**				**68**	
SVOCs (8270D or DSIM)	ug/L													
Acenaphthene		83-32-9	20*	NE	**4.9**		**7.9**		**150**				**4.6**	
Acenaphthylene		208-96-8	NE	NE	**1**		**23**		**60**				**0.2**	
Acetophenone		98-86-2	NE	NE	5	U	5	U	5	U			5	U
Anthracene		120-12-7	50*	NE	**0.45**		**2.8**		**9.6**				**1.3**	
Atrazine		1912-24-9	7.5	NE	10	U	10	U	10	U			10	U
Benzaldehyde		100-52-7	NE	NE	5	U	5	U	5	R			5	U
Benzo(a)anthracene		56-55-3	0.002*	NE	**0.05**	**J**	**1.1**	**J**	4	U			**0.17**	
Benzo(b)fluoranthene		205-99-2	0.002*	NE	**0.02**	**J**	**0.46**	**J**	4	U			**0.32**	
Benzo(k)fluoranthene		207-08-9	0.002*	NE	**0.01**	**J**	2.5	U	4	U			**0.09**	**J**
Benzo(g,h,i)perylene		191-24-2	NE	NE	0.1	U	2.5	U	4	U			**0.19**	
Benzo(a)pyrene		50-32-8	ND	NE	**0.02**	**J**	**0.74**	**J**	4	U			**0.13**	
Biphenyl (1,1-Biphenyl)		92-52-4	5	NE	**0.53**	**J**	**72**		**36**	**J**			**5.3**	
Bis(2-chloroethoxy)methane		111-91-1	5	NE	5	U	5	U	5	U			5	U
Bis(2-chloroethyl)ether		111-44-4	1	NE	2	U	2	U	2	U			2	U
2,2-oxybis(1-Chloropropane)		108-60-1	5	NE	2	U	2	U	2	U			2	U
Bis(2-ethylhexyl)phthalate		117-81-7	5	NE	3	U	3	U	3	U			**1.8**	**J**
4-Bromophenyl phenyl ether		101-55-3	NE	NE	2	U	2	U	2	U			2	U
Butyl benzyl phthalate		85-68-7	50*	NE	5	UJ	5	U	5	U			5	U
Caprolactam		105-60-2	NE	NE	10	U	10	U	10	U			10	U
Carbazole		86-74-8	NE	NE	2	UJ	**7.9**		**3.6**				**0.68**	**J**
4-Chloro-3-methylphenol		59-50-7	NE	NE	2	U	2	U	2	U			2	U
4-Chloroaniline		106-47-8	5	NE	5	UJ	5	U	5	U			5	U
2-Chloronaphthalene		91-58-7	10*	NE	0.2	U	5	U	8	U			0.2	U

Table 5B. Groundwater Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

				Task Description	Lower Scajaquada Creek Area									
				Sample Name	MW-LSC-3		MW-LSC-4		MW-LSC-5		DUP		BSA-MH-WATER	
				Sample Date	1/18/2022		1/19/2022		1/19/2022		1/19/2022		11/18/2021	
				Parent Sample							MW-LSC-5			
Analyte	Units	CAS No. or Identifier	NY AWQS	NYSDEC Interim Guidance										
2-Chlorophenol		95-57-8	NE	NE	2	U	2	U	2	U			2	U
4-Chlorophenyl phenyl ether		7005-72-3	NE	NE	2	U	2	U	2	U			2	U
Chrysene		218-01-9	0.002*	NE	**0.02**	**J**	**0.59**	**J**	4	U			**0.25**	
Dibenz(a,h)anthracene		53-70-3	NE	NE	0.1	U	2.5	U	4	U			**0.04**	**J**
Dibenzofuran		132-64-9	NE	NE	2	U	**13**		**8.3**				2	U
3,3-Dichlorobenzidine		91-94-1	5	NE	5	UJ	5	U	5	UJ			5	U
2,4-Dichlorophenol		120-83-2	5	NE	5	U	5	U	5	U			5	U
Diethyl phthalate		84-66-2	50*	NE	5	U	5	U	5	U			5	U
Dimethyl phthalate		131-11-3	50*	NE	5	U	5	U	5	U			5	U
2,4-Dimethylphenol		105-67-9	50*	NE	5	UJ	5	U	5	U			5	U
Di-n-butyl phthalate		84-74-2	50	NE	5	U	5	U	5	U			5	U
4,6-Dinitro-2-methylphenol		534-52-1	NE	NE	10	U	10	U	10	U			10	U
2,4-Dinitrophenol		51-28-5	10*	NE	20	U	20	U	20	U			20	U
2,4-Dinitrotoluene		121-14-2	5	NE	5	U	5	U	5	U			5	U
2,6-Dinitrotoluene		606-20-2	5	NE	5	U	5	U	5	U			5	U
Di-n-octyl phthalate		117-84-0	50*	NE	5	U	5	U	5	U			5	U
1,4-Dioxane		123-91-1	NE	0.35	**0.0785**	**J**	**0.343**		**4.23**				**12,200**	
Fluoranthene		206-44-0	50*	NE	**0.31**		2.5	U	4	U			**1**	
Fluorene		86-73-7	50*	NE	**2.1**		**7.5**		**41**				**6**	
Hexachlorobenzene		118-74-1	0.04	NE	0.8	U	20	U	32	U			0.8	U
1,3-Hexachlorobutadiene (C-46)		87-68-3	0.5	NE	0.5	U	12	U	20	U			0.5	U
Hexachlorocyclopentadiene		77-47-4	5	NE	20	U	20	U	20	U			20	U
Hexachloroethane		67-72-1	5	NE	0.8	U	20	U	32	U			**0.13**	**J**
Indeno(1,2,3-cd)pyrene		193-39-5	0.002*	NE	0.1	U	2.5	U	4	U			**0.21**	
Isophorone		78-59-1	50*	NE	5	U	5	U	5	U			5	U
2-Methylnaphthalene		91-57-6	NE	NE	**7.8**		**140**		**620**				**0.09**	**J**
2-Methylphenol (o-Cresol)		95-48-7	1	NE	5	U	**3.5**	**J**	5	U			5	U
3,4-Methylphenol (m,p-Cresol)		108394/106445	NE	NE	5	U	**2.8**	**J**	**0.83**	**J**			5	U
Naphthalene		91-20-3	10*	NE	**39**		**930**		**1,700**				**0.08**	**J**
2-Nitroaniline		88-74-4	5	NE	5	U	5	U	5	U			5	U
3-Nitroaniline		99-09-2	5	NE	5	U	5	U	5	U			5	U
4-Nitroaniline		100-01-6	5	NE	5	U	5	U	5	U			5	U
Nitrobenzene		98-95-3	0.4	NE	2	UJ	2	U	2	U			2	U
2-Nitrophenol		88-75-5	NE	NE	10	UJ	10	U	10	U			10	U
4-Nitrophenol		100-02-7	NE	NE	10	U	10	U	10	U			10	U
N-Nitrosodiphenylamine (NDFA)		86-30-6	50*	NE	2	U	2	U	2	U			2	U
N-Nitrosodi-n-propylamine (NDPA)		621-64-7	NE	NE	5	U	5	U	5	U			5	U
Pentachlorophenol		87-86-5	1	NE	0.8	U	20	UJ	32	UJ			0.8	U

Table 5B. Groundwater Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

				Task Description	Lower Scajaquada Creek Area									
				Sample Name	MW-LSC-3		MW-LSC-4		MW-LSC-5		DUP		BSA-MH-WATER	
				Sample Date	1/18/2022		1/19/2022		1/19/2022		1/19/2022		11/18/2021	
				Parent Sample							MW-LSC-5			
Analyte	**Units**	**CAS No. or Identifier**	**NY AWQS**	**NYSDEC Interim Guidance**										
Phenanthrene		85-01-8	50*	NE	**1.9**		**13**		**49**				**7**	
Phenol		108-95-2	1	NE	5	U	**5**		**0.7**	**J**			**8.6**	
Pyrene		129-00-0	50*	NE	**0.39**		**3.8**		**4**				**1.3**	
1,2,4,5-Tetrachlorobenzene		95-94-3	5	NE	10	U	10	U	10	U			10	U
2,3,4,6-Tetrachlorophenol		58-90-2	NE	NE	5	U	5	U	5	U			5	U
2,4,5-Trichlorophenol		95-95-4	NE	NE	5	U	5	U	5	U			5	U
2,4,6-Trichlorophenol		88-06-2	NE	NE	5	U	5	U	5	U			5	U
Total PAH (17) (ND=0)		TPAH17_ND0	NE	NE	**58**		**1,131**		**2,634**				23	
CYANIDES (9012B)	mg/L													
Total Cyanide		57-12-5	0.2	NE	**0.004**	**J**	**0.004**	**J**	**0.004**	**J**			**0.002**	**J**
METALS (6020B/7470A)	mg/L													
Aluminum		7429-90-5	NE	NE	**0.0296**	**J**	**0.114**		**0.03**				**2.59**	
Antimony		7440-36-0	0.003	NE	0.004	U	0.004	U	**0.00046**	**J**			**0.00331**	**J**
Arsenic		7440-38-2	0.025	NE	**0.00306**		**0.00027**	**J**	**0.0007**				**0.00217**	
Barium		7440-39-3	1	NE	**0.2552**		**0.1568**		**0.2859**				**0.1282**	
Beryllium		7440-41-7	0.003*	NE	0.0005	U	0.0005	U	0.0005	U			**0.00013**	**J**
Cadmium		7440-43-9	0.005	NE	0.0002	U	0.0002	U	0.0002	U			**8.00E-05**	**J**
Calcium		7440-70-2	NE	NE	**260**		**114**		**193**				**99.7**	
Chromium		7440-47-3	0.05	NE	**0.00034**	**J**	**0.00045**	**J**	**0.00027**	**J**			**0.00568**	
Cobalt		7440-48-4	NE	NE	**0.00184**	**J**	**0.00016**	**J**	**0.0002**	**J**			**0.00197**	
Copper		7440-50-8	0.2	NE	0.001	U	0.001	U	0.001	U			**0.01747**	
Iron		7439-89-6	0.3	NE	**11.3**		**0.239**	**J**	**0.854**				**4.21**	
Lead		7439-92-1	0.025	NE	0.001	U	0.001	U	0.001	U			**0.02769**	
Magnesium		7439-95-4	35*	NE	**52.7**		**22.8**		**59.9**				**20.8**	
Manganese		7439-96-5	0.3	NE	**0.4426**		**0.1539**		**0.6083**				**0.2258**	
Mercury		7439-97-6	0.0007	NE	0.0002	U	0.0002	U	0.0002	U			0.0002	U
Nickel		7440-02-0	0.1	NE	**0.00099**	**J**	0.002	U	0.002	U			**0.0057**	
Potassium		7440-09-7	NE	NE	**8**		**4.79**		**3.89**				**7.5**	
Selenium		7782-49-2	0.01	NE	0.005	U	0.005	U	0.005	U			0.005	U
Silver		7440-22-4	0.05	NE	0.0004	U	0.0004	U	0.0004	U			**0.00017**	**J**
Sodium		7440-23-5	20	NE	**380**		**182**		**332**				**157**	
Thallium		7440-28-0	0.0005*	NE	0.001	U	0.001	U	0.001	U			0.001	U
Vanadium		7440-62-2	NE	NE	0.005	U	**0.00184**	**J**	0.005	U			**0.00564**	
Zinc		7440-66-6	2*	NE	0.01	U	0.0114	U	0.01	U			**0.07793**	
PESTICIDES (8081B)	ug/L													
Aldrin		309-00-2	ND	NE	0.014	UJ	0.714	U	0.014	UJ			0.014	U
alpha-BHC (Hexachlorocyclohexane)		319-84-6	0.01	NE	0.014	UJ	0.714	U	0.014	UJ			0.014	U
beta-BHC (beta-Hexachlorocyclohexane)		319-85-7	0.04	NE	0.014	U	0.714	U	0.014	U			0.014	U

Table 5B. Groundwater Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

				Task Description	Lower Scajaquada Creek Area									
				Sample Name	MW-LSC-3		MW-LSC-4		MW-LSC-5		DUP		BSA-MH-WATER	
				Sample Date	1/18/2022		1/19/2022		1/19/2022		1/19/2022		11/18/2021	
				Parent Sample							MW-LSC-5			
Analyte	**Units**	**CAS No. or Identifier**	**NY AWQS**	**NYSDEC Interim Guidance**										
gamma-BHC (gamma-Hexachlorocyclohexane) (Lindane)		58-89-9	0.05	NE	0.014	UJ	0.714	U	0.014	UJ			0.014	U
delta-BHC (delta-Hexachlorocyclohexane)		319-86-8	0.04	NE	0.014	UJ	0.714	U	0.014	UJ			0.014	U
Chlordane (Technical)		12789-03-6	NE	NE	0.143	UJ	7.14	U	0.143	UJ			0.143	U
alpha-chlordane		5103-71-9	NE	NE	0.014	U	0.714	U	0.014	UJ			0.014	U
gamma-Chlordane		5103-74-2	NE	NE	0.014	U	0.714	U	0.014	UJ			0.014	U
2,4-D (2,4-Dichlorophenoxyacetic acid)		94-75-7	50	NE										
4,4'-DDT (p,p'-DDT)		50-29-3	0.2	NE	0.029	UJ	1.43	U	0.029	UJ			0.029	U
4,4'-DDE (p,p'-DDE)		72-55-9	0.2	NE	0.029	UJ	1.43	U	0.029	UJ			0.029	U
4,4'-DDD (p,p-DDD)		72-54-8	0.3	NE	0.029	UJ	1.43	U	0.029	UJ			0.029	U
Dieldrin		60-57-1	0.004	NE	0.029	UJ	1.43	U	0.029	UJ			0.029	U
alpha-Endosulfan (I)		959-98-8	NE	NE	0.014	UJ	0.714	U	0.014	UJ			0.014	U
beta-Endosulfan (II)		33213-65-9	NE	NE	0.029	UJ	1.43	U	0.029	UJ			0.029	U
Endosulfan sulfate		1031-07-8	NE	NE	0.029	UJ	1.43	U	0.029	UJ			0.029	U
Endrin		72-20-8	ND	NE	0.029	UJ	1.43	U	0.029	UJ			0.029	U
Endrin aldehyde		7421-93-4	5	NE	0.029	U	1.43	U	0.029	U			0.029	U
Endrin ketone		53494-70-5	5	NE	0.029	UJ	1.43	U	0.029	UJ			0.029	U
Heptachlor		76-44-8	0.04	NE	0.014	UJ	0.714	U	0.014	UJ			0.014	U
Heptachlor epoxide		1024-57-3	0.03	NE	0.014	UJ	0.714	U	0.014	UJ			0.014	U
Methoxychlor		72-43-5	35	NE	0.143	UJ	7.14	UJ	0.143	UJ			0.143	U
2,4,5-TP (Silvex)		93-72-1	0.26	NE										
Toxaphene		8001-35-2	0.06	NE	0.143	UJ	7.14	UJ	0.143	UJ			0.143	U
HERBICIDES (8151A)	ug/L													
2,4-D (2,4-Dichlorophenoxyacetic acid)		94-75-7	50	NE	10	U	10	U	10	U			10	U
2,4,5-T (2,4,5-Trichlorophenoxyacetic acid)		93-76-5	35	NE	2	UJ	2	UJ	2	UJ			2	U
2,4,5-TP (Silvex)		93-72-1	0.26	NE	2	U	2	U	2	U			2	U
PCBs (8082A)	ug/L													
Aroclor 1016		12674-11-2	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ			0.071	U
Aroclor 1221		11104-28-2	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ			0.071	U
Aroclor 1232		11141-16-5	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ			0.071	U
Aroclor 1242		53469-21-9	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ			0.071	U
Aroclor 1248		12672-29-6	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ			0.071	U
Aroclor 1254		11097-69-1	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ			**0.066**	**J**
Aroclor 1260		11096-82-5	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ			0.071	U
Aroclor 1262		37324-23-5	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ			0.071	U
Aroclor 1268		11100-14-4	NE	NE	0.071	UJ	0.071	UJ	0.071	UJ			0.071	U
Total PCBs (Lab calculated)		1336-36-3	0.09	NE	0.071	UJ	0.071	UJ	0.071	UJ			**0.066**	**J**
PFAS (LCMSMS-ID)	ng/L													

Table 5B. Groundwater Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

					Task Description	Lower Scajaquada Creek Area									
					Sample Name	MW-LSC-3		MW-LSC-4		MW-LSC-5		DUP		BSA-MH-WATER	
					Sample Date	1/18/2022		1/19/2022		1/19/2022		1/19/2022		11/18/2021	
					Parent Sample							MW-LSC-5			
Analyte	Units	CAS No. or Identifier	NY AWQS	NYSDEC Interim Guidance											
N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)		2991-50-6	NE	NE		1.86	U	10	U	1.84	U	1.79	U	2.46	U
N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)		2355-31-9	NE	NE		1.86	U	10	U	1.84	U	1.79	U	2.46	U
Perfluorobutanesulfonic acid (PFBS)		375-73-5	NE	NE		**1.63**	**J**	**1.92**	**J**	**1.43**	**J**	**1.32**	**J**	**2.32**	**J**
Perfluorobutanoic acid (PFBA)		375-22-4	NE	NE		**7.46**		**5.92**	**J**	**7.8**	**J**	**14.6**	**J**	**11.8**	
Perfluorodecanesulfonic acid (PFDS)		335-77-3	NE	NE		1.86	UJ	10	UJ	1.84	UJ	1.79	UJ	2.46	U
Perfluorodecanoic acid (PFDA)		335-76-2	NE	NE		1.86	U	10	U	1.84	U	1.79	U	**0.566**	**J**
Perfluorododecanoic acid (PFDoA)		307-55-1	NE	NE		1.86	U	10	U	1.84	U	1.79	U	2.46	U
Perfluoroheptanesulfonic acid (PFHpS)		375-92-8	NE	NE		1.86	U	10	U	1.84	U	1.79	U	2.46	U
Perfluoroheptanoic acid (PFHpA)		375-85-9	NE	NE		**4.24**		**5.4**	**J**	**6.18**	**J**	**10**	**J**	**2.58**	
Perfluorohexanoic acid (PFHxA)		307-24-4	NE	NE		**8.77**		**13.6**		**16.6**		**17.9**		**5**	
Perfluorooctanesulfonamide (FOSA)		754-91-6	NE	NE		1.86	U	10	U	1.84	U	1.79	U	2.46	U
Perfluoropentanoic Acid (PFPeA)		2706-90-3	NE	NE		**16.8**		**25.1**		**30.3**		**36**		**10.4**	
Perfluorotetradecanoic acid (PFTA/PFTeDA)		376-06-7	NE	NE		1.86	U	10	U	1.84	U	1.79	U	**0.462**	**J**
Perfluorotridecanoic acid (PFTriA/PFTrDA)		72629-94-8	NE	NE		1.86	U	10	U	1.84	U	1.79	U	2.46	U
Perfluoroundecanoic acid (PFUnA)		2058-94-8	NE	NE		1.86	U	10	U	1.84	U	1.79	U	2.46	U
Sodium 1H,1H,2H,2H-Perfluorodecane Sulfonate (8:2)		39108-34-4	NE	NE		1.86	UJ	10	UJ	1.84	UJ	1.79	UJ	2.46	U
Sodium 1H,1H,2H,2H-Perfluorooctane Sulfonate (6:2)		27619-97-2	NE	NE		1.86	U	**14.6**		**29.6**	**J**	**1.58**	**J**	**1.91**	**J**
Perfluorohexanesulfonic acid (PFHxS)		355-46-4	NE	NE		**2.23**		**4.52**	**J**	**5.77**		**2.2**		**1.52**	**J**
Perfluorononanoic Acid (PFNA)		375-95-1	NE	NE		**0.305**	**J**	**1.74**	**J**	**1.44**	**J**	1.79	U	**0.718**	**J**
Perfluorooctanesulfonic acid (PFOS)		1763-23-1	NE	10		**2.06**	**J**	**18**		**8.3**	**J**	**1.46**	**J**	**5.33**	
Perfluorooctanoic Acid (PFOA)		335-67-1	NE	10		**3.38**		**4.86**	**J**	**4.88**		**6.76**		**3.17**	
Total PFAS (ND=0)		TPFAS_ND0	NE	NE		**46.875**		**95.66**		**112.3**		**91.82**		**45.776**	

Table 5B. Groundwater Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

Notes:

mg/L = milligrams/liter
ng/L = nanogram per liter (ppt)
ug/L = micrograms per liter or parts per billion (ppb)

PAH = Polycyclic Aromatic Hydrocarbon
PCB = Polychlorinated Biphenyl
SVOC = Semi-Volatile Organic Compound
VOC = Volatile Organic Compound

CAS No. = Chemical Abstracts Service Number
ND = Not Detected
NE = Not Established

NYS AWQS = New York State Ambient Water Quality Standards and Guidance Values for GA groundwater
* indicates the value is a guidance value and not a standard
NYSDEC Interim Groundwater Screening Levels based on NYSDEC guidance as follows:
- February 2019 (1,4-dioxane)
- June 2021 (PFAS)
Bolding indicates a detected result concentration
Shading and bolding indicates that the detected concentration is above the NYSDOH guidance it was compared to

Validation Qualifiers:
J = The result is an estimated value.
R = The result is rejected.
U = The result was not detected above the reporting limit.
UJ = The results was not detected at or above the reporting limit shown and the reporting limit is estimated.

Table 5C. Creek Shallow Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

			Task Description	Scajaquada Creek Shallow Sediment Boring Sample Analytical Results											
			Sample Name	SED-BGL-1/0-0.5		SED-LSC-1/0-0.5		SED-LSC-2/0-0.5		SED-LSC-3/0-0.5		SED-LSC-DUP/0-0.5		SED-LSC-4/0-0.5	
			Start Depth	0		0		0		0		0		0	
			End Depth	0.5		0.5		0.5		0.5		0.5		0.5	
			Depth Unit	ft		ft		ft		ft		ft		ft	
			Sample Date	11/11/2021		11/11/2021		11/11/2021		11/11/2021		11/11/2021		11/11/2021	
			Parent Sample									SED-LSC-3/0-0.5			
Analyte	**Units**	**CAS No. or Identifier**	**Freshwater Sediment Guidance Values Class B**												
VOCs (SW 8260C)	ug/kg														
Acetone		67-64-1	NE	930	U	3,300	UJ	**150**	**J**	4,000	UJ	1,800	UJ	**2,500**	**J**
Benzene		71-43-2	1900	46	U	170	UJ	1.2	UJ	200	UJ	**34**	**J**	250	UJ
Bromochloromethane		74-97-5	NE	190	U	660	UJ	4.8	UJ	810	UJ	360	UJ	1000	UJ
Bromodichloromethane		75-27-4	NE	46	U	170	UJ	1.2	UJ	200	UJ	89	UJ	250	UJ
Bromoform		75-25-2	NE	370	U	1,300	UJ	9.6	UJ	1,600	UJ	710	UJ	2,000	UJ
Bromomethane		74-83-9	NE	190	U	660	UJ	4.8	UJ	810	UJ	360	UJ	1000	UJ
Carbon disulfide		75-15-0	NE	930	UJ	3,300	UJ	24	UJ	4,000	UJ	1,800	UJ	5,000	UJ
Carbon tetrachloride		56-23-5	9600	93	UJ	330	UJ	2.4	UJ	400	UJ	180	UJ	500	UJ
Chlorobenzene		108-90-7	1700	46	U	170	UJ	1.2	UJ	200	UJ	89	UJ	250	UJ
Chloroethane		75-00-3	NE	190	U	660	UJ	4.8	UJ	810	UJ	360	UJ	1000	UJ
Chloroform (Trichloromethane)		67-66-3	NE	140	U	**66**	**J**	3.6	UJ	610	UJ	270	UJ	760	UJ
Chloromethane		74-87-3	NE	370	U	1,300	UJ	9.6	UJ	1,600	UJ	710	UJ	2,000	UJ
Cyclohexane		110-82-7	NE	930	U	3,300	UJ	24	UJ	4,000	UJ	1,800	UJ	5,000	UJ
1,2-Dibromo-3-chloropropane		96-12-8	NE	280	U	1000	UJ	7.2	UJ	1,200	UJ	540	UJ	1,500	UJ
Dibromochloromethane		124-48-1	NE	93	U	330	UJ	2.4	UJ	400	UJ	180	UJ	500	UJ
1,2-Dibromoethane (EDB)		106-93-4	NE	93	U	330	UJ	2.4	UJ	400	UJ	180	UJ	500	UJ
1,2-Dichlorobenzene (o-DCB)		95-50-1	2500	190	U	660	UJ	4.8	UJ	810	UJ	360	UJ	1000	UJ
1,3-Dichlorobenzene (m-DCB)		541-73-1	7100	190	U	660	UJ	4.8	UJ	810	UJ	360	UJ	1000	UJ
1,4-Dichlorobenzene (p-DCB)		106-46-7	3300	190	U	660	UJ	4.8	UJ	810	UJ	360	UJ	1000	UJ
Dichlorodifluoromethane (Freon 12)		75-71-8	NE	930	U	3,300	UJ	24	UJ	4,000	UJ	1,800	UJ	5,000	UJ
1,1-Dichloroethane		75-34-3	NE	93	U	330	UJ	2.4	UJ	400	UJ	180	UJ	500	UJ
1,2-Dichloroethane		107-06-2	NE	93	U	330	UJ	2.4	UJ	400	UJ	180	UJ	500	UJ
1,1-Dichloroethene		75-35-4	4700	93	U	330	UJ	2.4	UJ	400	UJ	180	UJ	500	UJ
cis-1,2-Dichloroethene		156-59-2	NE	93	U	330	UJ	**1.2**	**J**	400	UJ	180	UJ	500	UJ
trans-1,2-Dichloroethene		156-60-5	11000	140	U	500	UJ	3.6	UJ	610	UJ	270	UJ	760	UJ
1,2-Dichloropropane		78-87-5	NE	93	U	330	UJ	2.4	UJ	400	UJ	180	UJ	500	UJ
cis-1,3-Dichloropropene		10061-01-5	NE	46	U	170	UJ	1.2	UJ	200	UJ	89	UJ	250	UJ
trans-1,3-Dichloropropene		10061-02-6	NE	93	U	330	UJ	2.4	UJ	400	UJ	180	UJ	500	UJ
1,4-Dioxane		123-91-1	NE		R		R		R		R		R		R
Ethylbenzene		100-41-4	3700	93	U	330	UJ	**0.68**	**J**	**5,300**	**J**	**3,000**	**J**	500	UJ
2-Hexanone		591-78-6	NE	930	U	3,300	UJ	24	UJ	4,000	UJ	1,800	UJ	5,000	UJ
Isopropylbenzene		98-82-8	1800	93	U	330	UJ	**0.41**	**J**	**560**	**J**	**280**	**J**	500	UJ
Methyl acetate		79-20-9	NE	**800**		**2,900**	**J**	9.6	UJ	**2,200**	**J**	**1000**	**J**	**15,000**	**J**
Methyl ethyl ketone (2-Butanone)		78-93-3	NE	930	U	3,300	UJ	**33**	**J**	4,000	UJ	1,800	UJ	5,000	UJ

Table 5C. Creek Shallow Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

			Task Description	Scajaquada Creek Shallow Sediment Boring Sample Analytical Results											
			Sample Name	SED-BGL-1/0-0.5		SED-LSC-1/0-0.5		SED-LSC-2/0-0.5		SED-LSC-3/0-0.5		SED-LSC-DUP/0-0.5		SED-LSC-4/0-0.5	
			Start Depth	0		0		0		0		0		0	
			End Depth	0.5		0.5		0.5		0.5		0.5		0.5	
			Depth Unit	ft		ft		ft		ft		ft		ft	
			Sample Date	11/11/2021		11/11/2021		11/11/2021		11/11/2021		11/11/2021		11/11/2021	
			Parent Sample									SED-LSC-3/0-0.5			
Analyte	**Units**	**CAS No. or Identifier**	**Freshwater Sediment Guidance Values Class B**												
Methyl tert-butyl ether (MTBE)		1634-04-4	NE	190	U	660	UJ	4.8	UJ	810	UJ	360	UJ	1000	UJ
4-Methyl-2-pentanone (MIBK)		108-10-1	NE	930	U	3,300	UJ	24	UJ	4,000	UJ	1,800	UJ	5,000	UJ
Methylcyclohexane		108-87-2	NE	370	UJ	1,300	UJ	9.6	UJ	1,600	UJ	710	UJ	2,000	UJ
Methylene chloride		75-09-2	NE	460	U	1,700	UJ	12	UJ	2,000	UJ	890	UJ	2,500	UJ
Styrene		100-42-5	NE	93	U	330	UJ	2.4	UJ	400	UJ	180	UJ	500	UJ
1,1,2,2-Tetrachloroethane		79-34-5	5400	46	U	170	UJ	1.2	UJ	200	UJ	89	UJ	250	UJ
Tetrachloroethene (PCE)		127-18-4	57000	46	U	170	UJ	1.2	UJ	200	UJ	89	UJ	250	UJ
Toluene		108-88-3	4500	93	U	330	UJ	2.4	UJ	**220**	**J**	**140**	**J**	500	UJ
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)		76-13-1	NE	370	UJ	1,300	UJ	9.6	UJ	1,600	UJ	710	UJ	2,000	UJ
1,2,3-Trichlorobenzene		87-61-6	2800	190	U	660	UJ	4.8	UJ	810	UJ	360	UJ	1000	UJ
1,2,4-Trichlorobenzene		120-82-1	55000	190	U	660	UJ	4.8	UJ	810	UJ	360	UJ	1000	UJ
1,1,1-Trichloroethane (TCA)		71-55-6	3500	46	UJ	170	UJ	1.2	UJ	200	UJ	89	UJ	250	UJ
1,1,2-Trichloroethane		79-00-5	3500	93	U	330	UJ	2.4	UJ	400	UJ	180	UJ	500	UJ
Trichloroethene (TCE)		79-01-6	8600	46	U	170	UJ	1.2	UJ	200	UJ	89	UJ	250	UJ
Trichlorofluoromethane (Freon 11)		75-69-4	NE	370	U	1,300	UJ	9.6	UJ	1,600	UJ	710	UJ	2,000	UJ
Vinyl chloride		75-01-4	NE	93	U	330	UJ	2.4	UJ	400	UJ	180	UJ	500	UJ
o-Xylene		95-47-6	7200	93	U	330	UJ	**1.4**	**J**	**1,600**	**J**	**870**	**J**	500	UJ
m/p-Xylene		179601-23-1	4200	190	U	660	UJ	4.8	UJ	**2,500**	**J**	**1,400**	**J**	1000	UJ
SVOCs (SW 8270D)	ug/kg														
Acenaphthene		83-32-9	NE	**120**	**J**	**3,200**	**J**	**4,500**	**J**	**520,000**	**J**	**390,000**	**J**	4,700	UJ
Acenaphthylene		208-96-8	NE	**570**	**J**	**6,100**	**J**	**8,700**	**J**	**130,000**	**J**	**96,000**	**J**	4,700	UJ
Acetophenone		98-86-2	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
Anthracene		120-12-7	NE	**300**	**J**	**5,500**	**J**	**6,100**	**J**	**390,000**	**J**	**310,000**	**J**	3,600	UJ
Atrazine		1912-24-9	NE	890	U	1,600	UJ	1,400	UJ	28,000	UJ	6,900	UJ	4,700	UJ
Benzaldehyde		100-52-7	NE	1,500	U	2,700	UJ	2,300	UJ	46,000	UJ	11,000	UJ	7,800	UJ
Benzo(a)anthracene		56-55-3	NE	**1000**		**10,000**	**J**	**17,000**	**J**	**250,000**	**J**	**220,000**	**J**	**1,200**	**J**
Benzo(b)fluoranthene		205-99-2	NE	**1,100**		**9,300**	**J**	**15,000**	**J**	**150,000**	**J**	**130,000**	**J**	**1,900**	**J**
Benzo(k)fluoranthene		207-08-9	NE	**320**	**J**	**2,800**	**J**	**4,200**	**J**	**40,000**	**J**	**32,000**	**J**	3,600	UJ
Benzo(g,h,i)perylene		191-24-2	NE	**580**	**J**	**6,400**	**J**	**9,000**	**J**	**95,000**	**J**	**82,000**	**J**	**1,100**	**J**
Benzo(a)pyrene		50-32-8	NE	**1,100**		**11,000**	**J**	**19,000**	**J**	**210,000**	**J**	**180,000**	**J**	4,700	UJ
Biphenyl (1,1-Biphenyl)		92-52-4	NE	2,500	U	4,600	UJ	4,000	UJ	**210,000**	**J**	**160,000**	**J**	14,000	UJ
Bis(2-chloroethoxy)methane		111-91-1	NE	1,200	U	2,200	UJ	1,900	UJ	38,000	UJ	9,300	UJ	6,400	UJ
Bis(2-chloroethyl)ether		111-44-4	NE	1000	U	1,800	UJ	1,600	UJ	32,000	UJ	7,800	UJ	5,300	UJ
2,2-oxybis(1-Chloropropane)		108-60-1	NE	1,300	UJ	2,400	UJ	2,100	UJ	42,000	UJ	10,000	UJ	7,100	UJ
Bis(2-ethylhexyl)phthalate		117-81-7	360000	1,100	UJ	**2,400**	**J**	**3,200**	**J**	35,000	UJ	**4,100**	**J**	5,900	UJ
4-Bromophenyl phenyl ether		101-55-3	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ

Table 5C. Creek Shallow Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

			Task Description	Scajaquada Creek Shallow Sediment Boring Sample Analytical Results											
			Sample Name	SED-BGL-1/0-0.5		SED-LSC-1/0-0.5		SED-LSC-2/0-0.5		SED-LSC-3/0-0.5		SED-LSC-DUP/0-0.5		SED-LSC-4/0-0.5	
			Start Depth	0		0		0		0		0		0	
			End Depth	0.5		0.5		0.5		0.5		0.5		0.5	
			Depth Unit	ft		ft		ft		ft		ft		ft	
			Sample Date	11/11/2021		11/11/2021		11/11/2021		11/11/2021		11/11/2021		11/11/2021	
			Parent Sample									SED-LSC-3/0-0.5			
Analyte	Units	CAS No. or Identifier	Freshwater Sediment Guidance Values Class B												
Butyl benzyl phthalate		85-68-7	NE	1,100	UJ	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
Caprolactam		105-60-2	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
Carbazole		86-74-8	NE	1,100	U	**240**	**J**	**230**	**J**	35,000	UJ	**1,900**	**J**	5,900	UJ
4-Chloro-3-methylphenol		59-50-7	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
4-Chloroaniline		106-47-8	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
2-Chloronaphthalene		91-58-7	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
2-Chlorophenol		95-57-8	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
4-Chlorophenyl phenyl ether		7005-72-3	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
Chrysene		218-01-9	NE	**980**		**9,400**	**J**	**14,000**	**J**	**200,000**	**J**	**160,000**	**J**	**1,500**	**J**
Dibenz(a,h)anthracene		53-70-3	NE	670	U	**1,400**	**J**	**1,900**	**J**	**23,000**	**J**	**20,000**	**J**	3,600	UJ
Dibenzofuran		132-64-9	NE	1,100	U	2,000	UJ	1,800	UJ	**69,000**	**J**	**44,000**	**J**	5,900	UJ
3,3-Dichlorobenzidine		91-94-1	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
2,4-Dichlorophenol		120-83-2	NE	1000	U	1,800	UJ	1,600	UJ	32,000	UJ	7,800	UJ	5,300	UJ
Diethyl phthalate		84-66-2	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
Dimethyl phthalate		131-11-3	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
2,4-Dimethylphenol		105-67-9	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
Di-n-butyl phthalate		84-74-2	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
4,6-Dinitro-2-methylphenol		534-52-1	NE	2,900	UJ	5,300	UJ	4,600	UJ	92,000	UJ	22,000	UJ	15,000	UJ
2,4-Dinitrophenol		51-28-5	NE	5,400	UJ	9,800	UJ	8,400	UJ	170,000	UJ	41,000	UJ	28,000	UJ
2,4-Dinitrotoluene		121-14-2	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
2,6-Dinitrotoluene		606-20-2	NE	1,100	UJ	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
Di-n-octyl phthalate		117-84-0	NE	1,100	UJ	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
Fluoranthene		206-44-0	NE	**1,300**		**13,000**	**J**	**17,000**	**J**	**400,000**	**J**	**340,000**	**J**	**2,700**	**J**
Fluorene		86-73-7	NE	**140**	**J**	**2,700**	**J**	**3,400**	**J**	**320,000**	**J**	**250,000**	**J**	5,900	UJ
Hexachlorobenzene		118-74-1	NE	670	U	1,200	UJ	1000	UJ	21,000	UJ	5,200	UJ	3,600	UJ
1,3-Hexachlorobutadiene (C-46)		87-68-3	12000	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
Hexachlorocyclopentadiene		77-47-4	8100	3,200	U	5,800	UJ	5,000	UJ	100,000	UJ	25,000	UJ	17,000	UJ
Hexachloroethane		67-72-1	NE	890	U	1,600	UJ	1,400	UJ	28,000	UJ	6,900	UJ	4,700	UJ
Indeno(1,2,3-cd)pyrene		193-39-5	NE	**510**	**J**	**5,600**	**J**	**7,900**	**J**	**81,000**	**J**	**67,000**	**J**	**1000**	**J**
Isophorone		78-59-1	NE	1000	U	1,800	UJ	1,600	UJ	32,000	UJ	7,800	UJ	5,300	UJ
2-Methylnaphthalene		91-57-6	NE	1,300	U	**600**	**J**	**750**	**J**	**1,200,000**	**J**	**960,000**	**J**	7,100	UJ
2-Methylphenol (o-Cresol)		95-48-7	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
3,4-Methylphenol (m,p-Cresol)		108394/106445	NE	1,600	U	2,900	UJ	2,500	UJ	51,000	UJ	12,000	UJ	8,500	UJ
Naphthalene		91-20-3	NE	1,100	U	**400**	**J**	**530**	**J**	**880,000**	**J**	**640,000**	**J**	5,900	UJ
2-Nitroaniline		88-74-4	NE	1,100	UJ	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
3-Nitroaniline		99-09-2	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ

Table 5C. Creek Shallow Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

			Task Description	Scajaquada Creek Shallow Sediment Boring Sample Analytical Results											
			Sample Name	SED-BGL-1/0-0.5		SED-LSC-1/0-0.5		SED-LSC-2/0-0.5		SED-LSC-3/0-0.5		SED-LSC-DUP/0-0.5		SED-LSC-4/0-0.5	
			Start Depth	0		0		0		0		0		0	
			End Depth	0.5		0.5		0.5		0.5		0.5		0.5	
			Depth Unit	ft		ft		ft		ft		ft		ft	
			Sample Date	11/11/2021		11/11/2021		11/11/2021		11/11/2021		11/11/2021		11/11/2021	
			Parent Sample									SED-LSC-3/0-0.5			
Analyte	**Units**	**CAS No. or Identifier**	**Freshwater Sediment Guidance Values Class B**												
4-Nitroaniline		100-01-6	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
Nitrobenzene		98-95-3	NE	1000	U	1,800	UJ	1,600	UJ	32,000	UJ	7,800	UJ	5,300	UJ
2-Nitrophenol		88-75-5	NE	2,400	UJ	4,400	UJ	3,800	UJ	76,000	UJ	19,000	UJ	13,000	UJ
4-Nitrophenol		100-02-7	NE	1,600	U	2,800	UJ	2,500	UJ	49,000	UJ	12,000	UJ	8,300	UJ
N-Nitrosodiphenylamine (NDFA)		86-30-6	NE	890	U	1,600	UJ	1,400	UJ	28,000	UJ	6,900	UJ	4,700	UJ
N-Nitrosodi-n-propylamine (NDPA)		621-64-7	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
Pentachlorophenol		87-86-5	19000	890	U	1,600	UJ	1,400	UJ	28,000	UJ	6,900	UJ	4,700	UJ
Phenanthrene		85-01-8	NE	**590**	**J**	**14,000**	**J**	**13,000**	**J**	**1,200,000**	**J**	**1,100,000**	**J**	**990**	**J**
Phenol		108-95-2	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
Pyrene		129-00-0	NE	**2,000**		**24,000**	**J**	**32,000**	**J**	**760,000**	**J**	**660,000**	**J**	**3,100**	**J**
1,2,4,5-Tetrachlorobenzene		95-94-3	14000	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
2,3,4,6-Tetrachlorophenol		58-90-2	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
2,4,5-Trichlorophenol		95-95-4	NE	1,100	U	2,000	UJ	1,800	UJ	35,000	UJ	8,600	UJ	5,900	UJ
2,4,6-Trichlorophenol		88-06-2	NE	670	U	1,200	UJ	1000	UJ	21,000	UJ	5,200	UJ	3,600	UJ
Total PAH (16) (ND=0)		TPAH16_ND0	35000	**10,610**		**124,800**		**173,230**		**5,649,000**		**4,677,000**		**13,490**	
Total PAH (17) (ND=0)		TPAH17_ND0	35000	**10,610**		**125,400**		**173,980**		**6,849,000**		**5,637,000**		**13,490**	
PCBs (SW 8082A)	ug/kg														
Aroclor 1016		12674-11-2	NE	76.5	U	140	UJ	124	UJ	232	UJ	112	UJ	587	UJ
Aroclor 1221		11104-28-2	NE	76.5	U	140	UJ	124	UJ	232	UJ	112	UJ	587	UJ
Aroclor 1232		11141-16-5	NE	76.5	U	140	UJ	124	UJ	232	UJ	112	UJ	587	UJ
Aroclor 1242		53469-21-9	NE	76.5	U	140	UJ	124	UJ	232	UJ	112	UJ	587	UJ
Aroclor 1248		12672-29-6	NE	**90.1**		**472**	**J**	**421**	**J**	**475**	**J**	**250**	**J**	**628**	**J**
Aroclor 1254		11097-69-1	NE	76.5	U	**294**	**J**	**327**	**J**	**487**	**J**	**122**	**J**	**350**	**J**
Aroclor 1260		11096-82-5	NE	**36.4**	**J**	**182**	**J**	**408**	**J**	**144**	**J**	**75.4**	**J**	**249**	**J**
Aroclor 1262		37324-23-5	NE	76.5	U	140	UJ	124	UJ	232	UJ	112	UJ	587	UJ
Aroclor 1268		11100-14-4	NE	76.5	U	140	UJ	124	UJ	232	UJ	112	UJ	587	UJ
Total PCBs (Lab calculated)		1336-36-3	1000	**186**	**J**	**948**	**J**	**1,160**	**J**	**1,110**	**J**	**447**	**J**	**1,230**	**J**
PESTICIDES (SW 8081B)	ug/kg														
Aldrin		309-00-2	NE	10.9	R	6.61	UJ	6.16	UJ	112	UJ	55.2	UJ	30.6	R
alpha-BHC (Hexachlorocyclohexane)		319-84-6	NE	4.54	R	2.75	UJ	2.56	UJ	46.5	UJ	23	UJ	12.8	R
beta-BHC (beta-Hexachlorocyclohexane)		319-85-7	NE	10.9	R	6.61	UJ	6.16	UJ	112	UJ	55.2	UJ	30.6	R
gamma-BHC (gamma-Hexachlorocyclohexane) (Lindane)		58-89-9	78	4.54	R	2.75	UJ	2.56	UJ	46.5	UJ	23	UJ	12.8	R
delta-BHC (delta-Hexachlorocyclohexane)		319-86-8	NE	10.9	R	6.61	UJ	6.16	UJ	112	UJ	55.2	UJ	30.6	R
Chlordane (Technical)		12789-03-6	NE	90.9	R	55.1	UJ	51.3	UJ	930	UJ	460	UJ	255	R
alpha-chlordane		5103-71-9	NE	13.6	R	8.26	UJ	7.7	UJ	140	UJ	69	UJ	38.3	R
gamma-Chlordane		5103-74-2	NE	13.6	R	8.26	UJ	7.7	UJ	140	UJ	69	UJ	38.3	R

Table 5C. Creek Shallow Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

			Task Description	Scajaquada Creek Shallow Sediment Boring Sample Analytical Results											
			Sample Name	SED-BGL-1/0-0.5		SED-LSC-1/0-0.5		SED-LSC-2/0-0.5		SED-LSC-3/0-0.5		SED-LSC-DUP/0-0.5		SED-LSC-4/0-0.5	
			Start Depth	0		0		0		0		0		0	
			End Depth	0.5		0.5		0.5		0.5		0.5		0.5	
			Depth Unit	ft		ft		ft		ft		ft		ft	
			Sample Date	11/11/2021		11/11/2021		11/11/2021		11/11/2021		11/11/2021		11/11/2021	
			Parent Sample									SED-LSC-3/0-0.5			
Analyte	**Units**	**CAS No. or Identifier**	**Freshwater Sediment Guidance Values Class B**												
4,4'-DDT (p,p'-DDT)		50-29-3	48000	20.4	R	**19.3**	**J**	11.5	UJ	209	UJ	103	UJ	57.5	R
4,4'-DDE (p,p'-DDE)		72-55-9	NE	10.9	R	**12**	**J**	6.16	UJ	112	UJ	55.2	UJ	30.6	R
4,4'-DDD (p,p-DDD)		72-54-8	NE	10.9	R	**10.4**	**J**	6.16	UJ	112	UJ	55.2	UJ	30.6	R
Dieldrin		60-57-1	780	6.82	R	4.13	UJ	3.85	UJ	69.8	UJ	34.5	UJ	19.2	R
alpha-Endosulfan (I)		959-98-8	NE	10.9	R	6.61	UJ	6.16	UJ	112	UJ	55.2	UJ	30.6	R
beta-Endosulfan (II)		33213-65-9	NE	10.9	R	6.61	UJ	6.16	UJ	112	UJ	55.2	UJ	30.6	R
Endosulfan sulfate		1031-07-8	NE	4.54	R	2.75	UJ	2.56	UJ	46.5	UJ	23	UJ	12.8	R
Endrin		72-20-8	220	4.54	R	2.75	UJ	2.56	UJ	46.5	UJ	23	UJ	12.8	R
Endrin aldehyde		7421-93-4	NE	13.6	R	8.26	UJ	7.7	UJ	140	UJ	69	UJ	38.3	R
Endrin ketone		53494-70-5	NE	10.9	R	6.61	UJ	6.16	UJ	112	UJ	55.2	UJ	30.6	R
Heptachlor		76-44-8	10000	5.45	R	3.3	UJ	3.08	UJ	55.8	UJ	27.6	UJ	15.3	R
Heptachlor epoxide		1024-57-3	2100	20.4	R	12.4	UJ	11.5	UJ	209	UJ	103	UJ	57.5	R
Methoxychlor		72-43-5	NE	20.4	R	12.4	UJ	11.5	UJ	209	UJ	103	UJ	57.5	R
Toxaphene		8001-35-2	250	204	R	124	UJ	115	UJ	2,090	UJ	1,030	UJ	575	R
HERBICIDES (SW 8151A)	ug/kg														
2,4-D (2,4-Dichlorophenoxyacetic acid)		94-75-7	NE	379	U	721	UJ	628	UJ	1,150	UJ	573	UJ	1,090	UJ
2,4,5-T (2,4,5-Trichlorophenoxyacetic acid)		93-76-5	NE	379	U	721	UJ	628	UJ	1,150	UJ	573	UJ	1,090	UJ
2,4,5-TP (Silvex)		93-72-1	NE	379	U	721	UJ	628	UJ	1,150	UJ	573	UJ	1,090	UJ
CYANIDES (SW 9012B)	mg/kg														
Total Cyanide		57-12-5	NE	2.2	UJ	4.3	UJ	3.5	UJ	6.5	UJ	3.3	UJ	6.4	UJ
METALS (SW 6010D)	mg/kg														
Aluminum		7429-90-5	NE	**5,920**		**10,200**	**J**	**11,500**	**J**	**14,100**	**J**	**40,500**	**J**	**19,100**	**J**
Antimony		7440-36-0	NE	8.96	U	16.7	UJ	15	UJ	26.4	UJ	13.7	UJ	24.9	UJ
Arsenic		7440-38-2	33	**5.77**	**J**	**8.44**	**J**	**9.96**	**J**	**12**	**J**	**11.4**	**J**	**15.6**	**J**
Barium		7440-39-3	NE	**68**		**141**	**J**	**174**	**J**	**175**	**J**	**287**	**J**	**196**	**J**
Beryllium		7440-41-7	NE	0.896	U	1.67	UJ	1.5	UJ	2.64	UJ	**2.06**	**J**	2.49	UJ
Cadmium		7440-43-9	5	**0.968**	**J**	**2.95**	**J**	**4.64**	**J**	**3.65**	**J**	**1.43**	**J**	**4.24**	**J**
Calcium		7440-70-2	NE	**211,000**		**82,500**	**J**	**92,900**	**J**	**137,000**	**J**	**96,200**	**J**	**111,000**	**J**
Chromium		7440-47-3	110	**33.6**	**J**	**58.3**	**J**	**67.4**	**J**	**81.9**	**J**	**62.3**	**J**	**85**	**J**
Cobalt		7440-48-4	NE	**4.46**		**8.87**	**J**	**9.63**	**J**	**14.4**	**J**	**36.6**	**J**	**15.8**	**J**
Copper		7440-50-8	150	**43**	**J**	**173**	**J**	**229**	**J**	**200**	**J**	**54.7**	**J**	**231**	**J**
Iron		7439-89-6	NE	**30,600**		**25,300**	**J**	**28,200**	**J**	**40,800**	**J**	**77,000**	**J**	**46,800**	**J**
Lead		7439-92-1	130	**40.6**	**J**	**208**	**J**	**277**	**J**	**326**	**J**	**25**	**J**	**310**	**J**
Magnesium		7439-95-4	NE	**12,400**		**13,800**	**J**	**13,000**	**J**	**28,800**	**J**	**30,400**	**J**	**22,400**	**J**
Manganese		7439-96-5	NE	**3,040**		**294**	**J**	**373**	**J**	**556**	**J**	**1,330**	**J**	**603**	**J**
Mercury (SW 7471B)		7439-97-6	1	0.146	U	**0.311**	**J**	**0.395**	**J**	**0.388**	**J**	0.218	UJ	**0.384**	**J**

Table 5C. Creek Shallow Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

			Task Description	Scajaquada Creek Shallow Sediment Boring Sample Analytical Results											
			Sample Name	SED-BGL-1/0-0.5		SED-LSC-1/0-0.5		SED-LSC-2/0-0.5		SED-LSC-3/0-0.5		SED-LSC-DUP/0-0.5		SED-LSC-4/0-0.5	
			Start Depth	0		0		0		0		0		0	
			End Depth	0.5		0.5		0.5		0.5		0.5		0.5	
			Depth Unit	ft		ft		ft		ft		ft		ft	
			Sample Date	11/11/2021		11/11/2021		11/11/2021		11/11/2021		11/11/2021		11/11/2021	
			Parent Sample									SED-LSC-3/0-0.5			
Analyte	**Units**	**CAS No. or Identifier**	**Freshwater Sediment Guidance Values Class B**												
Nickel		7440-02-0	49	**11.9**		**27.3**	**J**	**32.7**	**J**	**48.7**	**J**	**79.8**	**J**	**49.6**	**J**
Potassium		7440-09-7	NE	**772**		**1,290**	**J**	**1,380**	**J**	**1,810**	**J**	**6,040**	**J**	**2,380**	**J**
Selenium		7782-49-2	NE	3.58	U	6.7	UJ	5.98	UJ	10.6	UJ	5.49	UJ	9.97	UJ
Silver		7440-22-4	2.2	1.79	U	3.35	UJ	**1.4**	**J**	5.29	UJ	2.74	UJ	4.98	UJ
Sodium		7440-23-5	NE	**539**	**J**	**771**	**J**	598	UJ	1,060	UJ	**1,050**	**J**	997	UJ
Thallium		7440-28-0	NE	**3.58**	**J**	**1.1**	**J**	**1.02**	**J**	10.6	UJ	**1.32**	**J**	9.97	UJ
Vanadium		7440-62-2	NE	**25.6**	**J**	**24.1**	**J**	**29.9**	**J**	**36.6**	**J**	**80.4**	**J**	**44.3**	**J**
Zinc		7440-66-6	460	**116**	**J**	**745**	**J**	**812**	**J**	**1,020**	**J**	**160**	**J**	**989**	**J**
VPH/EPH	mg/kg														
C5-C8 Aliphatics (MAVPH)		ALIPH5-8	NE	9.31	UJ	22.3	UJ	27	UJ	81.2	UJ	35.7	UJ	50.5	UJ
C5-C8 Aliphatics, Adjusted (MAVPH)		ALIPH5-8ADJ	NE	9.31	UJ	22.3	UJ	27	UJ	81.2	UJ	35.7	UJ	50.5	UJ
C9-C12 Aliphatics (MAVPH)		ALIPH9-12	NE	9.31	UJ	22.3	UJ	27	UJ	81.2	UJ	**37.4**	**J**	50.5	UJ
C9-C12 Aliphatics, Adjusted (MAVPH)		ALIPH9-12ADJ	NE	9.31	UJ	22.3	UJ	27	UJ	81.2	UJ	35.7	UJ	50.5	UJ
C9-C10 Aromatics (MAVPH)		AROM9-10	NE	9.31	UJ	22.3	UJ	27	UJ	81.2	UJ	35.7	UJ	50.5	UJ
Total EPH Fractions (NJ EPH R3)		TEPH	NE	**69.4**		**1,270**	**J**	**1,950**	**J**	**67,000**	**J**	**21,500**	**J**	**1,060**	**J**
GENERAL CHEMISTRY	mg/kg														
Ammonia (SM 4500-NH3 BH)		7664-41-7	NE	**32**	**J**	**250**	**J**	**180**	**J**	**310**	**J**	**170**	**J**	**220**	**J**
Percent Solids (SM 2540G)	%	SOLIDS	NE	**42.8**		**22.9**	**J**	**25.9**	**J**	**14.2**	**J**	**28.7**	**J**	**15.2**	**J**
Sulfide (SW 9030B)		18496-25-8	NE	**2,200**		**4,400**	**J**	**5,600**	**J**	**9,400**	**J**	**5,800**	**J**	**2,800**	**J**
TOTAL ORGANIC CARBON	%														
Total Organic Carbon (SW 9060A)		TOC	NE	**7.97**		**9.94**	**J**	**9.02**	**J**	**13.5**	**J**	**12.4**	**J**	**10.2**	**J**

Table 5C. Creek Shallow Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

			Task Description	Scajaquada Creek Shallow Sediment Boring Sample Analytical Results							
			Sample Name	SED-LSC-5/0-0.5		SED-LSC-6/0-0.5		SED-LSC-7/0-0.5		SED-LSC-8/0-0.5	
			Start Depth	0		0		0		0	
			End Depth	0.5		0.5		0.5		0.5	
			Depth Unit	ft		ft		ft		ft	
			Sample Date	11/11/2021		11/3/2021		11/3/2021		11/3/2021	
			Parent Sample								
Analyte	**Units**	**CAS No. or Identifier**	**Freshwater Sediment Guidance Values Class B**								
VOCs (SW 8260C)	ug/kg										
Acetone		67-64-1	NE	**170**	**J**	**48**	**J**	**26**	**J**	**120**	**J**
Benzene		71-43-2	1900	**1.3**	**J**	0.9	UJ	0.44	UJ	**5.5**	**J**
Bromochloromethane		74-97-5	NE	3.7	UJ	3.6	UJ	1.7	UJ	3.2	UJ
Bromodichloromethane		75-27-4	NE	0.94	UJ	0.9	UJ	0.44	UJ	0.81	UJ
Bromoform		75-25-2	NE	7.5	UJ	7.2	UJ	3.5	UJ	6.5	UJ
Bromomethane		74-83-9	NE	3.7	UJ	3.6	UJ	1.7	UJ	3.2	UJ
Carbon disulfide		75-15-0	NE	19	UJ	18	UJ	8.7	UJ	16	UJ
Carbon tetrachloride		56-23-5	9600	1.9	UJ	1.8	UJ	0.87	UJ	1.6	UJ
Chlorobenzene		108-90-7	1700	0.94	UJ	0.9	UJ	0.44	UJ	0.81	UJ
Chloroethane		75-00-3	NE	3.7	UJ	3.6	UJ	1.7	UJ	3.2	UJ
Chloroform (Trichloromethane)		67-66-3	NE	2.8	UJ	2.7	UJ	1.3	UJ	2.4	UJ
Chloromethane		74-87-3	NE	7.5	UJ	7.2	UJ	3.5	UJ	6.5	UJ
Cyclohexane		110-82-7	NE	19	UJ	18	UJ	8.7	UJ	**1**	**J**
1,2-Dibromo-3-chloropropane		96-12-8	NE	5.6	UJ	5.4	UJ	2.6	UJ	4.9	UJ
Dibromochloromethane		124-48-1	NE	1.9	UJ	1.8	UJ	0.87	UJ	1.6	UJ
1,2-Dibromoethane (EDB)		106-93-4	NE	1.9	UJ	1.8	UJ	0.87	UJ	1.6	UJ
1,2-Dichlorobenzene (o-DCB)		95-50-1	2500	3.7	UJ	3.6	UJ	1.7	UJ	3.2	UJ
1,3-Dichlorobenzene (m-DCB)		541-73-1	7100	3.7	UJ	3.6	UJ	1.7	UJ	3.2	UJ
1,4-Dichlorobenzene (p-DCB)		106-46-7	3300	3.7	UJ	3.6	UJ	1.7	UJ	3.2	UJ
Dichlorodifluoromethane (Freon 12)		75-71-8	NE	19	UJ	18	UJ	8.7	UJ	16	UJ
1,1-Dichloroethane		75-34-3	NE	1.9	UJ	**0.6**	**J**	0.87	UJ	1.6	UJ
1,2-Dichloroethane		107-06-2	NE	1.9	UJ	1.8	UJ	0.87	UJ	1.6	UJ
1,1-Dichloroethene		75-35-4	4700	1.9	UJ	1.8	UJ	0.87	UJ	1.6	UJ
cis-1,2-Dichloroethene		156-59-2	NE	**0.48**	**J**	**4.4**	**J**	**0.3**	**J**	**0.72**	**J**
trans-1,2-Dichloroethene		156-60-5	11000	2.8	UJ	2.7	UJ	1.3	UJ	2.4	UJ
1,2-Dichloropropane		78-87-5	NE	1.9	UJ	1.8	UJ	0.87	UJ	1.6	UJ
cis-1,3-Dichloropropene		10061-01-5	NE	0.94	UJ	0.9	UJ	0.44	UJ	0.81	UJ
trans-1,3-Dichloropropene		10061-02-6	NE	1.9	UJ	1.8	UJ	0.87	UJ	1.6	UJ
1,4-Dioxane		123-91-1	NE		R		R		R		R
Ethylbenzene		100-41-4	3700	1.9	UJ	**0.36**	**J**	0.87	UJ	**20**	**J**
2-Hexanone		591-78-6	NE	19	UJ	18	UJ	8.7	UJ	16	UJ
Isopropylbenzene		98-82-8	1800	1.9	UJ	1.8	UJ	0.87	UJ	**13**	**J**
Methyl acetate		79-20-9	NE	7.5	UJ	7.2	UJ	3.5	UJ	6.5	UJ
Methyl ethyl ketone (2-Butanone)		78-93-3	NE	**32**	**J**	18	UJ	8.7	UJ	**13**	**J**

Table 5C. Creek Shallow Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

			Task Description	Scajaquada Creek Shallow Sediment Boring Sample Analytical Results							
			Sample Name	SED-LSC-5/0-0.5		SED-LSC-6/0-0.5		SED-LSC-7/0-0.5		SED-LSC-8/0-0.5	
			Start Depth	0		0		0		0	
			End Depth	0.5		0.5		0.5		0.5	
			Depth Unit	ft		ft		ft		ft	
			Sample Date	11/11/2021		11/3/2021		11/3/2021		11/3/2021	
			Parent Sample								
Analyte	**Units**	**CAS No. or Identifier**	**Freshwater Sediment Guidance Values Class B**								
Methyl tert-butyl ether (MTBE)		1634-04-4	NE	3.7	UJ	3.6	UJ	1.7	UJ	3.2	UJ
4-Methyl-2-pentanone (MIBK)		108-10-1	NE	19	UJ	18	UJ	8.7	UJ	16	UJ
Methylcyclohexane		108-87-2	NE	7.5	UJ	7.2	UJ	3.5	UJ	**2.5**	**J**
Methylene chloride		75-09-2	NE	9.4	UJ	9	UJ	4.4	UJ	8.1	UJ
Styrene		100-42-5	NE	1.9	UJ	1.8	UJ	0.87	UJ	1.6	UJ
1,1,2,2-Tetrachloroethane		79-34-5	5400	0.94	UJ	0.9	UJ	0.44	UJ	0.81	UJ
Tetrachloroethene (PCE)		127-18-4	57000	0.94	UJ	0.9	UJ	0.44	UJ	0.81	UJ
Toluene		108-88-3	4500	1.9	UJ	1.8	UJ	0.87	UJ	1.6	UJ
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)		76-13-1	NE	7.5	UJ	7.2	UJ	3.5	UJ	6.5	UJ
1,2,3-Trichlorobenzene		87-61-6	2800	3.7	UJ	3.6	UJ	1.7	UJ	3.2	UJ
1,2,4-Trichlorobenzene		120-82-1	55000	3.7	UJ	3.6	UJ	1.7	UJ	3.2	UJ
1,1,1-Trichloroethane (TCA)		71-55-6	3500	0.94	UJ	0.9	UJ	0.44	UJ	0.81	UJ
1,1,2-Trichloroethane		79-00-5	3500	1.9	UJ	1.8	UJ	0.87	UJ	1.6	UJ
Trichloroethene (TCE)		79-01-6	8600	0.94	UJ	0.9	UJ	0.44	UJ	0.81	UJ
Trichlorofluoromethane (Freon 11)		75-69-4	NE	7.5	UJ	7.2	UJ	3.5	UJ	6.5	UJ
Vinyl chloride		75-01-4	NE	1.9	UJ	1.8	UJ	0.87	UJ	1.6	UJ
o-Xylene		95-47-6	7200	1.9	UJ	1.8	UJ	0.87	UJ	**6.2**	**J**
m/p-Xylene		179601-23-1	4200	3.7	UJ	3.6	UJ	1.7	UJ	**2.2**	**J**
SVOCs (SW 8270D)	ug/kg										
Acenaphthene		83-32-9	NE	3,200	UJ	2,000	UJ	**160**	**J**	**2,600**	**J**
Acenaphthylene		208-96-8	NE	3,200	UJ	2,000	UJ	**320**	**J**	**4,400**	**J**
Acetophenone		98-86-2	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
Anthracene		120-12-7	NE	2,400	UJ	1,500	UJ	810	U	**8,300**	**J**
Atrazine		1912-24-9	NE	3,200	UJ	2,000	UJ	1,100	UJ	1,700	UJ
Benzaldehyde		100-52-7	NE	5,300	UJ	3,200	UJ	1,800	U	2,800	UJ
Benzo(a)anthracene		56-55-3	NE	**1000**	**J**	**960**	**J**	**760**	**J**	**11,000**	**J**
Benzo(b)fluoranthene		205-99-2	NE	**1,600**	**J**	**1,500**	**J**	**1,200**		**8,800**	**J**
Benzo(k)fluoranthene		207-08-9	NE	2,400	UJ	**430**	**J**	**370**	**J**	**3,000**	**J**
Benzo(g,h,i)perylene		191-24-2	NE	**900**	**J**	**960**	**J**	**800**	**J**	**6,000**	**J**
Benzo(a)pyrene		50-32-8	NE	**1,100**	**J**	**1,200**	**J**	**1,100**		**11,000**	**J**
Biphenyl (1,1-Biphenyl)		92-52-4	NE	9,100	UJ	5,600	UJ	3,100	U	**600**	**J**
Bis(2-chloroethoxy)methane		111-91-1	NE	4,300	UJ	2,600	UJ	1,500	U	2,300	UJ
Bis(2-chloroethyl)ether		111-44-4	NE	3,600	UJ	2,200	UJ	1,200	U	1,900	UJ
2,2-oxybis(1-Chloropropane)		108-60-1	NE	4,800	UJ	3,000	UJ	1,600	UJ	2,500	UJ
Bis(2-ethylhexyl)phthalate		117-81-7	360000	4,000	UJ	2,500	UJ	1,400	U	**1000**	**J**
4-Bromophenyl phenyl ether		101-55-3	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ

Table 5C. Creek Shallow Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

			Task Description	Scajaquada Creek Shallow Sediment Boring Sample Analytical Results							
			Sample Name	SED-LSC-5/0-0.5		SED-LSC-6/0-0.5		SED-LSC-7/0-0.5		SED-LSC-8/0-0.5	
			Start Depth	0		0		0		0	
			End Depth	0.5		0.5		0.5		0.5	
			Depth Unit	ft		ft		ft		ft	
			Sample Date	11/11/2021		11/3/2021		11/3/2021		11/3/2021	
			Parent Sample								
Analyte	**Units**	**CAS No. or Identifier**	**Freshwater Sediment Guidance Values Class B**								
Butyl benzyl phthalate		85-68-7	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
Caprolactam		105-60-2	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
Carbazole		86-74-8	NE	4,000	UJ	2,500	UJ	1,400	U	**330**	**J**
4-Chloro-3-methylphenol		59-50-7	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
4-Chloroaniline		106-47-8	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
2-Chloronaphthalene		91-58-7	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
2-Chlorophenol		95-57-8	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
4-Chlorophenyl phenyl ether		7005-72-3	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
Chrysene		218-01-9	NE	**1,200**	**J**	**1,100**	**J**	**880**		**9,200**	**J**
Dibenz(a,h)anthracene		53-70-3	NE	2,400	UJ	1,500	UJ	**160**	**J**	**1,200**	**J**
Dibenzofuran		132-64-9	NE	4,000	UJ	2,500	UJ	1,400	U	**510**	**J**
3,3-Dichlorobenzidine		91-94-1	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
2,4-Dichlorophenol		120-83-2	NE	3,600	UJ	2,200	UJ	1,200	U	1,900	UJ
Diethyl phthalate		84-66-2	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
Dimethyl phthalate		131-11-3	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
2,4-Dimethylphenol		105-67-9	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
Di-n-butyl phthalate		84-74-2	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
4,6-Dinitro-2-methylphenol		534-52-1	NE	10,000	UJ	6,400	UJ	3,500	U	5,500	UJ
2,4-Dinitrophenol		51-28-5	NE	19,000	UJ	12,000	UJ	6,500	U	10,000	UJ
2,4-Dinitrotoluene		121-14-2	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
2,6-Dinitrotoluene		606-20-2	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
Di-n-octyl phthalate		117-84-0	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
Fluoranthene		206-44-0	NE	**2,200**	**J**	**1,900**	**J**	**1,500**		**17,000**	**J**
Fluorene		86-73-7	NE	4,000	UJ	2,500	UJ	1,400	U	**3,400**	**J**
Hexachlorobenzene		118-74-1	NE	2,400	UJ	1,500	UJ	810	U	1,300	UJ
1,3-Hexachlorobutadiene (C-46)		87-68-3	12000	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
Hexachlorocyclopentadiene		77-47-4	8100	11,000	UJ	7,000	UJ	3,900	U	6,100	UJ
Hexachloroethane		67-72-1	NE	3,200	UJ	2,000	UJ	1,100	U	1,700	UJ
Indeno(1,2,3-cd)pyrene		193-39-5	NE	**850**	**J**	**1,400**	**J**	**930**	**J**	**5,200**	**J**
Isophorone		78-59-1	NE	3,600	UJ	2,200	UJ	1,200	U	1,900	UJ
2-Methylnaphthalene		91-57-6	NE	4,800	UJ	3,000	UJ	1,600	U	**620**	**J**
2-Methylphenol (o-Cresol)		95-48-7	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
3,4-Methylphenol (m,p-Cresol)		108394/106445	NE	5,800	UJ	3,500	UJ	2,000	U	3,000	UJ
Naphthalene		91-20-3	NE	4,000	UJ	2,500	UJ	1,400	U	**900**	**J**
2-Nitroaniline		88-74-4	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
3-Nitroaniline		99-09-2	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ

Table 5C. Creek Shallow Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

				Task Description	Scajaquada Creek Shallow Sediment Boring Sample Analytical Results							
				Sample Name	SED-LSC-5/0-0.5		SED-LSC-6/0-0.5		SED-LSC-7/0-0.5		SED-LSC-8/0-0.5	
				Start Depth	0		0		0		0	
				End Depth	0.5		0.5		0.5		0.5	
				Depth Unit	ft		ft		ft		ft	
				Sample Date	11/11/2021		11/3/2021		11/3/2021		11/3/2021	
				Parent Sample								
Analyte	**Units**	**CAS No. or Identifier**	**Freshwater Sediment Guidance Values Class B**									
4-Nitroaniline		100-01-6	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
Nitrobenzene		98-95-3	NE	3,600	UJ	2,200	UJ	1,200	U	1,900	UJ
2-Nitrophenol		88-75-5	NE	8,600	UJ	5,300	UJ	2,900	U	4,600	UJ
4-Nitrophenol		100-02-7	NE	5,600	UJ	3,400	UJ	1,900	U	3,000	UJ
N-Nitrosodiphenylamine (NDFA)		86-30-6	NE	3,200	UJ	2,000	UJ	1,100	U	1,700	UJ
N-Nitrosodi-n-propylamine (NDPA)		621-64-7	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
Pentachlorophenol		87-86-5	19000	3,200	UJ	2,000	UJ	1,100	U	1,700	UJ
Phenanthrene		85-01-8	NE	**1000**	**J**	**890**	**J**	**740**	**J**	**28,000**	**J**
Phenol		108-95-2	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
Pyrene		129-00-0	NE	**2,500**	**J**	**2,300**	**J**	**1,600**		**31,000**	**J**
1,2,4,5-Tetrachlorobenzene		95-94-3	14000	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
2,3,4,6-Tetrachlorophenol		58-90-2	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
2,4,5-Trichlorophenol		95-95-4	NE	4,000	UJ	2,500	UJ	1,400	U	2,100	UJ
2,4,6-Trichlorophenol		88-06-2	NE	2,400	UJ	1,500	UJ	810	U	1,300	UJ
Total PAH (16) (ND=0)		TPAH16_ND0	35000	**12,350**		**12,640**		**10,520**		**151,000**	
Total PAH (17) (ND=0)		TPAH17_ND0	35000	**12,350**		**12,640**		**10,520**		**151,620**	
PCBs (SW 8082A)	ug/kg										
Aroclor 1016		12674-11-2	NE	134	UJ	485	UJ	246	U	43.3	UJ
Aroclor 1221		11104-28-2	NE	134	UJ	485	UJ	246	U	43.3	UJ
Aroclor 1232		11141-16-5	NE	134	UJ	485	UJ	246	U	43.3	UJ
Aroclor 1242		53469-21-9	NE	134	UJ	485	UJ	246	U	43.3	UJ
Aroclor 1248		12672-29-6	NE	**313**	**J**	**195**	**J**	**153**	**J**	**47.9**	**J**
Aroclor 1254		11097-69-1	NE	**181**	**J**	**126**	**J**	**84.4**	**J**	**24**	**J**
Aroclor 1260		11096-82-5	NE	**131**	**J**	485	UJ	**55.6**	**J**	**15.7**	**J**
Aroclor 1262		37324-23-5	NE	134	UJ	485	UJ	246	U	43.3	UJ
Aroclor 1268		11100-14-4	NE	134	UJ	485	UJ	246	U	43.3	UJ
Total PCBs (Lab calculated)		1336-36-3	1000	**625**	**J**	**321**	**J**	**293**	**J**	**87.6**	**J**
PESTICIDES (SW 8081B)	ug/kg										
Aldrin		309-00-2	NE	19.2	R	115	UJ	68.7	U	101	UJ
alpha-BHC (Hexachlorocyclohexane)		319-84-6	NE	8.02	R	47.8	UJ	28.6	U	42.2	UJ
beta-BHC (beta-Hexachlorocyclohexane)		319-85-7	NE	19.2	R	115	UJ	68.7	U	101	UJ
gamma-BHC (gamma-Hexachlorocyclohexane) (Lindane)		58-89-9	78	8.02	R	47.8	UJ	28.6	U	42.2	UJ
delta-BHC (delta-Hexachlorocyclohexane)		319-86-8	NE	19.2	R	115	UJ	68.7	U	101	UJ
Chlordane (Technical)		12789-03-6	NE	160	R	955	UJ	572	U	845	UJ
alpha-chlordane		5103-71-9	NE	24	R	143	UJ	85.8	U	127	UJ
gamma-Chlordane		5103-74-2	NE	24	R	143	UJ	85.8	U	127	UJ

Table 5C. Creek Shallow Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

				Task Description	Scajaquada Creek Shallow Sediment Boring Sample Analytical Results							
				Sample Name	SED-LSC-5/0-0.5		SED-LSC-6/0-0.5		SED-LSC-7/0-0.5		SED-LSC-8/0-0.5	
				Start Depth	0		0		0		0	
				End Depth	0.5		0.5		0.5		0.5	
				Depth Unit	ft		ft		ft		ft	
				Sample Date	11/11/2021		11/3/2021		11/3/2021		11/3/2021	
				Parent Sample								
Analyte	**Units**	**CAS No. or Identifier**	**Freshwater Sediment Guidance Values Class B**									
4,4'-DDT (p,p'-DDT)		50-29-3	48000	36.1	R	215	UJ	129	U	190	UJ
4,4'-DDE (p,p'-DDE)		72-55-9	NE	19.2	R	115	UJ	68.7	U	101	UJ
4,4'-DDD (p,p-DDD)		72-54-8	NE	19.2	R	115	UJ	68.7	U	101	UJ
Dieldrin		60-57-1	780	12	R	71.6	UJ	42.9	U	63.4	UJ
alpha-Endosulfan (I)		959-98-8	NE	19.2	R	115	UJ	68.7	U	101	UJ
beta-Endosulfan (II)		33213-65-9	NE	19.2	R	115	UJ	68.7	U	101	UJ
Endosulfan sulfate		1031-07-8	NE	8.02	R	47.8	UJ	28.6	U	42.2	UJ
Endrin		72-20-8	220	8.02	R	47.8	UJ	28.6	U	42.2	UJ
Endrin aldehyde		7421-93-4	NE	24	R	143	UJ	85.8	UJ	127	UJ
Endrin ketone		53494-70-5	NE	19.2	R	115	UJ	68.7	U	101	UJ
Heptachlor		76-44-8	10000	9.62	R	57.3	UJ	34.3	U	50.7	UJ
Heptachlor epoxide		1024-57-3	2100	36.1	R	215	UJ	129	U	190	UJ
Methoxychlor		72-43-5	NE	36.1	R	215	UJ	129	U	190	UJ
Toxaphene		8001-35-2	250	361	R	2,150	UJ	1,290	U	1,900	UJ
HERBICIDES (SW 8151A)	ug/kg										
2,4-D (2,4-Dichlorophenoxyacetic acid)		94-75-7	NE	665	UJ	858	UJ	479	U	708	UJ
2,4,5-T (2,4,5-Trichlorophenoxyacetic acid)		93-76-5	NE	665	UJ	858	UJ	479	U	708	UJ
2,4,5-TP (Silvex)		93-72-1	NE	665	UJ	858	UJ	479	U	708	UJ
CYANIDES (SW 9012B)	mg/kg										
Total Cyanide		57-12-5	NE	3.8	UJ	5.2	UJ	2.6	UJ	4.3	UJ
METALS (SW 6010D)	mg/kg										
Aluminum		7429-90-5	NE	**10,400**	**J**	**9,290**	**J**	**5,750**		**11,200**	**J**
Antimony		7440-36-0	NE	15.8	UJ	20.4	UJ	11.2	U	17	UJ
Arsenic		7440-38-2	33	**6.33**	**J**	**9.08**	**J**	**5.66**		**7.44**	**J**
Barium		7440-39-3	NE	**148**	**J**	**128**	**J**	**77.9**		**136**	**J**
Beryllium		7440-41-7	NE	1.58	UJ	**0.489**	**J**	**0.334**	**J**	**0.612**	**J**
Cadmium		7440-43-9	5	**1.17**	**J**	**1.96**	**J**	**1.18**	**J**	**2.45**	**J**
Calcium		7440-70-2	NE	**316,000**	**J**	**81,800**	**J**	**92,800**		**75,500**	**J**
Chromium		7440-47-3	110	**31.1**	**J**	**44**	**J**	**28.2**		**54.6**	**J**
Cobalt		7440-48-4	NE	**4.94**	**J**	**7.86**	**J**	**5.49**		**8.98**	**J**
Copper		7440-50-8	150	**81.1**	**J**	**152**	**J**	**84.2**	**J**	**151**	**J**
Iron		7439-89-6	NE	**16,300**	**J**	**25,800**	**J**	**17,300**	**J**	**26,400**	**J**
Lead		7439-92-1	130	**95**	**J**	**199**	**J**	**101**	**J**	**179**	**J**
Magnesium		7439-95-4	NE	**70,200**	**J**	**9,850**	**J**	**6,730**		**13,000**	**J**
Manganese		7439-96-5	NE	**2,230**	**J**	**348**	**J**	**236**	**J**	**379**	**J**
Mercury (SW 7471B)		7439-97-6	1	0.256	UJ	**0.261**	**J**	**0.156**	**J**	**0.342**	**J**

Table 5C. Creek Shallow Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

				Task Description	Scajaquada Creek Shallow Sediment Boring Sample Analytical Results							
				Sample Name	SED-LSC-5/0-0.5		SED-LSC-6/0-0.5		SED-LSC-7/0-0.5		SED-LSC-8/0-0.5	
				Start Depth	0		0		0		0	
				End Depth	0.5		0.5		0.5		0.5	
				Depth Unit	ft		ft		ft		ft	
				Sample Date	11/11/2021		11/3/2021		11/3/2021		11/3/2021	
				Parent Sample								
Analyte	**Units**	**CAS No. or Identifier**	**Freshwater Sediment Guidance Values Class B**									
Nickel		7440-02-0	49	**14.2**	**J**	**23.3**	**J**	**16.3**		**27.9**	**J**
Potassium		7440-09-7	NE	**1,680**	**J**	**1,460**	**J**	**774**		**1,510**	**J**
Selenium		7782-49-2	NE	6.33	UJ	**1.63**	**J**	**1.36**	**J**	**1.84**	**J**
Silver		7440-22-4	2.2	3.16	UJ	4.07	UJ	2.23	U	3.4	UJ
Sodium		7440-23-5	NE	**1,530**	**J**	**654**	**J**	**599**	**J**	**802**	**J**
Thallium		7440-28-0	NE	**2.5**	**J**	8.15	UJ	4.46	U	6.8	UJ
Vanadium		7440-62-2	NE	**15.3**	**J**	**23**	**J**	**16**		**26.3**	**J**
Zinc		7440-66-6	460	**304**	**J**	**589**	**J**	**324**	**J**	**645**	**J**
VPH/EPH	mg/kg										
C5-C8 Aliphatics (MAVPH)		ALIPH5-8	NE	37.5	UJ	30	UJ	15.4	UJ	30.4	UJ
C5-C8 Aliphatics, Adjusted (MAVPH)		ALIPH5-8ADJ	NE	37.5	UJ	30	UJ	15.4	UJ	30.4	UJ
C9-C12 Aliphatics (MAVPH)		ALIPH9-12	NE	37.5	UJ	30	UJ	15.4	UJ	30.4	UJ
C9-C12 Aliphatics, Adjusted (MAVPH)		ALIPH9-12ADJ	NE	37.5	UJ	30	UJ	15.4	UJ	30.4	UJ
C9-C10 Aromatics (MAVPH)		AROM9-10	NE	37.5	UJ	30	UJ	15.4	UJ	30.4	UJ
Total EPH Fractions (NJ EPH R3)		TEPH	NE	**303**	**J**	**526**	**J**	177	U	269	UJ
GENERAL CHEMISTRY	mg/kg										
Ammonia (SM 4500-NH3 BH)		7664-41-7	NE	**53**	**J**	**84**	**J**	**29**	**J**	**85**	**J**
Percent Solids (SM 2540G)	%	SOLIDS	NE	**24.5**	**J**	**19**		**34.2**		**22.8**	
Sulfide (SW 9030B)		18496-25-8	NE	**10,000**	**J**	**5,200**	**J**	**2,200**	**J**	**4,800**	**J**
TOTAL ORGANIC CARBON	%										
Total Organic Carbon (SW 9060A)		TOC	NE	**11.2**	**J**	**4.67**	**J**	**7.95**		**7.96**	**J**

Table 5C. Creek Shallow Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

Notes:
Sample depth is upper 0.5 feet of surface sediment
mg/kg = milligrams/kilogram or parts per million (ppm)
% = Percent
ug/kg = micrograms per kilogram

PAH = Polycyclic Aromatic Hydrocarbon
PCB = Polychlorinated Biphenyl
SVOC = Semi-Volatile Organic Compound
VOC = Volatile Organic Compound

CAS No. = Chemical Abstracts Service Number
ND = Not Detected
NE = Not Established

Bolding indicates a detected result concentration
Shading and bolding indicate that the detected concentration is above the maximum range of sediment guidance values

Validation Qualifiers:
J = The result is an estimated value.
JN = The analyte is presumptively present at an approximated quantity.
R = The result is rejected.
U = The result was not detected above the reporting limit.
UJ = The results was not detected at or above the reporting limit shown and the reporting limit is estimated.

1,4-Dioxane - Rejected Data

Due to the extremely low calibration response factor for 1,4-dioxane under Method 8260C, this compound is typically rejected during validation as it does not meet the minimal criteria set for accurate quantitation. Non-detect results for 1,4-dioxane are rejected as the presence or absence of the compound cannot be verified. Positive results are estimated due to the high variability inherent in the quantitation when using an extremely low response factor.

Table 5D. Creek Deeper Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

							Task Description	Creek Deeper Sediment Boring Sample Analytical Results																									
							Sample Name	SED-BGL-1/12-14		SED-LSC-1/11-15		SED-LSC-2/14-16		SED-LSC-3/13-16		SED-LSC-4/14-16		SED-LSC-5/14-16		SED-LSC-6/12-19.5		SED-LSC-7/12-18		SED-LSC-8/6.8-15								
							Start Depth	562.2		563.1		560.1		561.1		560.0		560.0		561.2		561.8		566.6								
							End Depth	560.2		559.1		558.1		558.1		550.0		550.0		553.7		553.8		558.4								
							Depth Unit	Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft								
							Sample Depth Interval (ft) below Top of Sediment	1.0 to 3.0		4.5 to 8.5		8.0 to 10.0		5.2 to 8.2		4.7 to 6.7		5.2 to 7.2		3.5 to 11.0		4.0 to 12.0		0 to 8.2								
							Sample Date	11/10/2021		11/9/2021		11/9/2021		11/9/2021		11/10/2021		11/10/2021		11/3/2021		11/3/2021		11/3/2021								
Analyte	**Units**	**CAS No. or Identifier**	**375-6.8(a) Unrestricted SCO**	**375-6.8(b) Protection of Ecological Resources SCO**	**Freshwater Sediment Guidance Values Class B**	**NY CP-51 RESTRICTED USE ECO SSCO**																											
VOCs (SW 8260C)	ug/kg																															
Acetone		67-64-1	50	2200	NE	NE	9.4	UJ	3,000	U	1,300	U	2,000	U	**5**	J	**12**		920	U	**37**	J	**40**	J									
Benzene		71-43-2	60	70000	1900	NE	0.47	U	**11000**		**12000**		**46000**		**43**		**3200**		**460**		**350**	J	**550**										
Bromochloromethane		74-97-5	NE	NE	NE	NE	1.9	U	590	U	270	U	400	U	1.7	U	2.5	U	180	U	4	UJ	2.5	UJ									
Bromodichloromethane		75-27-4	NE	NE	NE	NE	0.47	U	150	UJ	67	UJ	100	U	0.42	U	0.62	U	46	U	0.99	UJ	0.63	UJ									
Bromoform		75-25-2	NE	NE	NE	NE	3.8	U	1,200	U	530	U	810	U	3.3	U	5	U		U	7.9	UJ	5	UJ									
Bromomethane		74-83-9	NE	NE	NE	NE	1.9	U	590	U	270	U	400	U	1.7	U	2.5	UJ	180	U	4	UJ	2.5	UJ									
Carbon disulfide		75-15-0	NE	NE	NE	NE	9.4	U	3,000	UJ	1,300	UJ	2,000	UJ	8.4	UJ	12	UJ	920	U	20	UJ	13	UJ									
Carbon tetrachloride		56-23-5	760	NE	9600	NE	0.94	U	300	U	130	U	200	U	0.84	U	1.2	U	92	U	2	UJ	1.3	UJ									
Chlorobenzene		108-90-7	1100	40000	1700	NE	0.47	U	150	U	67	U	100	U	0.42	U	0.62	U	46	U	0.99	UJ	0.63	UJ									
Chloroethane		75-00-3	NE	NE	NE	NE	1.9	UJ	590	U	270	U	400	U	1.7	U	2.5	UJ	180	U	4	UJ	**50**	J									
Chloroform (Trichloromethane)		67-66-3	370	12000	NE	NE	1.4	U	440	U	200	U	300	U	1.2	U	1.9	U	140	U	3	UJ	1.9	UJ									
Chloromethane		74-87-3	NE	NE	NE	NE	3.8	U	1,200	U	530	U	810	U	3.3	U	5	U	370	U	7.9	UJ	5	UJ									
Cyclohexane		110-82-7	NE	NE	NE	NE	9.4	U	3,000	U	1,300	U	2,000	U	8.4	U	**1.2**	J	**56**	J	**1.2**	J	**2.8**	J									
1,2-Dibromo-3-chloropropane		96-12-8	NE	NE	NE	NE	2.8	UJ	890	U	400	U	610	U	2.5	U	3.8	U	280	U	6	UJ	3.8	UJ									
Dibromochloromethane		124-48-1	NE	NE	NE	10000	0.94	U	300	U	130	U	200	U	0.84	U	1.2	U	92	U	2	UJ	1.3	UJ									
1,2-Dibromoethane (EDB)		106-93-4	NE	NE	NE	NE	0.94	U	300	U	130	U	200	U	0.84	U	1.2	U	92	U	2	UJ	1.3	UJ									
1,2-Dichlorobenzene (o-DCB)		95-50-1	1100	NE	2500	NE	1.9	U	590	U	270	U	**69**	J	1.7	U	**0.3**	J	180	U	4	UJ	**0.2**	J									
1,3-Dichlorobenzene (m-DCB)		541-73-1	2400	NE	7100	NE	1.9	U	590	U	270	U	400	U	1.7	U	2.5	U	180	U	4	UJ	2.5	UJ									
1,4-Dichlorobenzene (p-DCB)		106-46-7	1800	20000	3300	NE	1.9	U	590	U	270	U	**150**	J	1.7	U	**0.34**	J	180	U	4	UJ	2.5	UJ									
Dichlorodifluoromethane (Freon 12)		75-71-8	NE	NE	NE	NE	9.4	U	3,000	U	1,300	U	2,000	UJ	8.4	U	12	UJ	920	U	20	UJ	13	UJ									
1,1-Dichloroethane		75-34-3	270	NE	NE	NE	0.94	U	300	U	130	U	200	U	0.84	U	1.2	U	92	U	2	UJ	1.3	UJ									
1,2-Dichloroethane		107-06-2	20	10000	NE	NE	0.94	U	300	U	130	U	200	U	**1.1**		1.2	U	92	U	2	UJ	1.3	UJ									
1,1-Dichloroethene		75-35-4	330	NE	4700	NE	0.94	U	300	U	130	U	200	U	0.84	U	1.2	U	92	U	2	UJ	1.3	UJ									
cis-1,2-Dichloroethene		156-59-2	250	NE	NE	NE	0.94	U	300	U	130	U	200	U	**0.5**	J	**5.2**		**1,900**		**1.7**	J	**0.64**	J									
trans-1,2-Dichloroethene		156-60-5	190	NE	11000	NE	1.4	U	440	U	200	U	300	U	1.2	U	1.9	U	**300**		3	UJ	**0.18**	J									
1,2-Dichloropropane		78-87-5	NE	NE	NE	700000	0.94	U	300	U	130	U	200	U	0.84	U	1.2	U	92	U	2	UJ	1.3	UJ									
cis-1,3-Dichloropropene		10061-01-5	NE	NE	NE	NE	0.47	U	150	U	67	U	100	U	0.42	U	0.62	U	46	U	0.99	UJ	0.63	UJ									
trans-1,3-Dichloropropene		10061-02-6	NE	NE	NE	NE	0.94	U	300	U	130	U	200	U	0.84	U	1.2	U	92	U	2	UJ	1.3	UJ									
1,4-Dioxane		123-91-1	100	100	NE	NE		R		R		R		R		R		R		R		R		R								
Ethylbenzene		100-41-4	1000	NE	3700	NE	**0.22**	J	**110000**		**76000**		**13000**		**120**		**12000**		**17000**		**700**		**5900**										
2-Hexanone		591-78-6	NE	NE	NE	NE	9.4	UJ	3,000	U	1,300	U	2,000	U	8.4	U	12	U	920	U	20	UJ	13	UJ									
Isopropylbenzene		98-82-8	NE	NE	1800	NE	0.94	U	**12000**		**5300**		**6400**	J	**9.7**		**660**		**1900**		**250**	J	**710**										
Methyl acetate		79-20-9	NE	NE	NE	NE	3.8	UJ	**1,400**	J	530	U	**700**	J	3.3	U	5	U	**500**		**61**	J	**360**	J									
Methyl ethyl ketone (2-Butanone)		78-93-3	120	100000	NE	NE	9.4	UJ	3,000	U	1,300	U	**480**	J	8.4	U	12	U	920	U	**8.4**	J	13	UJ									
Methyl tert-butyl ether (MTBE)		1634-04-4	930	NE	NE	NE	1.9	U	590	U	270	U	400	U	1.7	U	2.5	U	180	U	4	UJ	2.5	UJ									
4-Methyl-2-pentanone (MIBK)		108-10-1	NE	NE	NE	NE	9.4	UJ	3,000	U	1,300	U	2,000	U	8.4	U	12	U	920	U	20	UJ	13	UJ									
Methylcyclohexane		108-87-2	NE	NE	NE	NE	3.8	U	**190**	J	530	U	**310**	J	3.3	U	**4.2**	J	**170**	J	**5.8**	J	**94**	J									
Methylene chloride		75-09-2	50	12000	NE	NE	4.7	U	1,500	U	670	U	1000	UJ	4.2	U	6.2	U	460	U	9.9	UJ	6.3	UJ									
Styrene		100-42-5	NE	NE	NE	300000	0.94	U	**260**	J	**180**		**170**	J	**2**		**3.7**	J	**360**		**2**	J	**0.63**	J									
1,1,2,2-Tetrachloroethane		79-34-5	NE	NE	5400	NE	0.47	U	150	U	67	U	100	U	0.42	U	0.62	U	46	U	0.99	UJ	0.63	UJ									
Tetrachloroethene (PCE)		127-18-4	1300	2000	57000	NE	0.47	U	150	U	67	U	100	U	0.42	U	0.62	U	46	U	0.99	UJ	0.63	UJ									
Toluene		108-88-3	700	36000	4500	NE	0.94	U	**630**		**1,400**		**360**		**15**		**76**		**1,500**		**140**	J	**100**										

Table 5D. Creek Deeper Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

							Task Description	Creek Deeper Sediment Boring Sample Analytical Results																					
							Sample Name	SED-BGL-1/12-14		SED-LSC-1/11-15		SED-LSC-2/14-16		SED-LSC-3/13-16		SED-LSC-4/14-16		SED-LSC-5/14-16		SED-LSC-6/12-19.5		SED-LSC-7/12-18		SED-LSC-8/6.8-15					
							Start Depth	562.2		563.1		560.1		561.1		560.0		560.0		561.2		561.8		566.6					
							End Depth	560.2		559.1		558.1		558.1		550.0		550.0		553.7		553.8		558.4					
							Depth Unit	Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft					
							Sample Depth Interval (ft) below Top of Sediment	1.0 to 3.0		4.5 to 8.5		8.0 to 10.0		5.2 to 8.2		4.7 to 6.7		5.2 to 7.2		3.5 to 11.0		4.0 to 12.0		0 to 8.2					
							Sample Date	11/10/2021		11/9/2021		11/9/2021		11/9/2021		11/10/2021		11/10/2021		11/3/2021		11/3/2021		11/3/2021					
Analyte	Units	CAS No. or Identifier	375-6.8(a) Unrestricted SCO	375-6.8(b) Protection of Ecological Resources SCO	Freshwater Sediment Guidance Values Class B	NY CP-51 RESTRICTED USE ECO SSCO																							
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)		76-13-1	NE	NE	NE	NE		3.8	U	1,200	U	530	U	810	U	3.3	U	5	U	370	U	7.9	UJ	5	UJ				
1,2,3-Trichlorobenzene		87-61-6	NE	NE	2800	20000		1.9	U	590	U	270	U	400	U	1.7	U	2.5	U	180	U	4	UJ	2.5	UJ				
1,2,4-Trichlorobenzene		120-82-1	NE	NE	55000	20000		1.9	U	590	U	270	U	400	U	1.7	U	2.5	U	180	U	4	UJ	2.5	UJ				
1,1,1-Trichloroethane (TCA)		71-55-6	680	NE	3500	NE		0.47	U	150	U	67	U	100	U	0.42	U	0.62	U	46	U	0.99	UJ	0.63	UJ				
1,1,2-Trichloroethane		79-00-5	NE	NE	3500	NE		0.94	U	300	U	130	U	200	U	0.84	U	1.2	U	92	U	2	UJ	1.3	UJ				
Trichloroethene (TCE)		79-01-6	470	2000	8600	NE		0.47	U	150	U	67	U	100	U	0.42	U	**0.44**	**J**	**79**		**0.62**	**J**	0.63	UJ				
Trichlorofluoromethane (Freon 11)		75-69-4	NE	NE	NE	NE		3.8	UJ	1,200	U	530	U	810	UJ	3.3	U	5	U	370	U	7.9	UJ	5	UJ				
Vinyl chloride		75-01-4	20	NE	NE	NE		0.94	UJ	300	U	130	U	200	U	**0.81**	**J**	**2.9**		**620**		**1.4**	**J**	**0.88**	**J**				
o-Xylene		95-47-6	260	NE	7200	NE		0.94	U	**25000**		**14000**		**4,400**	**J**	**32**		**2,500**		**4,100**		**720**	**J**	**1,600**					
m/p-Xylene		179601-23-1	260	260	NE	NE		1.9	U	**29,000**		**36,000**		**1,700**		**63**		**4,400**		**8,400**		**1,100**	**J**	**1,100**					
Total BTEX (ND=0)		TBTEX_ND0	NE	NE	NE	NE		**0.22**		**175,630**		**139,400**		**65,460**		**273**		**22,176**		**31,460**		**3,010**		**9,250**					
SVOCs (SW 8270D)	ug/kg																												
Acenaphthene		83-32-9	20000	20000	NE	NE		160	U	**37,000**		**20,000**		**110,000**		**320**		**1,700**		**25,000**		**3,300**		**5,400**	**J**				
Acenaphthylene		208-96-8	100000	NE	NE	NE		160	U	**3,000**		**2,800**		**3,700**		**150**	**J**	**110**	**J**	**5,200**		**360**		**650**	**J**				
Acetophenone		98-86-2	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ				
Anthracene		120-12-7	100000	NE	NE	NE		120	U	**17,000**		**12,000**		**45,000**		**200**		**740**		**17,000**		**1,700**		**3,000**	**J**				
Atrazine		1912-24-9	NE	NE	NE	NE		160	U	2,600	U	1,700	U	2,400	U	170	U	180	U	1,700	U	160	U	190	UJ				
Benzaldehyde		100-52-7	NE	NE	NE	NE		270	U	4,200	U	2,700	U	4,000	U	280	U	290	U	2,800	U	270	U	310	UJ				
Benzo(a)anthracene		56-55-3	1000	NE	NE	NE		**28**	**J**	**8,900**		**5,500**		**20,000**		**130**		**380**		**9,400**		**950**		**2,100**	**J**				
Benzo(b)fluoranthene		205-99-2	1000	NE	NE	NE		120	U	**5,500**		**3,000**		**12,000**		**80**	**J**	**230**		**5,800**		**590**		**1,400**	**J**				
Benzo(k)fluoranthene		207-08-9	800	NE	NE	NE		120	U	**1,200**	**J**	**900**	**J**	**3,000**		130	U	**77**	**J**	**1,800**		**150**		**460**	**J**				
Benzo(g,h,i)perylene		191-24-2	100000	NE	NE	NE		160	U	**3,600**		**2,500**		**8,700**		**49**	**J**	**150**	**J**	**3,600**		**380**		**890**	**J**				
Benzo(a)pyrene		50-32-8	1000	2600	NE	NE		160	U	**8,000**		**4,600**		**16,000**		**100**	**J**	**340**		**8,400**		**820**		**1,800**	**J**				
Biphenyl (1,1-Biphenyl)		92-52-4	NE	NE	NE	60000		460	U	**11,000**		**6,400**		**31,000**		**140**	**J**	**470**	**J**	**8,000**		**880**		**1,300**	**J**				
Bis(2-chloroethoxy)methane		111-91-1	NE	NE	NE	NE		220	U	3,500	U	2,200	U	3,200	U	230	U	240	U	2,300	U	220	U	260	UJ				
Bis(2-chloroethyl)ether		111-44-4	NE	NE	NE	NE		180	U	2,900	U	1,900	U	2,700	U	190	U	200	U	1,900	U	180	U	210	UJ				
2,2-oxybis(1-Chloropropane)		108-60-1	NE	NE	NE	NE		240	UJ	3,900	UJ	2,500	UJ	3,600	UJ	260	U	260	UJ	2,600	UJ	240	UJ	280	UJ				
Bis(2-ethylhexyl)phthalate		117-81-7	NE	NE	14	239000		200	U	3,200	UJ	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ				
4-Bromophenyl phenyl ether		101-55-3	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ				
Butyl benzyl phthalate		85-68-7	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ				
Caprolactam		105-60-2	NE	NE	NE	NE		200	U	3,200	U	2,100	UJ	3,000	UJ	220	U	220	U	2,200	UJ	200	U	240	UJ				
Carbazole		86-74-8	NE	NE	NE	NE		200	U	3,200	U	2,100	U	**1000**	**J**	220	U	220	U	2,200	U	200	U	**96**	**J**				
4-Chloro-3-methylphenol		59-50-7	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ				
4-Chloroaniline		106-47-8	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	UJ	220	U	2,200	UJ	200	UJ	240	UJ				
2-Chloronaphthalene		91-58-7	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ				
2-Chlorophenol		95-57-8	NE	NE	NE	800		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ				
4-Chlorophenyl phenyl ether		7005-72-3	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ				
Chrysene		218-01-9	1000	NE	NE	NE		**22**	**J**	**7,200**		**4,200**		**16,000**		**96**	**J**	**310**		**7,300**		**780**		**1,800**	**J**				
Dibenz(a,h)anthracene		53-70-3	330	NE	NE	NE		120	U	**720**	**J**	**480**	**J**	**2,000**		130	U	**32**	**J**	**740**	**J**	**90**	**J**	**220**	**J**				
Dibenzofuran		132-64-9	7000	NE	NE	NE		200	U	**2,600**	**J**	**1,400**	**J**	**7,300**		**39**	**J**	**140**	**J**	**2,200**		**320**		**490**	**J**				
3,3-Dichlorobenzidine		91-94-1	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ				
2,4-Dichlorophenol		120-83-2	NE	NE	NE	20000		180	U	2,900	U	1,900	U	2,700	U	190	U	200	U	1,900	U	180	U	210	UJ				
Diethyl phthalate		84-66-2	NE	NE	NE	100000		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ				
Dimethyl phthalate		131-11-3	NE	NE	NE	200000		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ				

Table 5D. Creek Deeper Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

							Task Description	Creek Deeper Sediment Boring Sample Analytical Results																	
							Sample Name	SED-BGL-1/12-14		SED-LSC-1/11-15		SED-LSC-2/14-16		SED-LSC-3/13-16		SED-LSC-4/14-16		SED-LSC-5/14-16		SED-LSC-6/12-19.5		SED-LSC-7/12-18		SED-LSC-8/6.8-15	
							Start Depth	562.2		563.1		560.1		561.1		560.0		560.0		561.2		561.8		566.6	
							End Depth	560.2		559.1		558.1		558.1		550.0		550.0		553.7		553.8		558.4	
							Depth Unit	Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft	
							Sample Depth Interval (ft) below Top of Sediment	1.0 to 3.0		4.5 to 8.5		8.0 to 10.0		5.2 to 8.2		4.7 to 6.7		5.2 to 7.2		3.5 to 11.0		4.0 to 12.0		0 to 8.2	
							Sample Date	11/10/2021		11/9/2021		11/9/2021		11/9/2021		11/10/2021		11/10/2021		11/3/2021		11/3/2021		11/3/2021	
Analyte	**Units**	**CAS No. or Identifier**	**375-6.8(a) Unrestricted SCO**	**375-6.8(b) Protection of Ecological Resources SCO**	**Freshwater Sediment Guidance Values Class B**	**NY CP-51 RESTRICTED USE ECO SSCO**																			
2,4-Dimethylphenol		105-67-9	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
Di-n-butyl phthalate		84-74-2	NE	NE	NE	14		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
4,6-Dinitro-2-methylphenol		534-52-1	NE	NE	NE	NE		520	U	8,400	UJ	5,400	R	7,800	R	560	U	570	U	5,600	U	530	U	620	UJ
2,4-Dinitrophenol		51-28-5	NE	NE	NE	20000		970	U	15,000	UJ	10,000	U	14,000	U	1000	U	1000	U	10,000	U	970	U	1,100	UJ
2,4-Dinitrotoluene		121-14-2	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
2,6-Dinitrotoluene		606-20-2	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
Di-n-octyl phthalate		117-84-0	NE	NE	NE	NE		200	UJ	3,200	UJ	2,100	U	3,000	U	220	U	220	UJ	2,200	U	200	U	240	UJ
Fluoranthene		206-44-0	100000	NE	NE	NE		**35**	**J**	**16,000**		**9,000**		**42,000**		**210**		**680**		**17,000**		**1,600**		**3,500**	**J**
Fluorene		86-73-7	30000	30000	NE	NE		200	U	**15,000**		**9,800**		**35,000**		**190**	**J**	**640**		**13,000**		**1,300**		**2,400**	**J**
Hexachlorobenzene		118-74-1	330	NE	NE	NE		120	U	1,900	U	1,200	U	1,800	U	130	U	130	U	1,300	U	120	U	140	UJ
1,3-Hexachlorobutadiene (C-46)		87-68-3	NE	NE	12000	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
Hexachlorocyclopentadiene		77-47-4	NE	NE	8100	10000		580	U	9,200	U	5,900	UJ	8,600	UJ	620	U	630	U	6,200	U	580	U	680	UJ
Hexachloroethane		67-72-1	NE	NE	NE	NE		160	U	2,600	U	1,700	U	2,400	U	170	U	180	U	1,700	U	160	U	190	UJ
Indeno(1,2,3-cd)pyrene		193-39-5	500	NE	NE	NE		160	U	**2,900**		**1,800**		**7,600**		**40**	**J**	**120**	**J**	**3,200**		**330**		**830**	**J**
Isophorone		78-59-1	NE	NE	NE	NE		180	U	2,900	U	1,900	U	2,700	U	190	U	200	U	1,900	U	180	U	210	UJ
2-Methylnaphthalene		91-57-6	NE	NE	NE	NE		240	U	**95,000**		**50,000**		**280,000**		**1000**		**3,400**		**42,000**		**3,600**		**6,900**	**J**
2-Methylphenol (o-Cresol)		95-48-7	330	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
3,4-Methylphenol (m,p-Cresol)		08394/10644	NE	NE	NE	NE		290	U	4,600	U	3,000	U	4,300	U	310	U	320	U	3,100	U	290	U	340	UJ
Naphthalene		91-20-3	12000	NE	NE	NE		200	U	**110,000**		**80,000**		**450,000**		**2,200**		**6,400**		**69,000**		**4,900**		**8,900**	**J**
2-Nitroaniline		88-74-4	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
3-Nitroaniline		99-09-2	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
4-Nitroaniline		100-01-6	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
Nitrobenzene		98-95-3	NE	NE	NE	40000		180	U	2,900	U	1,900	U	2,700	U	190	U	200	U	1,900	U	180	U	210	UJ
2-Nitrophenol		88-75-5	NE	NE	NE	7000		440	U	7,000	UJ	4,500	U	6,500	U	460	U	470	U	4,700	U	440	U	510	UJ
4-Nitrophenol		100-02-7	NE	NE	NE	7000		280	U	4,500	U	2,900	UJ	4,200	UJ	300	U	310	U	3,000	UJ	280	U	330	UJ
N-Nitrosodiphenylamine (NDFA)		86-30-6	NE	NE	NE	20000		160	U	2,600	U	1,700	U	2,400	U	170	U	180	U	1,700	U	160	U	190	UJ
N-Nitrosodi-n-propylamine (NDPA)		621-64-7	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
Pentachlorophenol		87-86-5	800	800	19000	NE		160	U	2,600	U	1,700	U	2,400	U	170	U	180	U	1,700	U	160	U	190	UJ
Phenanthrene		85-01-8	100000	NE	NE	NE		120	U	**59,000**		**42,000**		**150,000**		**750**		**2,600**		**60,000**		**5,000**		**7,900**	**J**
Phenol		108-95-2	330	30000	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
Pyrene		129-00-0	100000	NE	NE	NE		**58**	**J**	**32,000**		**24,000**		**83,000**		**440**		**1,400**		**34,000**		**3,000**		**5,500**	**J**
1,2,4,5-Tetrachlorobenzene		95-94-3	NE	NE	14000	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
2,3,4,6-Tetrachlorophenol		58-90-2	NE	NE	NE	NE		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
2,4,5-Trichlorophenol		95-95-4	NE	NE	NE	4000		200	U	3,200	U	2,100	U	3,000	U	220	U	220	U	2,200	U	200	U	240	UJ
2,4,6-Trichlorophenol		88-06-2	NE	NE	NE	NE		120	U	1,900	U	1,200	U	1,800	U	130	U	130	U	1,300	U	120	U	140	UJ
Total PAH (16) (ND=0)		TPAH16_ND0	NE	NE	35000	NE		**143**		**327020**		**222580**		**1004000**		**4,955**		**15,909**		**280440**		**25,250**		**46750**	
Total PAH (17) (ND=0)		TPAH17_ND0	NE	NE	35000	NE		**143**		**422020**		**272580**		**1284000**		**5,955**		**19,309**		**322440**		**28,850**		**53650**	
CYANIDES (SW 9012B)	mg/kg																								
Total Cyanide		57-12-5	27	NE	NE	NE		1.2	U	**0.73**	**J**	1.2	UJ	**4.6**	**J**	1.2	UJ	1.2	U	1.2	UJ	1.1	UJ	1.3	UJ
PESTICIDES (SW 8081B)	ug/kg																								
Aldrin		309-00-2	5	140	NE	NE		1.94	R	3	R	1.96	U	2.9	R	1.98	U	2.1	R	2.07	UJ	1.86	R	10.8	UJ
alpha-BHC (Hexachlorocyclohexane)		319-84-6	20	40	NE	NE		0.807	R	1.25	R	0.819	U	1.21	R	0.825	U	0.876	R	0.863	UJ	0.773	R	4.51	UJ
beta-BHC (beta-Hexachlorocyclohexane)		319-85-7	36	600	NE	NE		1.94	R	3	R	1.96	U	2.9	R	1.98	U	2.1	R	2.07	UJ	1.86	R	10.8	UJ
gamma-BHC (gamma-Hexachlorocyclohexane) (Lindane)		58-89-9	100	6000	78	NE		0.807	R	1.25	R	0.819	U	1.21	R	0.825	U	0.876	R	0.863	UJ	0.773	R	4.51	UJ

Table 5D. Creek Deeper Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

							Task Description	Creek Deeper Sediment Boring Sample Analytical Results																									
							Sample Name	SED-BGL-1/12-14		SED-LSC-1/11-15		SED-LSC-2/14-16		SED-LSC-3/13-16		SED-LSC-4/14-16		SED-LSC-5/14-16		SED-LSC-6/12-19.5		SED-LSC-7/12-18		SED-LSC-8/6.8-15								
							Start Depth	562.2		563.1		560.1		561.1		560.0		560.0		561.2		561.8		566.6								
							End Depth	560.2		559.1		558.1		558.1		550.0		550.0		553.7		553.8		558.4								
							Depth Unit	Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft								
							Sample Depth Interval (ft) below Top of Sediment	1.0 to 3.0		4.5 to 8.5		8.0 to 10.0		5.2 to 8.2		4.7 to 6.7		5.2 to 7.2		3.5 to 11.0		4.0 to 12.0		0 to 8.2								
							Sample Date	11/10/2021		11/9/2021		11/9/2021		11/9/2021		11/10/2021		11/10/2021		11/3/2021		11/3/2021		11/3/2021								
Analyte	**Units**	**CAS No. or Identifier**	**375-6.8(a) Unrestricted SCO**	**375-6.8(b) Protection of Ecological Resources SCO**	**Freshwater Sediment Guidance Values Class B**	**NY CP-51 RESTRICTED USE ECO SSCO**																											
delta-BHC (delta-Hexachlorocyclohexane)		319-86-8	40	40	NE	NE		1.94	R	3	R	1.96	U	2.9	R	1.98	U	2.1	R	2.07	UJ	1.86	R	10.8	UJ							
Chlordane (Technical)		12789-03-6	NE	NE	NE	NE		16.1	R	25	R	16.4	U	24.2	R	16.5	U	17.5	R	17.2	UJ	15.5	R	90.1	UJ							
alpha-chlordane		5103-71-9	94	1300	NE	NE		2.42	R	3.74	R	2.46	U	3.63	R	2.48	U	2.63	R	2.59	UJ	2.32	R	13.5	UJ							
gamma-Chlordane		5103-74-2	NE	NE	NE	NE		2.42	R	3.74	R	2.46	U	3.63	R	2.48	U	2.63	R	2.59	UJ	2.32	R	13.5	UJ							
4,4'-DDT (p,p'-DDT)		50-29-3	3.3	3.3	48000	NE		3.63	R	5.62	R	3.68	U	5.44	R	3.71	U	3.94	R	**6.51**	**J**	3.48	R	20.3	UJ							
4,4'-DDE (p,p'-DDE)		72-55-9	3.3	3.3	NE	NE		1.94	R	3	R	1.96	U	2.9	R	1.98	U	2.1	R	2.07	UJ	1.86	R	10.8	UJ							
4,4'-DDD (p,p-DDD)		72-54-8	3.3	3.3	NE	NE		1.94	R	3	R	1.96	U	**5.96**	**J**	1.98	U	2.1	R	**8.81**	**J**	1.86	R	10.8	UJ							
Dieldrin		60-57-1	5	6	780	NE		1.21	R	1.87	R	1.23	U	1.81	R	1.24	U	1.31	R	1.29	UJ	1.16	R	6.76	UJ							
alpha-Endosulfan (I)		959-98-8	2400	NE	NE	NE		1.94	R	3	R	1.96	U	2.9	R	1.98	U	2.1	R	2.07	UJ	1.86	R	10.8	UJ							
beta-Endosulfan (II)		33213-65-9	2400	NE	NE	NE		1.94	R	3	R	1.96	U	2.9	R	1.98	U	2.1	R	2.07	UJ	1.86	R	10.8	UJ							
Endosulfan sulfate		1031-07-8	2400	NE	NE	NE		0.807	R	1.25	R	0.819	U	1.21	R	0.825	U	0.876	R	0.863	UJ	0.773	R	4.51	UJ							
Endrin		72-20-8	14	14	220	NE		0.807	R	1.25	R	0.819	U	1.21	R	0.825	U	0.876	R	0.863	UJ	0.773	R	4.51	UJ							
Endrin aldehyde		7421-93-4	NE	NE	NE	NE		2.42	R	3.74	R	2.46	U	3.63	R	2.48	UJ	2.63	R	2.59	UJ	2.32	R	13.5	UJ							
Endrin ketone		53494-70-5	NE	NE	NE	NE		1.94	R	3	R	1.96	U	2.9	R	1.98	U	2.1	R	2.07	UJ	1.86	R	10.8	UJ							
Heptachlor		76-44-8	42	140	10000	NE		0.969	R	1.5	R	0.982	U	1.45	R	0.99	U	1.05	R	1.04	UJ	0.928	R	5.41	UJ							
Heptachlor epoxide		1024-57-3	NE	NE	2100	NE		3.63	R	5.62	R	3.68	U	5.44	R	3.71	U	3.94	R	3.88	UJ	3.48	R	20.3	UJ							
Methoxychlor		72-43-5	NE	NE	NE	1200		3.63	R	5.62	R	3.68	U	5.44	R	3.71	U	3.94	R	3.88	UJ	3.48	R	20.3	UJ							
Toxaphene		8001-35-2	NE	NE	250	NE		36.3	R	56.2	R	36.8	U	54.4	R	37.1	U	39.4	R	38.8	UJ	34.8	R	203	UJ							
HERBICIDES (SW 8151A)	ug/kg																															
2,4-D (2,4-Dichlorophenoxyacetic acid)		94-75-7	NE	NE	NE	NE		200	U	323	U	209	U	302	U	216	U	217	U	215	U	203	UJ	234	UJ							
2,4,5-T (2,4,5-Trichlorophenoxyacetic acid)		93-76-5	NE	NE	NE	NE		200	U	323	U	209	U	302	U	216	U	217	U	215	U	203	UJ	234	UJ							
2,4,5-TP (Silvex)		93-72-1	3800	NE	NE	NE		200	U	323	U	209	U	302	U	216	U	217	U	215	U	203	UJ	234	UJ							
PCBs (SW 8082A)	ug/kg																															
Aroclor 1016		12674-11-2	NE	NE	NE	NE		38.6	U	61.8	UJ	42.3	UJ	59.2	UJ	43.4	U	44.2	U	42.8	UJ	39	UJ	46.2	UJ							
Aroclor 1221		11104-28-2	NE	NE	NE	NE		38.6	U	61.8	UJ	42.3	UJ	59.2	UJ	43.4	U	44.2	U	42.8	UJ	39	UJ	46.2	UJ							
Aroclor 1232		11141-16-5	NE	NE	NE	NE		38.6	U	61.8	UJ	42.3	UJ	59.2	UJ	43.4	U	44.2	U	42.8	UJ	39	UJ	46.2	UJ							
Aroclor 1242		53469-21-9	NE	NE	NE	NE		38.6	U	61.8	UJ	42.3	UJ	59.2	UJ	43.4	U	44.2	U	42.8	UJ	39	UJ	**362**	**J**							
Aroclor 1248		12672-29-6	NE	NE	NE	NE		38.6	U	61.8	UJ	42.3	UJ	59.2	UJ	43.4	U	44.2	U	42.8	UJ	39	UJ	46.2	UJ							
Aroclor 1254		11097-69-1	NE	NE	NE	NE		38.6	U	61.8	UJ	42.3	UJ	**780**	**J**	43.4	U	44.2	U	**77.5**	**J**	**16.6**	**J**	46.2	UJ							
Aroclor 1260		11096-82-5	NE	NE	NE	NE		38.6	U	61.8	UJ	42.3	UJ	**206**	**J**	43.4	U	44.2	U	42.8	UJ	39	UJ	46.2	UJ							
Aroclor 1262		37324-23-5	NE	NE	NE	NE		38.6	U	61.8	UJ	42.3	UJ	59.2	UJ	43.4	U	44.2	U	42.8	UJ	39	UJ	46.2	UJ							
Aroclor 1268		11100-14-4	NE	NE	NE	NE		38.6	U	61.8	UJ	42.3	UJ	59.2	UJ	43.4	U	44.2	U	42.8	UJ	39	UJ	46.2	UJ							
Total PCBs (Lab calculated)		1336-36-3	100	1000	1000	NE		38.6	U	61.8	UJ	42.3	UJ	**986**	**J**	43.4	U	44.2	U	**77.5**	**J**	**16.6**	**J**	**362**	**J**							
METALS (SW 6010D)	mg/kg																															
Aluminum		7429-90-5	NE	NE	NE	10000		**8,840**		**10,800**		**6,870**		**6,850**		**8,800**		**8,190**		**15,800**	**J**	**2,410**		**4,620**								
Antimony		7440-36-0	NE	NE	NE	12		4.75	U	7.37	U	4.88	U	7.08	U	5.15	U	5.14	U	5.08	U	4.63	U	5.6	U							
Arsenic		7440-38-2	13	13	33	NE		**3.15**	**J**	**9.49**		**6.85**		**11.4**		**1.77**	**J**	**2.14**	**J**	**3.29**	**J**	**3.47**		**4.69**								
Barium		7440-39-3	350	433	NE	NE		**95**		**159**		**74.4**		**134**		**102**		**88.3**		**145**		**32.9**		**57.5**								
Beryllium		7440-41-7	7.2	10	NE	NE		**0.475**		0.737	U	0.488	U	0.708	U	**0.36**	**J**	0.514	U	**2.18**		**0.111**	**J**	**0.324**	**J**							
Cadmium		7440-43-9	2.5	4	5	NE		**0.104**	**J**	**1.81**	**J**	**0.556**	**J**	**3.66**	**J**	**0.69**	**J**	**0.144**	**J**	**0.386**	**J**	**0.528**	**J**	**0.996**	**J**							
Calcium		7440-70-2	NE	NE	NE	10000		**97,100**		**37,800**		**80,600**		**25,800**		**81,500**		**66,800**		**141,000**		**121,000**		**75,800**								
Chromium		7440-47-3	30	41	110	NE		**14.5**		**20.3**		**11.6**		**35**		**14.5**		**14.1**		**8.76**		**5.81**		**13.9**								
Cobalt		7440-48-4	NE	NE	NE	20		**9.83**		**9.39**		**7.47**		**13.5**		**8.17**		**8.1**		**2**	**J**	**2.52**		**4.74**								
Copper		7440-50-8	50	50	150	NE		**17.7**		**133**		**14.4**		**235**		**21.4**		**65**		**73.9**		**29.8**		**90.6**								

Table 5D. Creek Deeper Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

							Task Description	Creek Deeper Sediment Boring Sample Analytical Results																							
							Sample Name	SED-BGL-1/12-14		SED-LSC-1/11-15		SED-LSC-2/14-16		SED-LSC-3/13-16		SED-LSC-4/14-16		SED-LSC-5/14-16		SED-LSC-6/12-19.5		SED-LSC-7/12-18		SED-LSC-8/6.8-15						
							Start Depth	562.2		563.1		560.1		561.1		560.0		560.0		561.2		561.8		566.6						
							End Depth	560.2		559.1		558.1		558.1		550.0		550.0		553.7		553.8		558.4						
							Depth Unit	Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft		Elevation ft						
							Sample Depth Interval (ft) below Top of Sediment	1.0 to 3.0		4.5 to 8.5		8.0 to 10.0		5.2 to 8.2		4.7 to 6.7		5.2 to 7.2		3.5 to 11.0		4.0 to 12.0		0 to 8.2						
							Sample Date	11/10/2021		11/9/2021		11/9/2021		11/9/2021		11/10/2021		11/10/2021		11/3/2021		11/3/2021		11/3/2021						
Analyte	**Units**	**CAS No. or Identifier**	**375-6.8(a) Unrestricted SCO**	**375-6.8(b) Protection of Ecological Resources SCO**	**Freshwater Sediment Guidance Values Class B**	**NY CP-51 RESTRICTED USE ECO SSCO**																									
Iron		7439-89-6	NE	NE	NE	NE		**19,500**		**26,400**		**13,600**		**37,800**		**16,900**		**17,600**		**6,760**		**7,540**	**J**	**17,100**	**J**					
Lead		7439-92-1	63	63	130	NE		**17**		**291**		**13.8**		**392**		**15.5**		**22**	**J**	**58.4**		**34**		**98.8**						
Magnesium		7439-95-4	NE	NE	NE	NE		**21,000**		**8,830**		**19,800**		**5,840**		**18,300**		**21,000**		**21,800**	**J**	**39,000**		**21,700**						
Manganese		7439-96-5	1600	1600	NE	NE		**467**		**247**		**373**		**404**		**451**		**444**		**2,100**		**383**		**428**						
Mercury (SW 7471B)		7439-97-6	0.18	0.1800	1	NE		0.089	U	**1.11**		0.081	U	**0.669**		0.085	U	0.092	U	**0.243**		0.079	U	**0.207**						
Nickel		7440-02-0	30	30	49	NE		**18.3**		**22.2**		**15.8**		**31.6**		**17.7**		**17.4**		**4.58**		**5.22**		**9.9**						
Potassium		7440-09-7	NE	NE	NE	NE		**1,750**		**991**		**1,200**		**746**		**1,570**		**1,710**		**1,120**		**384**		**644**						
Selenium		7782-49-2	3.9	3.9	NE	NE		**0.37**	**J**	2.95	U	1.95	U	2.83	U	2.06	U	**0.854**	**J**	**2.21**		**0.287**	**J**	**0.571**	**J**					
Silver		7440-22-4	2	2	2.2	NE		0.95	U	**0.752**	**J**	0.976	U	**1.09**	**J**	1.03	U	1.03	U	1.02	U	0.927	U	1.12	U					
Sodium		7440-23-5	NE	NE	NE	NE		**663**	**J**	**339**		**238**		**332**	**J**	**254**		**300**	**J**	**543**		**368**		**671**						
Thallium		7440-28-0	NE	NE	NE	5		1.9	U	2.95	UJ	1.95	U	2.83	UJ	2.06	U	2.06	U	2.03	U	1.85	U	2.24	U					
Vanadium		7440-62-2	NE	NE	NE	39		**20.6**		**23.8**		**15.6**		**24.5**		**19.7**		**18.8**		**3.9**		**8.08**		**16**						
Zinc		7440-66-6	109	109	460	NE		**62.6**		**547**		**57.2**		**598**		**57.8**		**78.2**		**91.2**		**86.1**		**194**						
GENERAL CHEMISTRY (SM 2540G)	%																													
Percent Solids		SOLIDS	NE	NE	NE	NE		**81.6**		**51.4**		**77.8**		**54.9**		**75.8**		**74.9**		**76.6**		**81.2**		**69.7**						
TOTAL ORGANIC CARBON (SW 9060A)	%																													
Total Organic Carbon		TOC	NE	NE	NE	NE		0.01	U	**8**		**3.57**		**3.86**		**3.83**		**3.7**		**3.53**		**3.77**		**4.17**						

Table 5D. Creek Deeper Sediment Boring Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

Notes:
Sample ID contains sample depth below the measuring point on barge; sample elevation interval provided for sample depth
mg/kg = milligrams/kilogram or parts per million (ppm)
% = Percent
ug/kg = micrograms per kilogram

BTEX = Benzene, Toluene, Ethylbenzene, and Xylenes
PAH = Polycyclic Aromatic Hydrocarbon
PCB = Polychlorinated Biphenyl
SVOC = Semi-Volatile Organic Compound
VOC = Volatile Organic Compound

CAS No. = Chemical Abstracts Service Number
ND = Not Detected
NE = Not Established

Bolding indicates a detected result concentration
Shading and bolding indicates that the detected concentration is above the Guidance it was compared to
6 NYCRR = New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York

Comparison of detected results are performed against one or more of the following NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCO)s: Unrestricted Use, Restricted-Residential, Protection of Ecological Resources

(1) Source: NYSDEC CP-51 Soil Cleanup Guidance as presented in Tables 1 through 3 dated October 2010 applying to the Inactive Hazardous Waste Disposal Site Remedial Program

Validation Qualifiers:
J = The result is an estimated value.
JN = The analyte is presumptively present at an approximated quantity.
U = The result was not detected above the reporting limit.
UJ = The results was not detected at or above the reporting limit shown and the reporting limit is estimated.
R = The results were rejected by laboratory (see **1,4-Dioxane, Pesticides Group**)

1,4-Dioxane - Rejected Data
Due to the extremely low calibration response factor for 1,4-dioxane under Method 8260C, this compound is typically rejected during validation as it does not meet the minimal criteria set for accurate quantitation. Non-detect results for 1,4-dioxane are rejected as the presence or absence of the compound cannot be verified. Positive results are estimated due to the high variability inherent in the quantitation when using an extremely low response factor.

Pesticides (SW 8081B) - Rejected Data
The pesticide nondetect results in sample SED-BGL-1/12-14, SED-LSC-1/11-15, SED-LSC-3/13-16, SED-LSC-5/14-16, and SED-LSC-7/12-18 were rejected (R) due to surrogate recovery less than 10%. These results should not be used for decision-making purposes.

Table 5E. Vault Debris Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

		Sample Name	BSA-VAULT-DEBRIS	
		Sample Date	11/18/2021	
Analyte	**Units**	**CAS No.**		
VOCs (SW 8260C)	ug/kg			
Acetone		67-64-1	810	U
Benzene		71-43-2	**1,800**	
Bromochloromethane		74-97-5	160	U
Bromodichloromethane		75-27-4	41	U
Bromoform		75-25-2	320	U
Bromomethane		74-83-9	160	U
Carbon disulfide		75-15-0	**430**	**J**
Carbon tetrachloride		56-23-5	81	U
Chlorobenzene		108-90-7	41	U
Chloroethane		75-00-3	**160**	
Chloroform (Trichloromethane)		67-66-3	120	U
Chloromethane		74-87-3	320	U
Cyclohexane		110-82-7	**63**	**J**
1,2-Dibromo-3-chloropropane		96-12-8	240	U
Dibromochloromethane		124-48-1	81	U
1,2-Dibromoethane (EDB)		106-93-4	81	U
1,2-Dichlorobenzene (o-DCB)		95-50-1	160	U
1,3-Dichlorobenzene (m-DCB)		541-73-1	160	U
1,4-Dichlorobenzene (p-DCB)		106-46-7	160	U
Dichlorodifluoromethane (Freon 12)		75-71-8	810	U
1,1-Dichloroethane		75-34-3	**280**	
1,2-Dichloroethane		107-06-2	81	U
1,1-Dichloroethene		75-35-4	**23**	**J**
cis-1,2-Dichloroethene		156-59-2	**2,200**	
trans-1,2-Dichloroethene		156-60-5	**14**	**J**
1,2-Dichloropropane		78-87-5	81	U
cis-1,3-Dichloropropene		10061-01-5	41	U
trans-1,3-Dichloropropene		10061-02-6	81	U
1,4-Dioxane		123-91-1		R
Ethylbenzene		100-41-4	**62,000**	
2-Hexanone		591-78-6	810	U
Isopropylbenzene		98-82-8	**5,900**	
Methyl acetate		79-20-9	**470**	
Methyl ethyl ketone (2-Butanone)		78-93-3	810	U
Methyl tert-butyl ether (MTBE)		1634-04-4	160	U
4-Methyl-2-pentanone (MIBK)		108-10-1	810	U
Methylcyclohexane		108-87-2	**240**	**J**
Methylene chloride		75-09-2	410	U
Styrene		100-42-5	**260**	
1,1,2,2-Tetrachloroethane		79-34-5	41	U
Tetrachloroethene (PCE)		127-18-4	41	U
Toluene		108-88-3	**2,900**	
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)		76-13-1	320	U
1,2,3-Trichlorobenzene		87-61-6	160	U
1,2,4-Trichlorobenzene		120-82-1	160	U

Table 5E. Vault Debris Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

		Sample Name	BSA-VAULT-DEBRIS	
		Sample Date	11/18/2021	
Analyte	**Units**	**CAS No.**		
1,1,1-Trichloroethane (TCA)		71-55-6	**440**	
1,1,2-Trichloroethane		79-00-5	81	U
Trichloroethene (TCE)		79-01-6	**61**	
Trichlorofluoromethane (Freon 11)		75-69-4	320	U
Vinyl chloride		75-01-4	**450**	
o-Xylene		95-47-6	**16,000**	
m/p-Xylene		179601-23-1	**17,000**	
SVOCs (SW 8270D)	ug/kg			
Acenaphthene		83-32-9	**58**	**J**
Acenaphthylene		208-96-8	**53**	**J**
Acetophenone		98-86-2	240	U
Anthracene		120-12-7	**140**	
Atrazine		1912-24-9	190	U
Benzaldehyde		100-52-7	320	U
Benzo(a)anthracene		56-55-3	**400**	
Benzo(b)fluoranthene		205-99-2	**480**	
Benzo(k)fluoranthene		207-08-9	**130**	**J**
Benzo(g,h,i)perylene		191-24-2	**220**	
Benzo(a)pyrene		50-32-8	**330**	
Biphenyl (1,1-Biphenyl)		92-52-4	550	U
Bis(2-chloroethoxy)methane		111-91-1	260	U
Bis(2-chloroethyl)ether		111-44-4	220	U
2,2-oxybis(1-Chloropropane)		108-60-1	290	U
Bis(2-ethylhexyl)phthalate		117-81-7	240	U
4-Bromophenyl phenyl ether		101-55-3	240	U
Butyl benzyl phthalate		85-68-7	**160**	**J**
Caprolactam		105-60-2	240	U
Carbazole		86-74-8	**46**	**J**
4-Chloro-3-methylphenol		59-50-7	240	U
4-Chloroaniline		106-47-8	240	U
2-Chloronaphthalene		91-58-7	240	U
2-Chlorophenol		95-57-8	240	U
4-Chlorophenyl phenyl ether		7005-72-3	240	U
Chrysene		218-01-9	**410**	
Dibenz(a,h)anthracene		53-70-3	**61**	**J**
Dibenzofuran		132-64-9	**33**	**J**
3,3-Dichlorobenzidine		91-94-1	240	U
2,4-Dichlorophenol		120-83-2	220	U
Diethyl phthalate		84-66-2	240	U
Dimethyl phthalate		131-11-3	240	U
2,4-Dimethylphenol		105-67-9	240	U
Di-n-butyl phthalate		84-74-2	240	U
4,6-Dinitro-2-methylphenol		534-52-1	620	U
2,4-Dinitrophenol		51-28-5	1,200	U
2,4-Dinitrotoluene		121-14-2	240	U
2,6-Dinitrotoluene		606-20-2	240	U

Table 5E. Vault Debris Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

		Sample Name	BSA-VAULT-DEBRIS	
		Sample Date	11/18/2021	
Analyte	**Units**	**CAS No.**		
Di-n-octyl phthalate		117-84-0	240	U
Fluoranthene		206-44-0	**780**	
Fluorene		86-73-7	**75**	**J**
Hexachlorobenzene		118-74-1	140	U
1,3-Hexachlorobutadiene (C-46)		87-68-3	240	U
Hexachlorocyclopentadiene		77-47-4	680	U
Hexachloroethane		67-72-1	190	U
Indeno(1,2,3-cd)pyrene		193-39-5	**250**	
Isophorone		78-59-1	220	U
2-Methylnaphthalene		91-57-6	290	U
2-Methylnaphthalene		91-57-6	290	U
2-Methylphenol (o-Cresol)		95-48-7	240	U
3,4-Methylphenol (m,p-Cresol)		108394/106445	340	U
Naphthalene		91-20-3	**30**	**J**
2-Nitroaniline		88-74-4	240	U
3-Nitroaniline		99-09-2	240	U
4-Nitroaniline		100-01-6	240	U
Nitrobenzene		98-95-3	220	U
2-Nitrophenol		88-75-5	520	U
4-Nitrophenol		100-02-7	340	U
N-Nitrosodiphenylamine (NDFA)		86-30-6	190	U
N-Nitrosodi-n-propylamine (NDPA)		621-64-7	240	U
Pentachlorophenol		87-86-5	190	U
Phenanthrene		85-01-8	**450**	
Phenol		108-95-2	240	U
Pyrene		129-00-0	**660**	
1,2,4,5-Tetrachlorobenzene		95-94-3	240	U
2,3,4,6-Tetrachlorophenol		58-90-2	240	U
2,4,5-Trichlorophenol		95-95-4	240	U
2,4,6-Trichlorophenol		88-06-2	140	U
PCB Aroclors (8082A)	ug/kg			
Aroclor 1016		12674-11-2	45.7	U
Aroclor 1221		11104-28-2	45.7	U
Aroclor 1232		11141-16-5	45.7	U
Aroclor 1242		53469-21-9	45.7	U
Aroclor 1248		12672-29-6	45.7	U
Aroclor 1254		11097-69-1	**102**	**J**
Aroclor 1260		11096-82-5	**33.3**	**J**
Aroclor 1262		37324-23-5	45.7	U
Aroclor 1268		11100-14-4	45.7	U
Total PCBs (Lab calculated)		1336-36-3	**135**	**J**
PESTICIDES (SW 8081B)	ug/kg			
Aldrin		309-00-2	21.8	U
alpha-BHC (Hexachlorocyclohexane)		319-84-6	9.09	U
beta-BHC (beta-Hexachlorocyclohexane)		319-85-7	21.8	U
gamma-BHC (gamma-Hexachlorocyclohexane) (Lindane)		58-89-9	9.09	U

Table 5E. Vault Debris Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

		Sample Name	BSA-VAULT-DEBRIS	
		Sample Date	11/18/2021	
Analyte	**Units**	**CAS No.**		
delta-BHC (delta-Hexachlorocyclohexane)		319-86-8	21.8	U
Chlordane (Technical)		12789-03-6	182	U
alpha-chlordane		5103-71-9	27.3	U
gamma-Chlordane		5103-74-2	27.3	U
4,4'-DDT (p,p'-DDT)		50-29-3	40.9	U
4,4'-DDE (p,p'-DDE)		72-55-9	21.8	U
4,4'-DDD (p,p-DDD)		72-54-8	**13.8**	**J**
Dieldrin		60-57-1	13.6	U
alpha-Endosulfan (I)		959-98-8	21.8	U
beta-Endosulfan (II)		33213-65-9	21.8	U
Endosulfan sulfate		1031-07-8	9.09	U
Endrin		72-20-8	9.09	U
Endrin aldehyde		7421-93-4	27.3	U
Endrin ketone		53494-70-5	21.8	U
Heptachlor		76-44-8	10.9	U
Heptachlor epoxide		1024-57-3	40.9	U
Methoxychlor		72-43-5	40.9	U
Toxaphene		8001-35-2	409	U
HERBICIDES (SW 8151A)	ug/kg			
2,4-D (2,4-Dichlorophenoxyacetic acid)		94-75-7	237	U
2,4,5-T (2,4,5-Trichlorophenoxyacetic acid)		93-76-5	237	U
2,4,5-TP (Silvex)		93-72-1	237	U
METALS (SW 6010D)	mg/kg			
Aluminum		7429-90-5	**3,370**	
Antimony		7440-36-0	**3.82**	**J**
Arsenic		7440-38-2	**5.35**	
Barium		7440-39-3	**64.8**	
Beryllium		7440-41-7	**0.305**	**J**
Cadmium		7440-43-9	**1.72**	
Calcium		7440-70-2	**98,000**	
Chromium		7440-47-3	**81.6**	
Cobalt		7440-48-4	**4.71**	
Copper		7440-50-8	**131**	
Iron		7439-89-6	**29,200**	
Lead		7439-92-1	**833**	
Magnesium		7439-95-4	**21,300**	
Manganese		7439-96-5	**550**	
Mercury		7439-97-6	0.092	U
Nickel		7440-02-0	**19.9**	
Potassium		7440-09-7	**342**	
Selenium		7782-49-2	**0.588**	**J**
Silver		7440-22-4	1.13	U
Sodium		7440-23-5	**271**	
Thallium		7440-28-0	**0.768**	**J**
Vanadium		7440-62-2	**12**	
Zinc		7440-66-6	**600**	

Table 5E. Vault Debris Sample Analytical Results
Site Characterization Report
Former Buffalo Gaslight Iroquois Gas Corp. Site
Buffalo, New York

		Sample Name	BSA-VAULT-DEBRIS	
		Sample Date	11/18/2021	
Analyte	**Units**	**CAS No.**		
CYANIDES (SW 9012B)	mg/kg			
Total Cyanide		57-12-5	**4.9**	
VPH/EPH				
C5-C8 Aliphatics (MAVPH)	mg/kg	ALIPH5-8	8.46	U
C5-C8 Aliphatics, Adjusted (MAVPH)	mg/kg	ALIPH5-8ADJ	8.46	U
C9-C12 Aliphatics (MAVPH)	mg/kg	ALIPH9-12	**11.9**	
C9-C12 Aliphatics, Adjusted (MAVPH)	mg/kg	ALIPH9-12ADJ	**9.86**	
C9-C10 Aromatics (MAVPH)	mg/kg	AROM9-10	8.46	U
Total EPH Fractions (NJ EPH R3)	mg/kg	TEPH	**6,440**	
GENERAL CHEMISTRY				
Ammonia (SM 4500-NH3 BH)	mg/kg	7664-41-7	**18**	
Percent Solids (SM 2540G)	%	SOLIDS	**69.5**	
Sulfide (SW 9030B)	mg/kg	18496-25-8	**2,400**	
TOTAL ORGANIC CARBON (SW 9060A)				
Total Organic Carbon	%	TOC	**9.73**	

Notes:

mg/kg = milligrams/kilogram or parts per million (ppm)
% = Percent
ug/kg = micrograms per kilogram

PAH = Polycyclic Aromatic Hydrocarbon
PCB = Polychlorinated Biphenyl
SVOC = Semi-Volatile Organic Compound
VOC = Volatile Organic Compound

CAS No. = Chemical Abstracts Service Number

Bolding indicates a detected result concentration

Validation Qualifiers:

J = The result is an estimated value.
R = The result is rejected.
U = The result was not detected above the reporting limit.

1,4-Dioxane - Rejected Data:

Due to the extremely low calibration response factor for 1,4-dioxane under Method 8260C, this compound is typically rejected during validation as it does not meet the minimal criteria set for accurate quantitation. Non-detect results for 1,4-dioxane are rejected as the presence or absence of the compound cannot be verified. Positive results are estimated due to the high variability inherent in the quantitation when using an extremely low response factor.

Figures

P-Site Location

Site Location Map

0 1,000 2,000 Feet

Former Buffalo Gaslight Iroquois Gas Corp. Site	GEI Consultants	SITE LOCATION
Buffalo, New York	Project 2101620	June 2022 Figure 1

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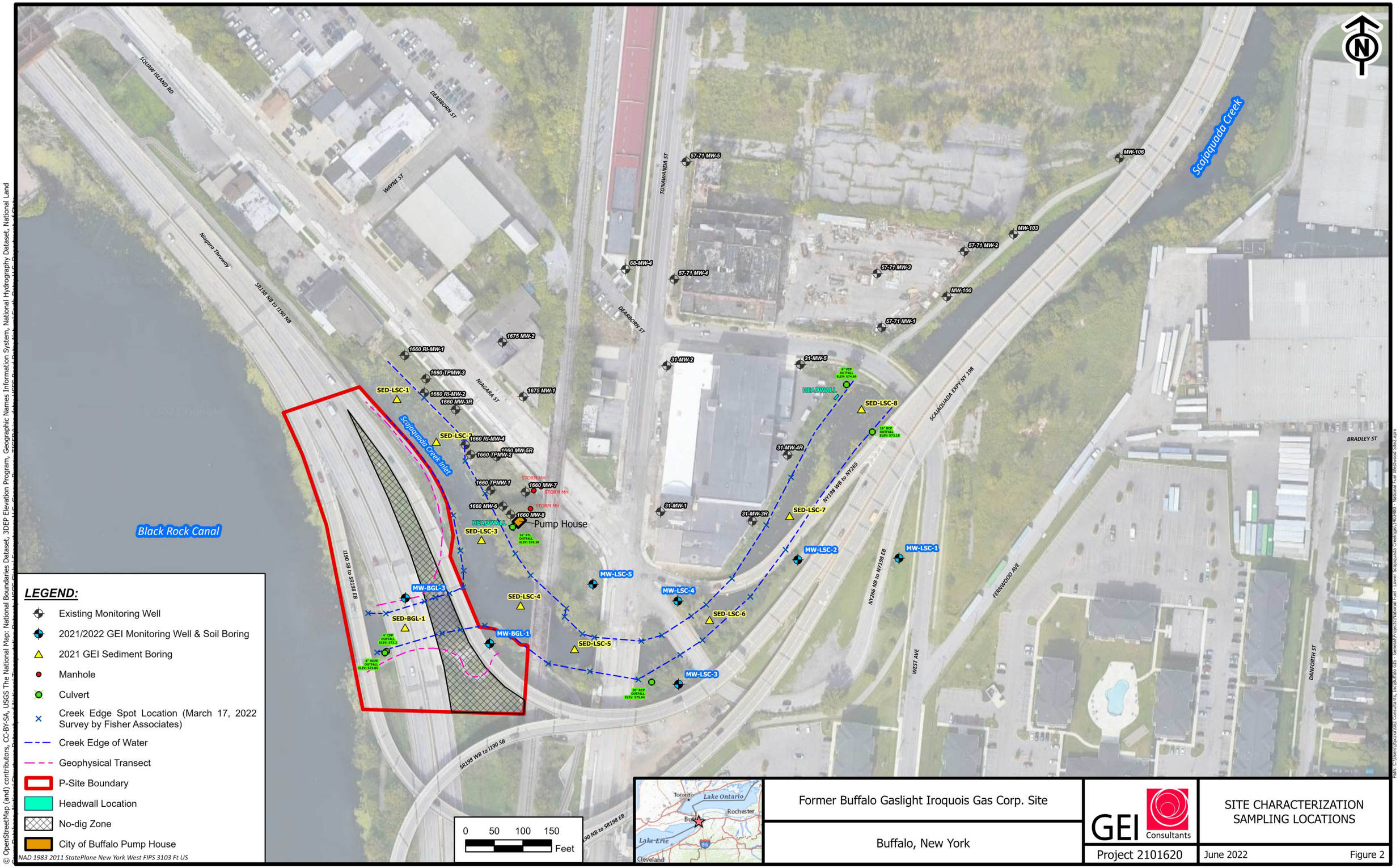

LEGEND:

- Existing Monitoring Well
- 2021/2022 GEI Monitoring Well & Soil Boring
- 2021 GEI Sediment Boring
- Manhole
- Culvert
- Creek Edge Spot Location (March 17, 2022 Survey by Fisher Associates)
- Creek Edge of Water
- Geophysical Transect
- P-Site Boundary
- Headwall Location
- No-dig Zone
- City of Buffalo Pump House

NAD 1983 2011 StatePlane New York West FIPS 3103 Ft US

Former Buffalo Gaslight Iroquois Gas Corp. Site

Buffalo, New York

GEI Consultants

Project 2101620

SITE CHARACTERIZATION SAMPLING LOCATIONS

June 2022

Figure 2

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Path: C:\Users\cjoyce\Desktop\GEI Consultants, Inc\Buffalo GIS - General\Projects\National Fuel - Lower Scajaquada Creek\gis\1403480 - National Fuel Westwood SI02.aprx

189-205 Tonawanda St

166 Dart St

68 Tonawanda St

75 Tonawanda St

Dart & Bradley St (Riparian MGP Site)

57-71 Tonawanda St

Dart & Bradley St (Terrestrial MGP Site)

1675 Niagara St

1660 Niagara St

31 Tonawanda St

1451 West Ave

1654 Niagara St

P-Site #915351

Pump House

1626 Niagara St

Scajaquada Creek

Scajaquada Creek Slip

Black Rock Canal

AMHERST ST

EAST ST

DEARBORN ST

BRIDGE ST

PARISH ST

WATTS

TONAWANDA ST

SCAJAQUADA EXPY NY 198

DART ST

LETCHWORTH ST

GRANT ST

NIAGARA ST

WAYNE ST

SQUAW ISLAND RD

Niagara Thruway

Damn St to I190 SB

I190 NB to NY266

SR198 NB to I190 NB

I190 SB to SR198 EB

SR198 WB to I190 SB

NY198 WB to NY266

NY266 NB to NY198 EB

WEST AVE

FERNWOOD AVE

DANFORTH ST

BRADLEY ST

HAWLEY ST

Water Plant - Black Rock Harbor

FOREST

0 125 250 Feet

NAD 1983 2011 StatePlane New York West FIPS 3103 Ft US

LEGEND:

- P-Site Boundary
- Possible Area of Environmental Concern
- City of Buffalo Pump House

Former Buffalo Gaslight Iroquois Gas Corp. Site

Buffalo, New York

GEI Consultants

Project 2101620

PROPERTIES WITH RECOGNIZED ENVIRONMENTAL CONDITIONS (RECS)

June 2022 Figure 3

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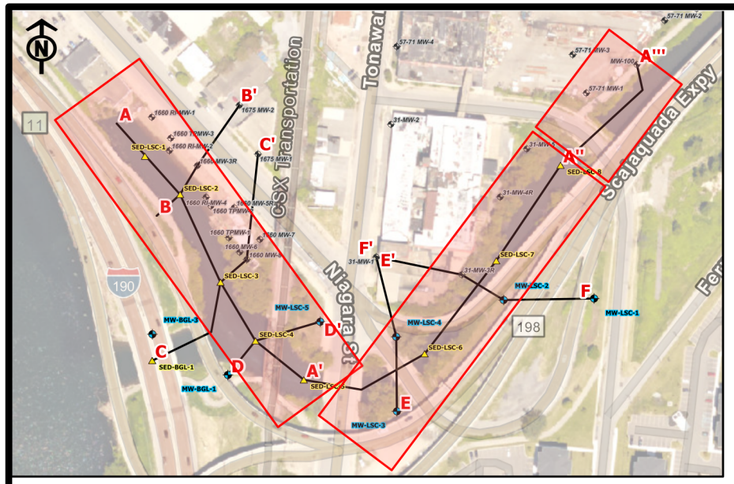


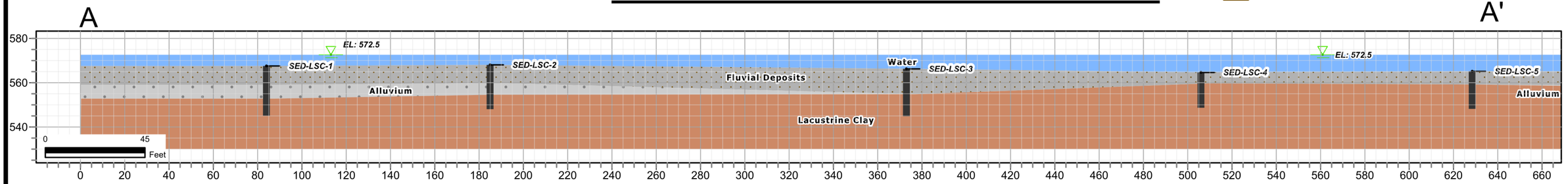
LEGEND:

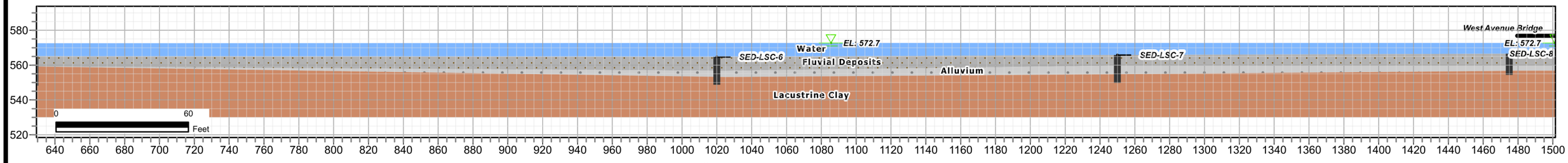
- Groundwater / Surface Elevation (March 2022)
- Monitoring Well
- Sediment Boring
- Well Screen Length
- Perforated Sheet Pile
- Sheet Pile Barrier
- South Vault NAPL Collector

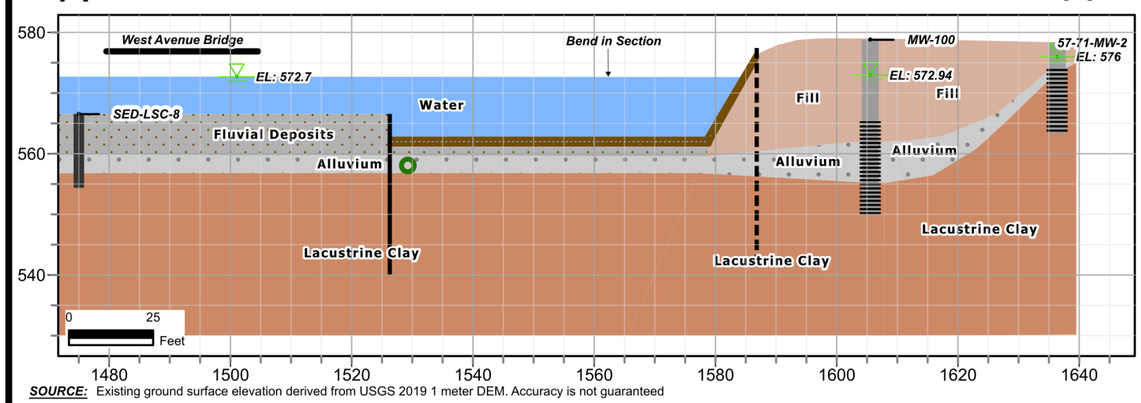
Material Types

- Water
- Fill
- Fluvial Deposits
- Alluvium
- Lacustrine Clay
- Sediment Cap

GEOLOGICAL CROSS-SECTIONS A to A'''







SOURCE: Existing ground surface elevation derived from USGS 2019 1 meter DEM. Accuracy is not guaranteed

Former Buffalo Gaslight Iroquois Gas Corp. Site	GEI Consultants	A to A''' GEOLOGIC CROSSSECTIONS	
Buffalo, New York	Project 2101620	June 2022	Figure 4A

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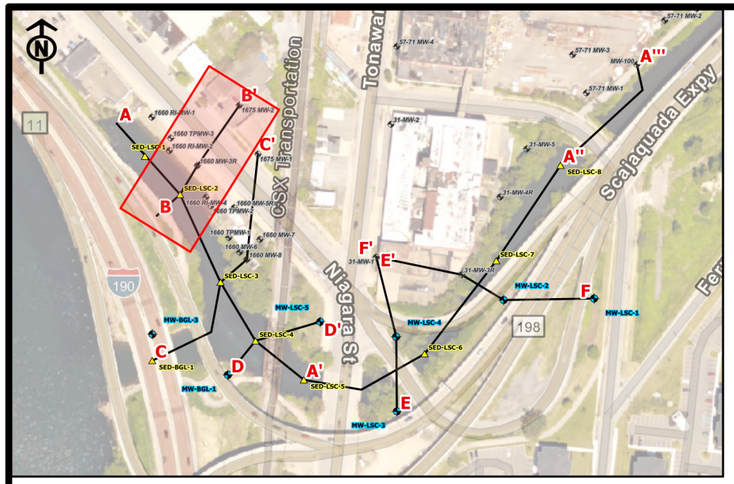
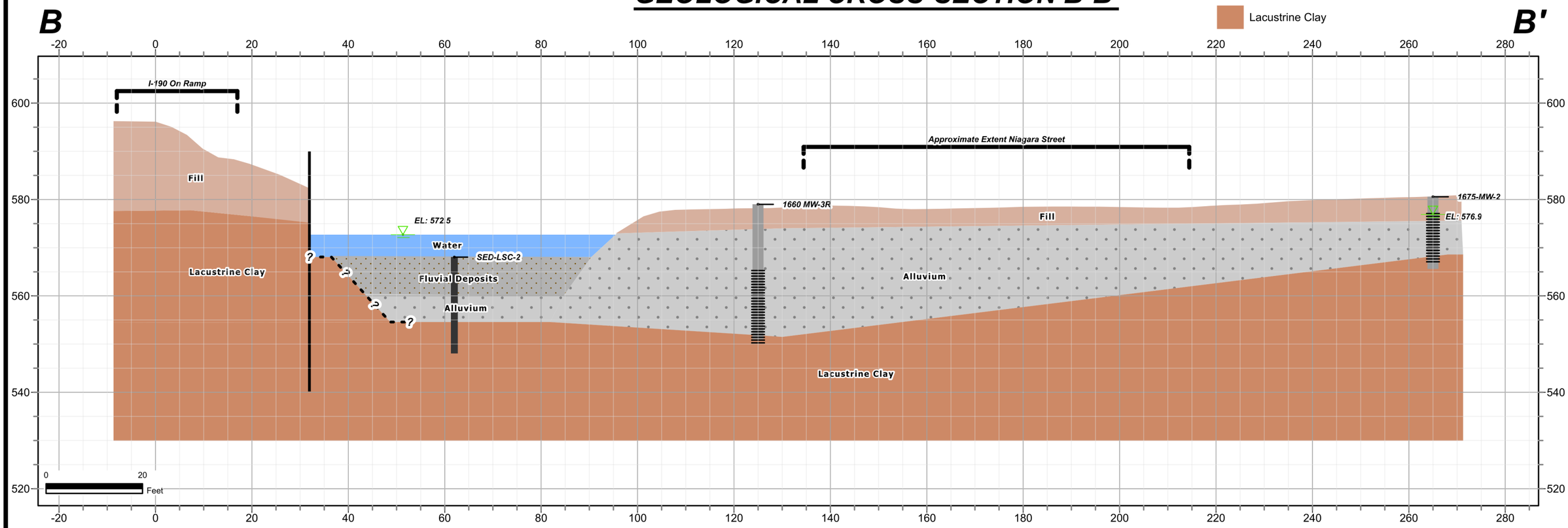


LEGEND:

- Groundwater / Surface Elevation (March 2022)
- Well Screen Length
- Monitoring Well
- Sediment Boring
- Sheet Pile Barrier
- ? -? Undefined Edge
- Water
- Fill
- Fluvial Deposits
- Alluvium
- Lacustrine Clay

GEOLOGICAL CROSS-SECTION B-B'



SOURCE: Existing ground surface elevation derived from USGS 2019 1 meter DEM. Accuracy is not guaranteed

Former Buffalo Gaslight Iroquois Gas Corp. Site	GEI Consultants	B to B' GEOLOGIC CROSSSECTION
Buffalo, New York	Project 2101620	June 2022 Figure 4B

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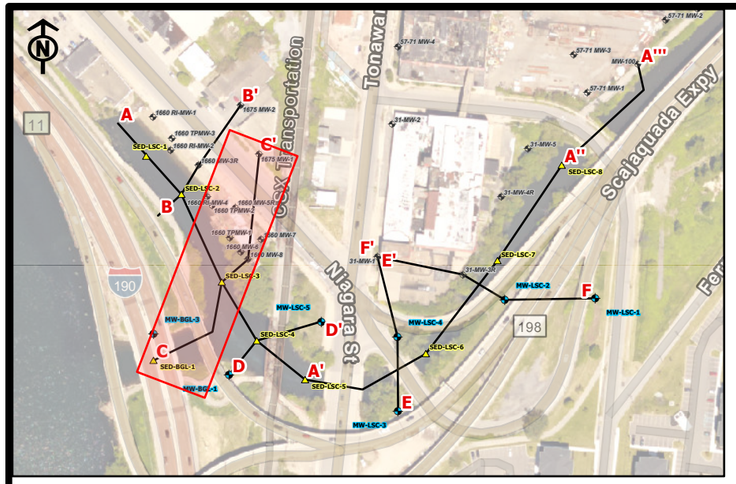
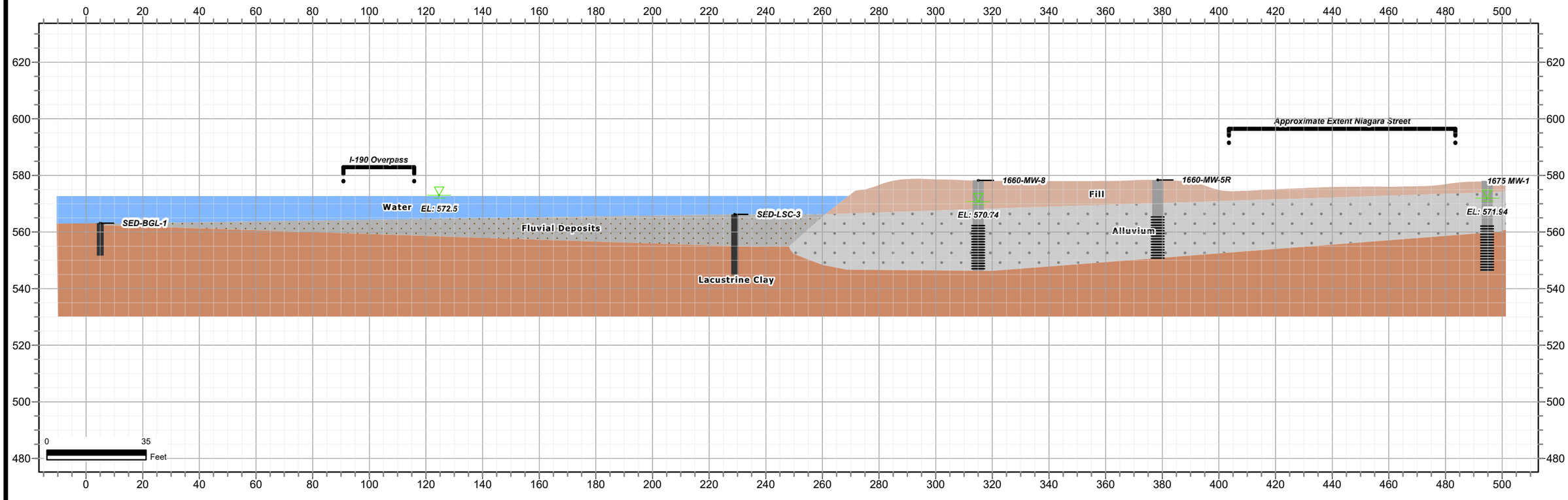


LEGEND:

- Groundwater / Surface Elevation (March 2022)
- Well Screen Length
- Monitoring Well
- Sediment Boring
- Water
- Fill
- Fluvial Deposits
- Alluvium
- Lacustrine Clay

GEOLOGICAL CROSS-SECTION C-C'



SOURCE: Existing ground surface elevation derived from USGS 2019 1 meter DEM. Accuracy is not guaranteed

Former Buffalo Gaslight Iroquois Gas Corp. Site	GEI Consultants	C to C' GEOLOGICAL CROSS-SECTIONS
Buffalo, New York	Project 2101620	June 2022 Figure 4C

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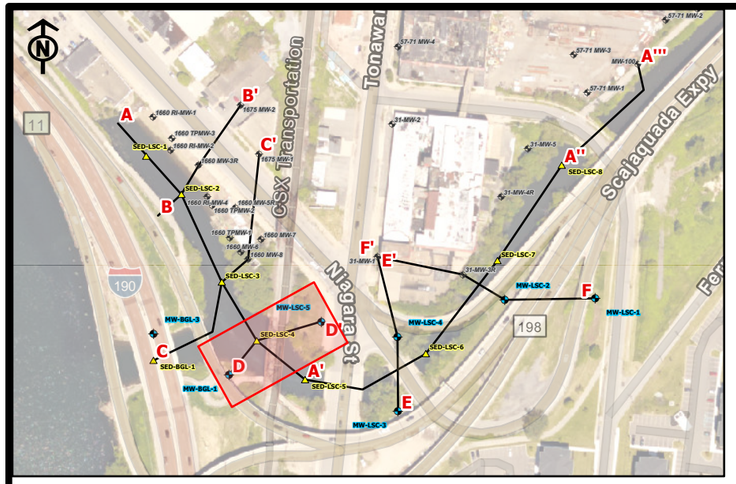
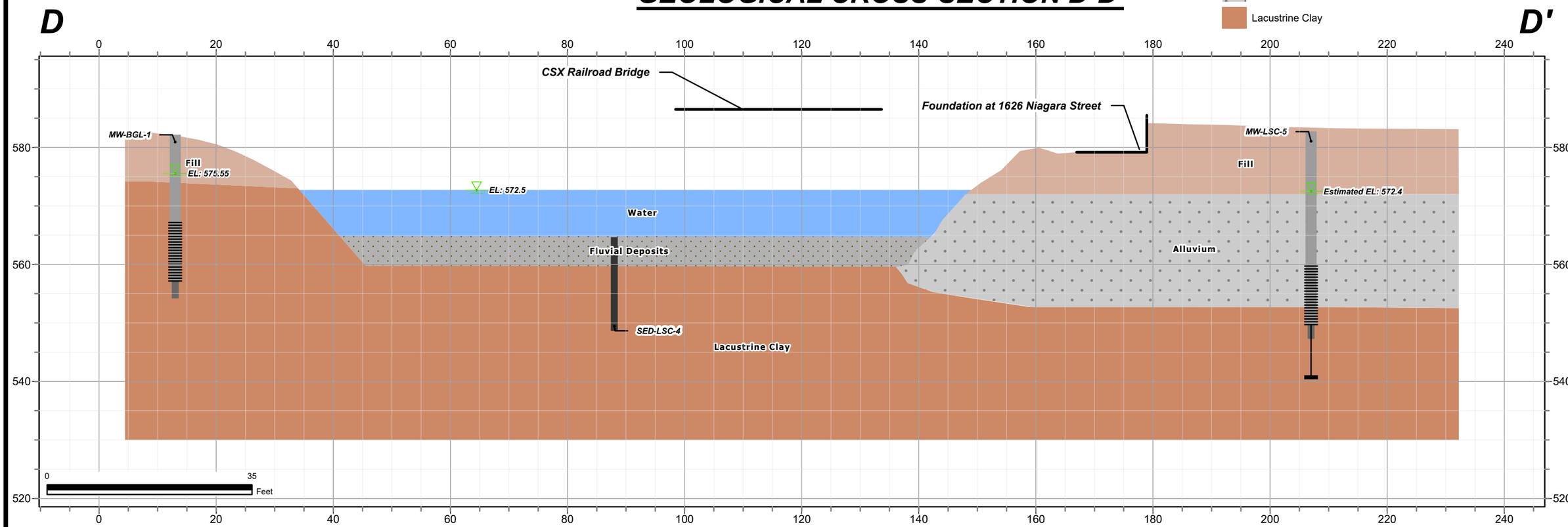


LEGEND:

- Groundwater / Surface Elevation (March 2022)
- Well Screen Length
- Well Sump (2ft Length)
- Exploratory Boring (Bentonite Chip Backfill)
- Monitoring Well
- Sediment Boring
- Concrete Foundation Wall
- Water
- Fill
- Fluvial Deposits
- Alluvium
- Lacustrine Clay

GEOLOGICAL CROSS-SECTION D-D'



SOURCE: Existing ground surface elevation derived from USGS 2019 1 meter DEM. Accuracy is not guaranteed

Former Buffalo Gaslight
Iroquois Gas Corp. Site

Buffalo, New York

GEI Consultants

D to D' GEOLOGICAL CROSS-SECTIONS

Project 2101620

June 2022

Figure 4D

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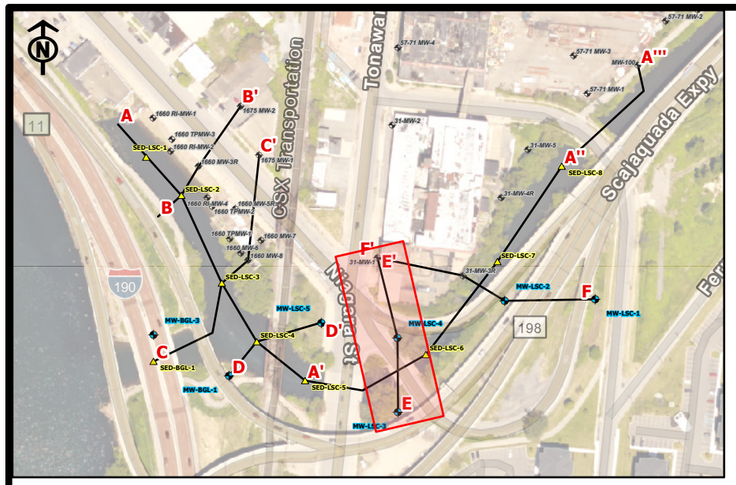
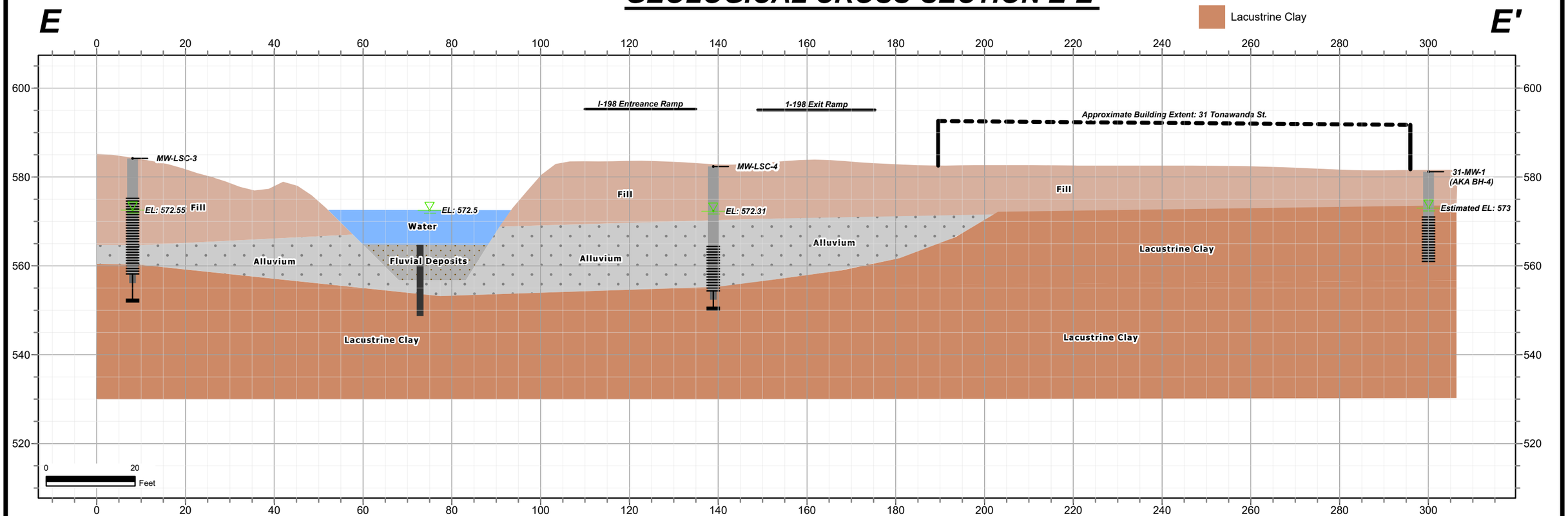


LEGEND:

- Groundwater / Surface Elevation (March 2022)
- Well Screen Length
- Well Sump (2ft Length)
- Exploratory Boring (Bentonite Chip Backfill)
- Monitoring Well
- Sediment Boring
- Water
- Fill
- Fluvial Deposits
- Alluvium
- Lacustrine Clay

GEOLOGICAL CROSS-SECTION E-E'



SOURCE: Existing ground surface elevation derived from USGS 2019 1 meter DEM. Accuracy is not guaranteed

Former Buffalo Gaslight Iroquois Gas Corp. Site	GEI Consultants	E to E' GEOLOGICAL CROSS-SECTIONS
Buffalo, New York	Project 2101620	June 2022 — Figure 4E

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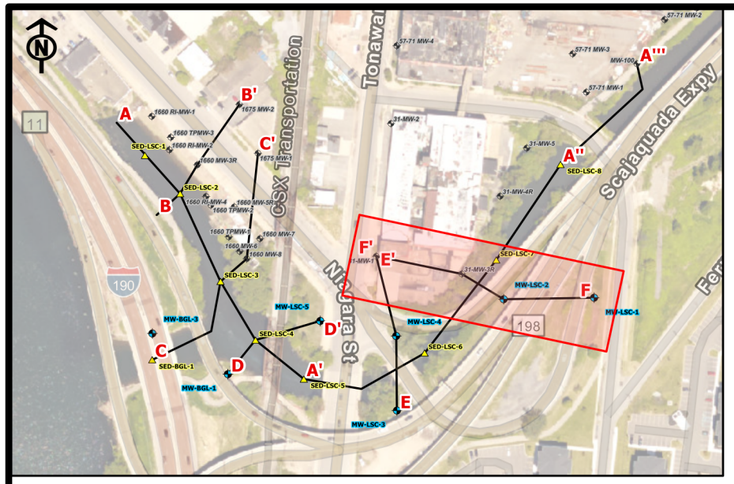
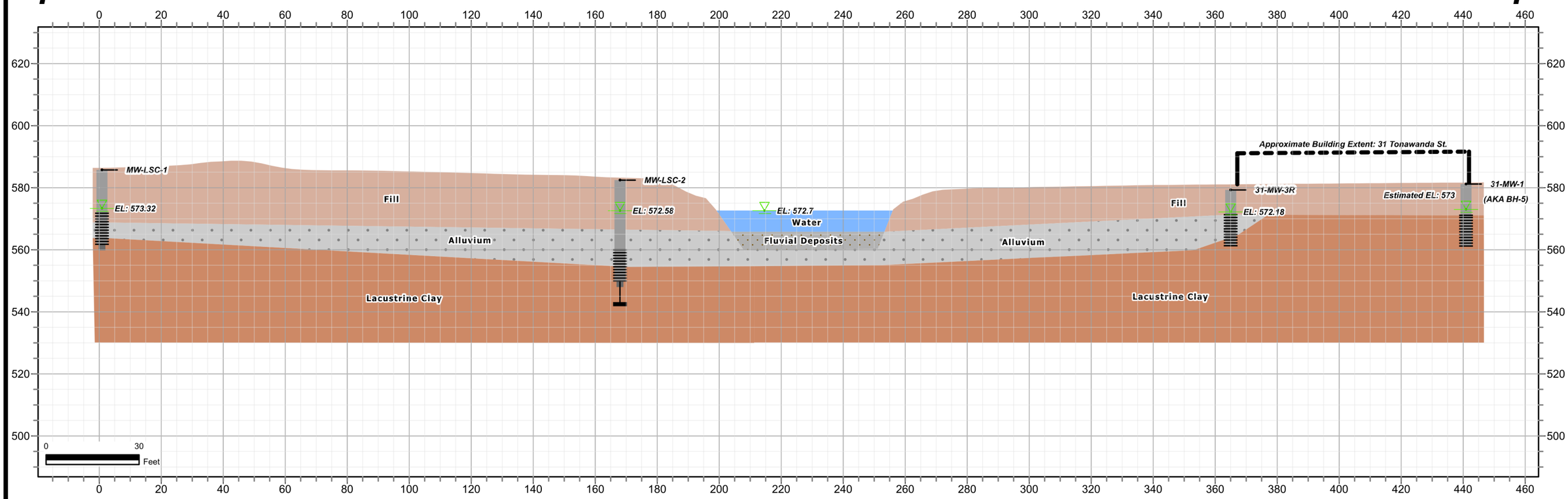


LEGEND:

- Groundwater / Surface Elevation (March 2022)
- Well Screen Length
- Well Sump (2ft Length)
- Exploratory Boring (Bentonite Chip Backfill)
- Monitoring Well
- Water
- Fill
- Fluvial Deposits
- Alluvium
- Lacustrine Clay

GEOLOGICAL CROSS-SECTION F-F'



SOURCE: Existing ground surface elevation derived from USGS 2019 1 meter DEM. Accuracy is not guaranteed

Former Buffalo Gaslight Iroquois Gas Corp. Site	GEI Consultants	F to F' GEOLOGIC CROSSSECTION
Buffalo, New York	Project 2101620	June 2022 Figure 4F

Path: C:\Users\jvazak\OneDrive - GEI Consultants, Inc\Scajaquada\140\640 - National Fuel Westwood Site2.aprx

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68-MW-3
57-71-MW-5
582'
MW-106
559.84'
MW-103
556.31'
57-71-MW-2
573.5'
57-71-MW-3
571.5'
68-MW-4
57-71-MW-4
581'
MW-100
555.32'
57-71-MW-1
570.5'
1675-MW-2
568.58'
1660-MW-1
31-MW-2
566'
31-MW-5
1660 TPMW-3
SED-LSC-1
552.76'
1660-MW-2
1675-MW-1
560.1'
SED-LSC-8
556.56'
1660-MW-3R
551.51'
SED-LSC-2
554.56'
1660-MW-4
31-MW-4R
1660 TPMW-2
1660-MW-5R
552.02'
1660 TPMW-1
1660-MW-7
31-MW-1
562'
31-MW-3R
566.18'
SED-LSC-7
554.81'
1660-MW-6
1660-MW-8
546.27'
SED-LSC-3
554.76'
MW-LSC-2
554.46'
MW-LSC-1
563.76'
MW-LSC-5
552.72'
MW-BGL-3
557.88'
SED-LSC-4
559.66'
MW-LSC-4
555.37'
SED-BGL-1
562.96'
SED-LSC-6
553.21'
MW-BGL-1
574.17'
SED-LSC-5
559.16'
MW-LSC-3
560.19'

578' 576' 574' 572' 570' 568' 566' 564' 562' 560' 558' 556' 554' 552'

Scajaquada Creek
Black Rock Canal
Niagara Thruway
SQUAW ISLAND RD
DEARBORN ST
WAYNE ST
TONAWANDA ST
NIAGARA ST
SCAJAQUADA EXPY NY 198
BRADLEY ST
FENNWOOD AVE
WEST AVE
DANFORTH ST
SR198 NB to I190 NB
I190 SB to SR198 EB
SR198 WB to I190 SB
I190 NB to SR198 EB
NY266 NB to NY198 EB
NY198 WB to NY265

0 50 100 150 Feet

LEGEND:

Boring Type w/Clay Elevation (famsl)

- 2021 GEI Sediment Boring
- 2021/2022 GEI Monitoring Well
- Existing Monitoring Well
- Top of Clay Contour (famsl)

NAD 1983 2011 StatePlane New York West FIPS 3103 Ft US

Former Buffalo Gaslight Iroquois Gas Corp. Site

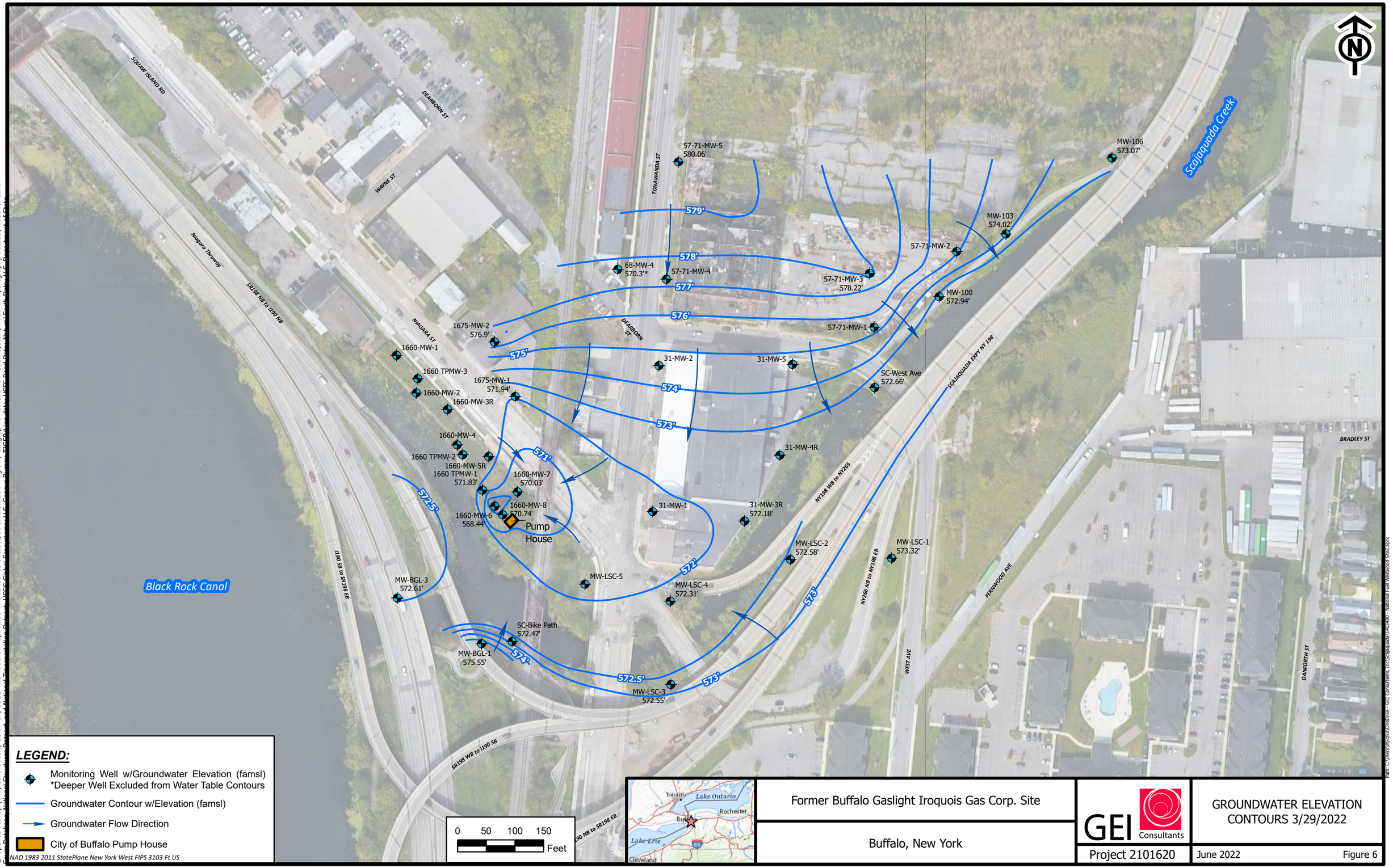
Buffalo, New York

GEI Consultants

TOP OF CLAY SURFACE ELEVATION CONTOURS

Project 2101620 | June 2022 | Figure 5

Path: C:\Users\jdyczak\OneDrive - GEI Consultants, Inc\Scajaquada\1403580 - National Fuel Westwood Site2.aprx



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Former Buffalo Gaslight Iroquois Gas Corp. Site

Buffalo, New York

GEI Consultants

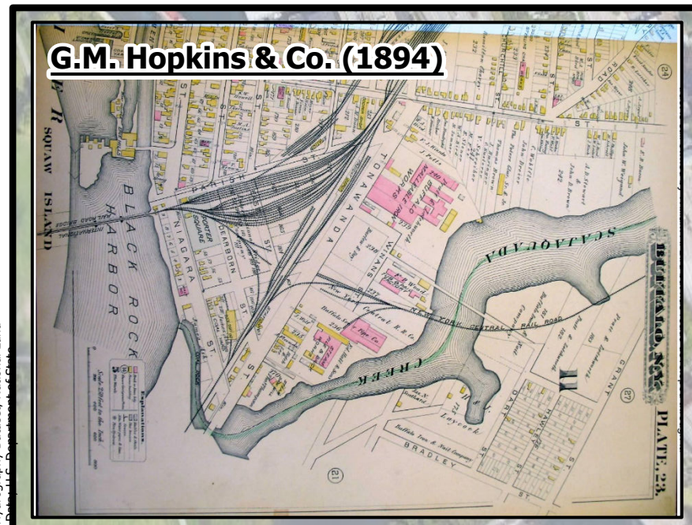
Project 2101620

GROUNDWATER ELEVATION CONTOURS 3/29/2022

June 2022

Figure 6

Path: C:\Users\dzyzik\OneDrive - GEI Consultants, Inc\Scajaquada\Buffalo - National Fuel Westwood 5462.aprx



LEGEND:

Historic Creek Extent - G.M. Hopkins & Co. (1884)

NAD 1983 2011 StatePlane New York West FIPS 3103 Ft US

0 75 150 300 Feet

Former Buffalo Gaslight Iroquois Gas Corp. Site

Buffalo, New York

GEI Consultants

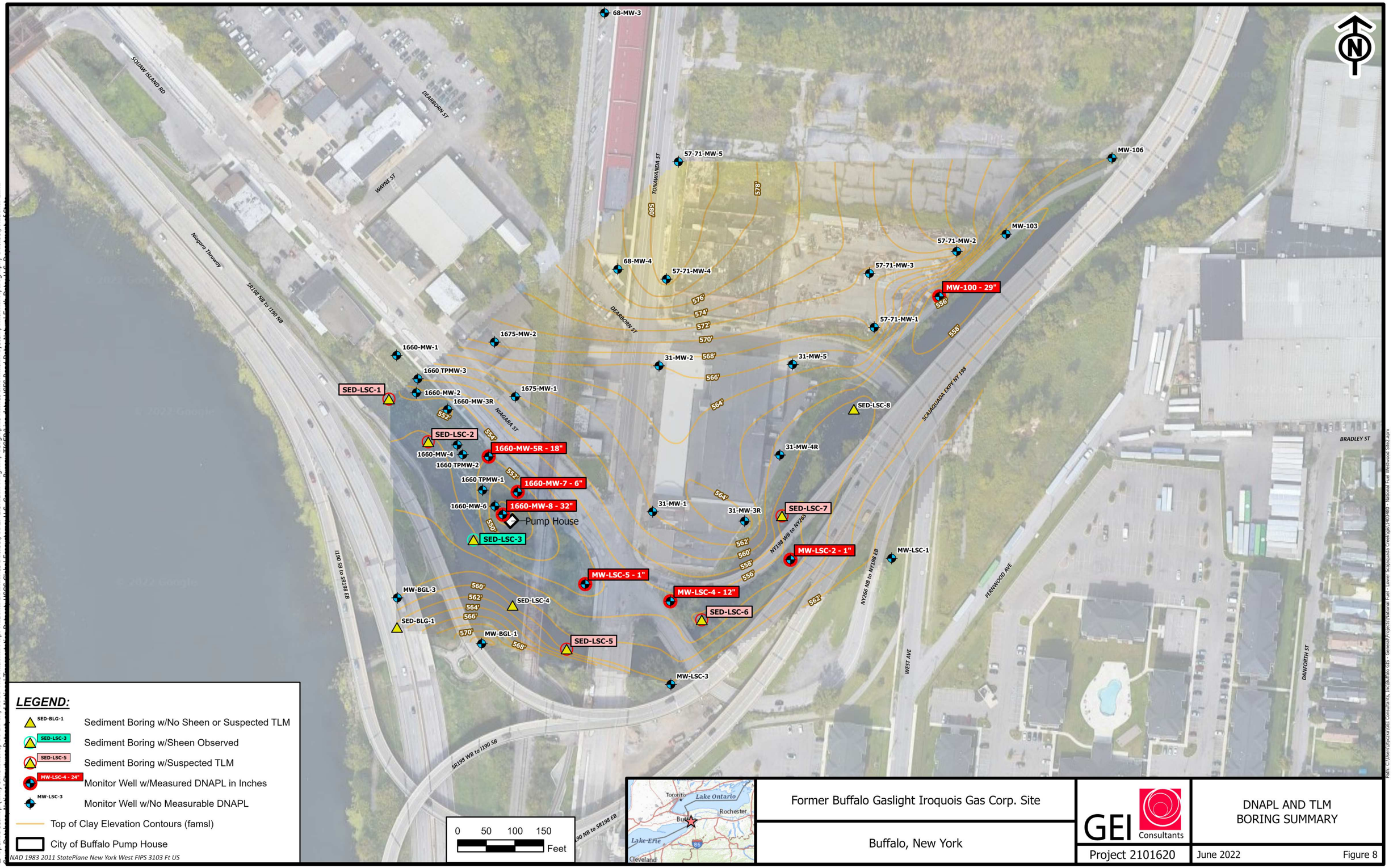
Project 2101620

HISTORIC SCAJAQUADA CREEK EXTENT

June 2022 Figure 7

© OpenStreetMap (and) contributors, CC-BY-SA, USGS The National Map: National Boundaries Dataset, 3DEP Elevation Program, Geographic Names Information System, National Hydrography Dataset, Source: USGS 3 arc second | 0 | 100 | Source: USGS National Elevation Dataset (NED) 1/3 arc-second | Date: 6/2/2022 | Publication Date: 2022-05-25

Path: C:\Users\ryzzo\OneDrive - GEI Consultants, Inc\Scajaquada\MXD\80 - National Park Westwood Sheet.aprx



LEGEND:

- SED-BLG-1 — Sediment Boring w/No Sheen or Suspected TLM
- SED-LSC-3 — Sediment Boring w/Sheen Observed
- SED-LSC-5 — Sediment Boring w/Suspected TLM
- MW-LSC-4 - 24" — Monitor Well w/Measured DNAPL in Inches
- MW-LSC-3 — Monitor Well w/No Measurable DNAPL
- Top of Clay Elevation Contours (famsl)
- City of Buffalo Pump House

NAD 1983 2011 StatePlane New York West FIPS 3103 Ft US

Former Buffalo Gaslight Iroquois Gas Corp. Site

Buffalo, New York

GEI Consultants

Project 2101620

DNAPL AND TLM BORING SUMMARY

June 2022

Figure 8

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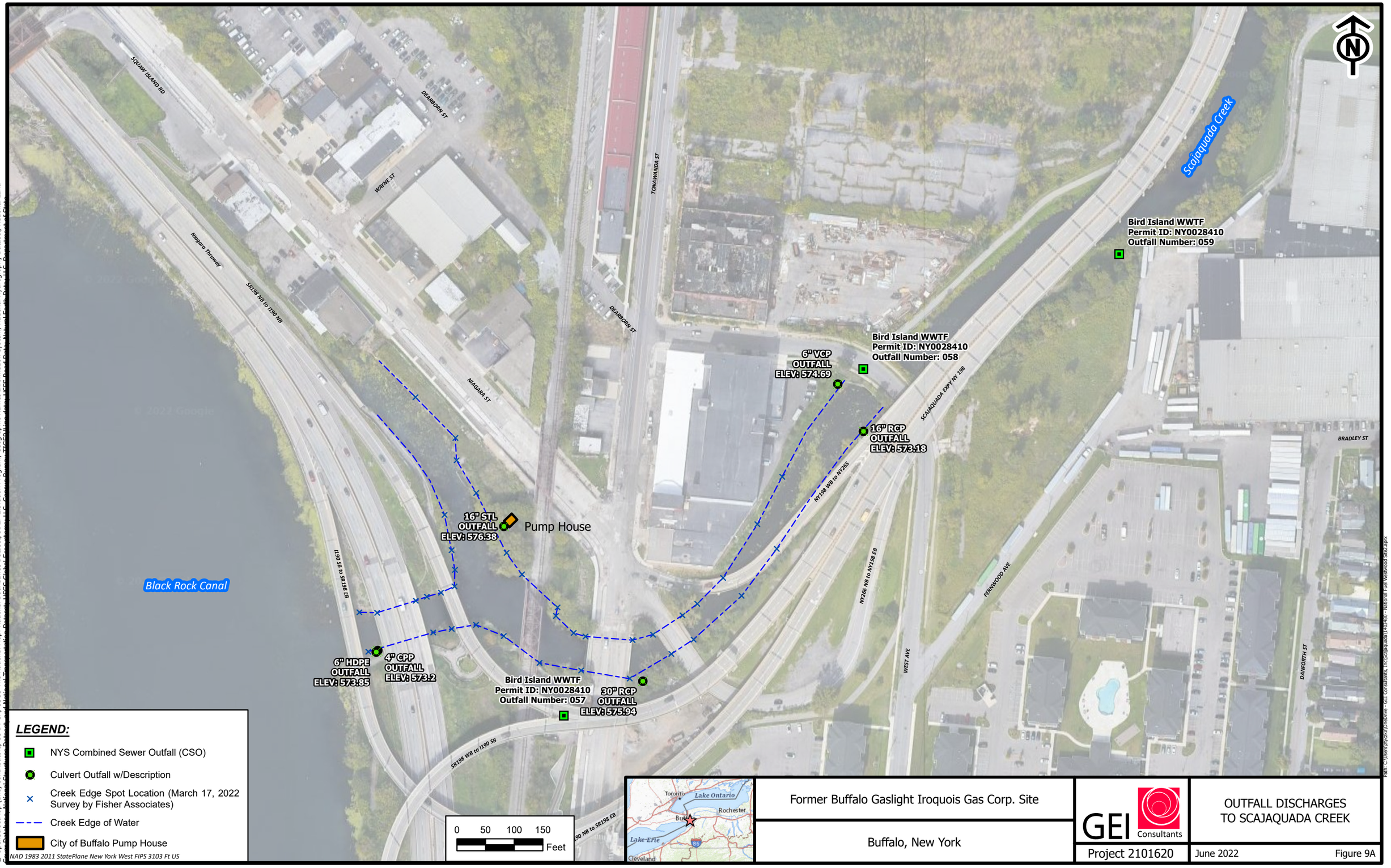


LEGEND:

- NYS Combined Sewer Outfall (CSO)
- Culvert Outfall w/Description
- Creek Edge Spot Location (March 17, 2022 Survey by Fisher Associates)
- Creek Edge of Water
- City of Buffalo Pump House

NAD 1983 2011 StatePlane New York West FIPS 3103 Ft US

Former Buffalo Gaslight Iroquois Gas Corp. Site

Buffalo, New York

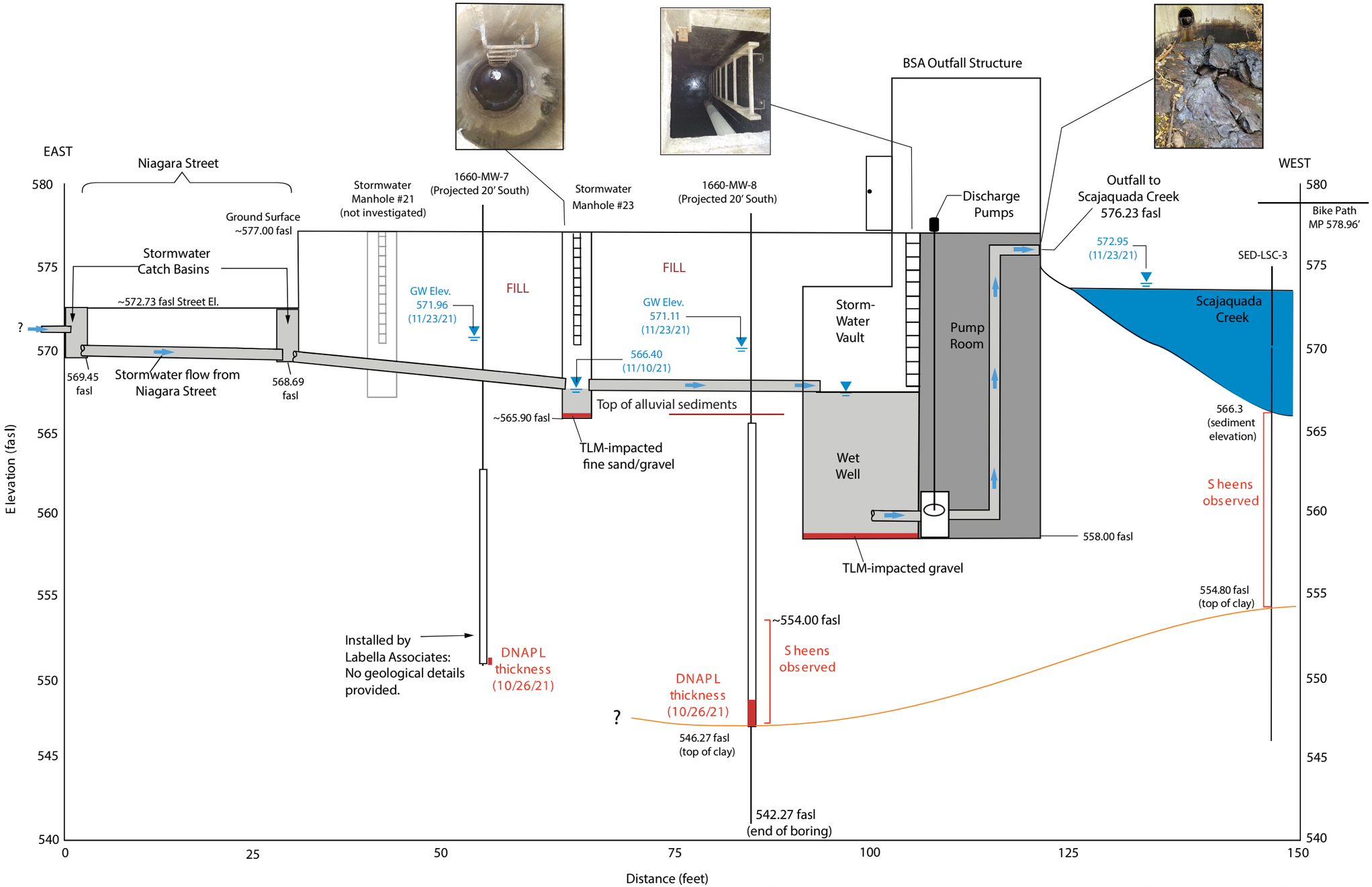
GEI Consultants

OUTFALL DISCHARGES TO SCAJAQUADA CREEK

Project 2101620 | June 2022 | Figure 9A

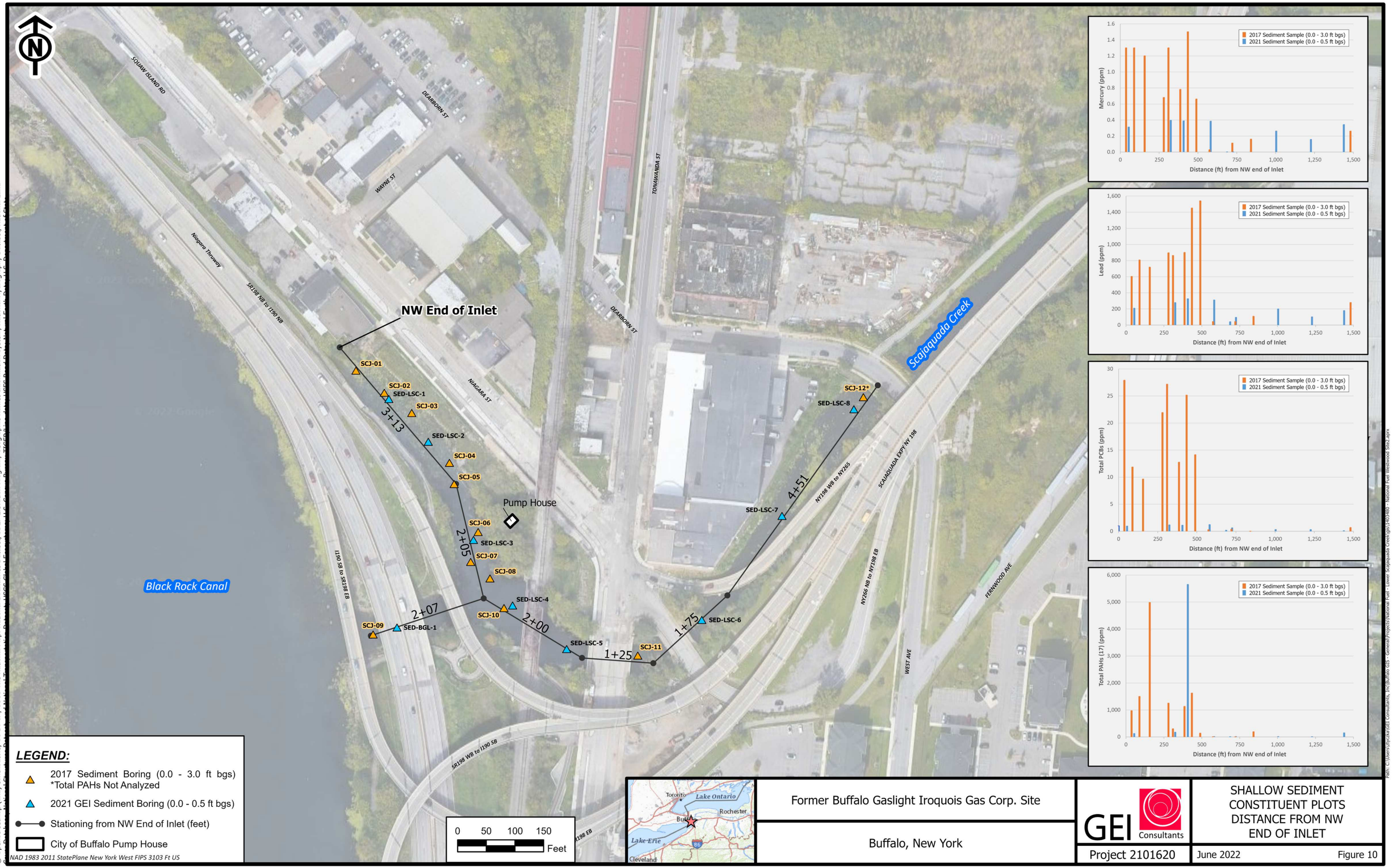
© OpenStreetMap (and) contributors, CC-BY-SA, USGS The National Map: National Boundaries Dataset, 3DEP Elevation Program, Geographic Names Information System, National Hydrography Dataset, Source: USGS National Map 3DEP Elevation Program, USGS National Geospatial Program

Path: C:\Users\yzarzar\OneDrive - GEI Consultants, Inc\Scajaquada\140480 - National Fuel Westwood SAGE.aprx



Former Buffalo Gaslight Iroquois Gas Corp. Site	GEI Consultants	CITY OF BUFFALO PUMP STATION PROFILE	
Buffalo, New York	Project 2101620	June 2022	Figure 9B

Path: C:\Users\dvizza\OneDrive - GEI Consultants, Inc\Scajaquada\140480 - National Fuel Wellwood Site.aprx



LEGEND:

- 2017 Sediment Boring (0.0 - 3.0 ft bgs) *Total PAHs Not Analyzed
- 2021 GEI Sediment Boring (0.0 - 0.5 ft bgs)
- Stationing from NW End of Inlet (feet)
- City of Buffalo Pump House

NAD 1983 2011 StatePlane New York West FIPS 3103 Ft US

Former Buffalo Gaslight Iroquois Gas Corp. Site

Buffalo, New York

GEI Consultants

Project 2101620

SHALLOW SEDIMENT CONSTITUENT PLOTS DISTANCE FROM NW END OF INLET

June 2022 Figure 10

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Path: C:\Users\chycka\GEI Consultants, Inc\Buffalo GIS - General\Projects\National Fuel - Lower Scajaquada Creek\gis\MXD\MD - National Fuel Westwood Site2.aprx

Appendix B

Soil Boring and Well Construction Logs

Appendix B.1

BCP Sites

DRILLING SUMMARY

Geologist/Technician: Pete Gorton

Drilling Company: Nature's Way

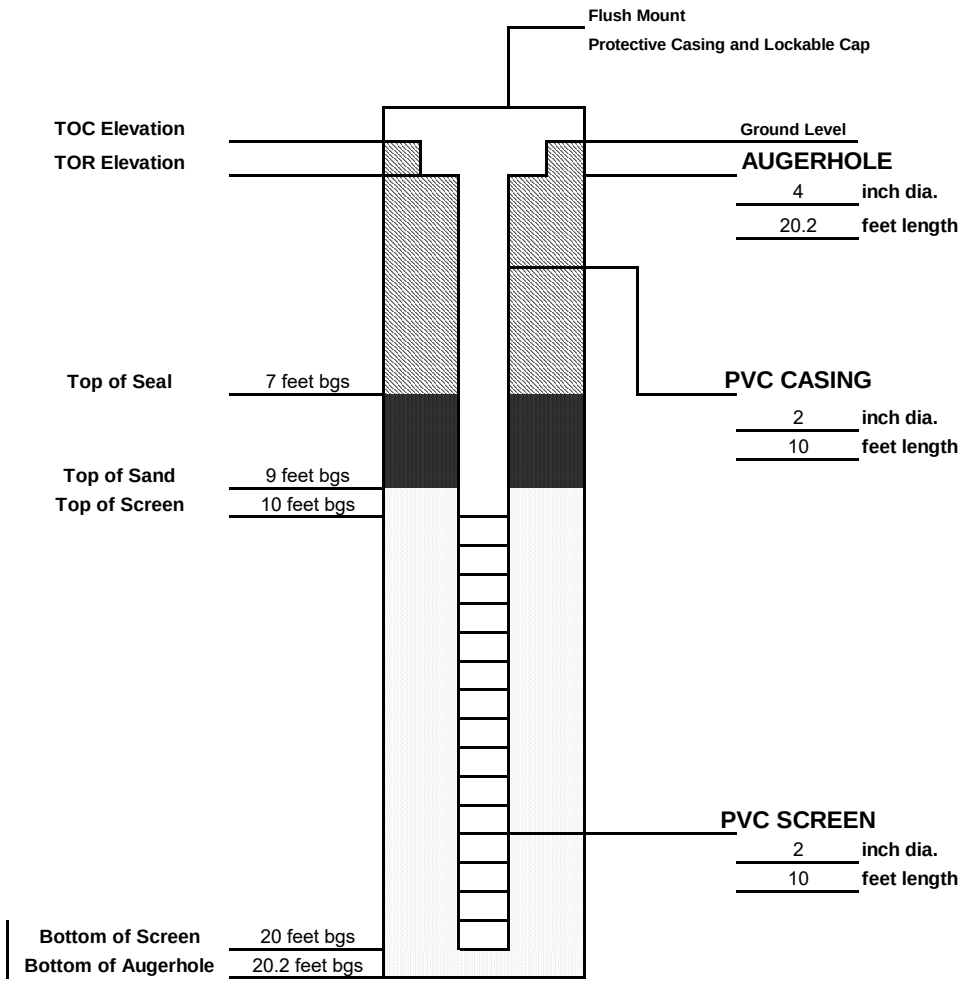
Driller: Brett

Model: Truck Mounted Rig, Hollow Stem Auger

Date: 8/29/2018

GEOLOGIC LOG

Depth(ft.)	Description
	N/A



WELL DESIGN

CASING MATERIAL	*SCREEN MATERIAL*	*FILTER MATERIAL*
Surface: Steel grade box	**Type:** 2" PVC	**Type:** #0 Sand **Setting:** 10 to 9 feet bgs
	Slot Size: 0.010"	***SEAL MATERIAL***
Monitor: N/A	**Setting:** 20 to 10 feet bgs	**Type:** Bentonite Chips **Setting:** 9 to 7 feet bgs

COMMENTS:

LEGEND

- Cement Grout
- Bentonite Seal
- Silica Sandpack

Client: 31 Tonawanda St, LLC	**Location:** 31 Tonawanda St, Buffalo, NY	**Project No.:**
BE3 CORP PANAMERICAN ENVIRONMENT • ENGINEERING • ENERGY	**MONITORING WELL CONSTRUCTION DETAILS**	**Well Number: 31-MW-1**

DRILLING SUMMARY

Geologist/Technician: Pete Gorton

Drilling Company: Nature's Way

Driller: Brett

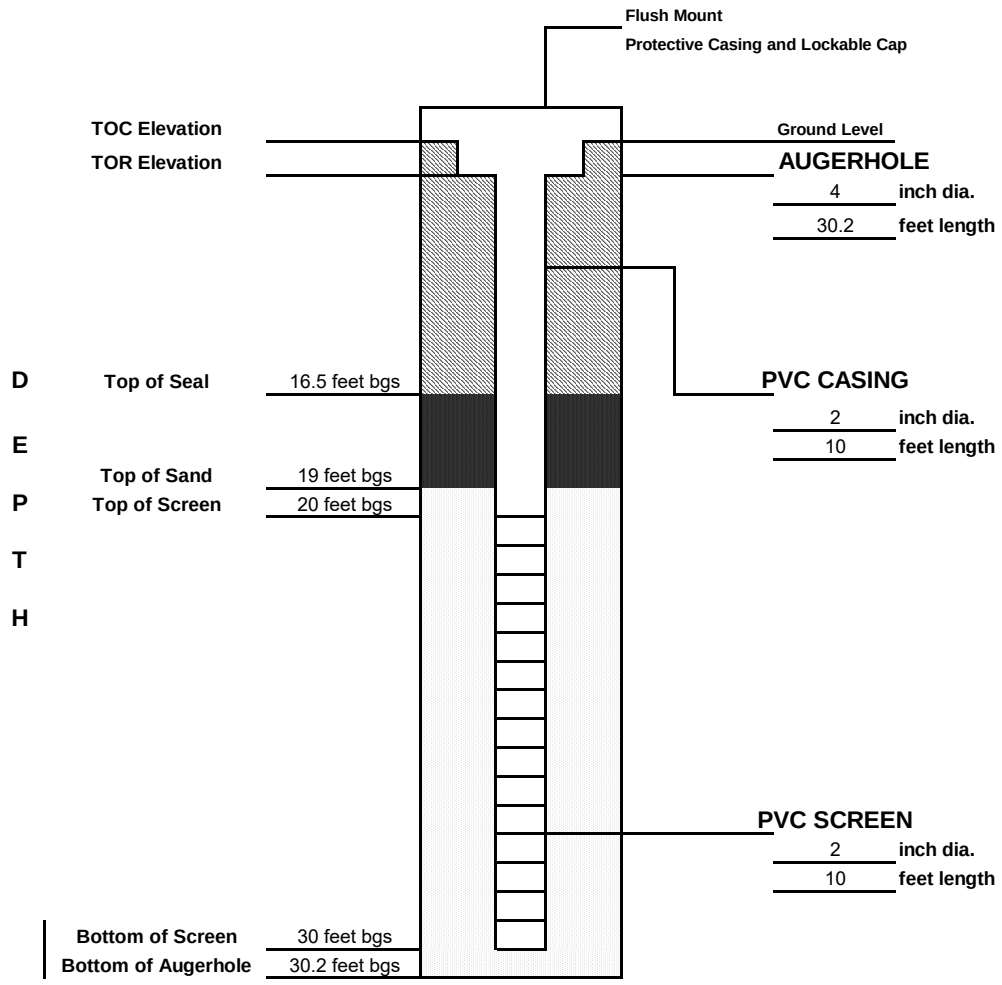
Model: Truck Mounted Rig, Hollow Stem Auger

Date: 8/29/2018

GEOLOGIC LOG

Depth(ft.)	Description
	N/A

WELL DESIGN



CASING MATERIAL	*SCREEN MATERIAL*	*FILTER MATERIAL*
Surface: Steel grade box	**Type:** 2" PVC	**Type:** #0 Sand **Setting:** 19 to 30 feet bgs
Monitor: N/A	**Slot Size:** 0.010" **Setting:** 30 to 20 feet bgs	***SEAL MATERIAL*** **Type:** Bentonite Chips **Setting:** 19 to 16.5 feet bgs

COMMENTS:

LEGEND

- Cement Grout
- Bentonite Seal
- Silica Sandpack

Client: 31 Tonawanda St, LLC

Location: 31 Tonawanda St, Buffalo, NY

Project No.:

BE3 CORP PANAMERICAN
ENVIRONMENT • ENGINEERING • ENERGY

MONITORING WELL CONSTRUCTION DETAILS

Well Number: 31-MW-2

DRILLING SUMMARY

Geologist/Technician: Alex Brennen

Drilling Company: Nature's Way

Driller: Brett

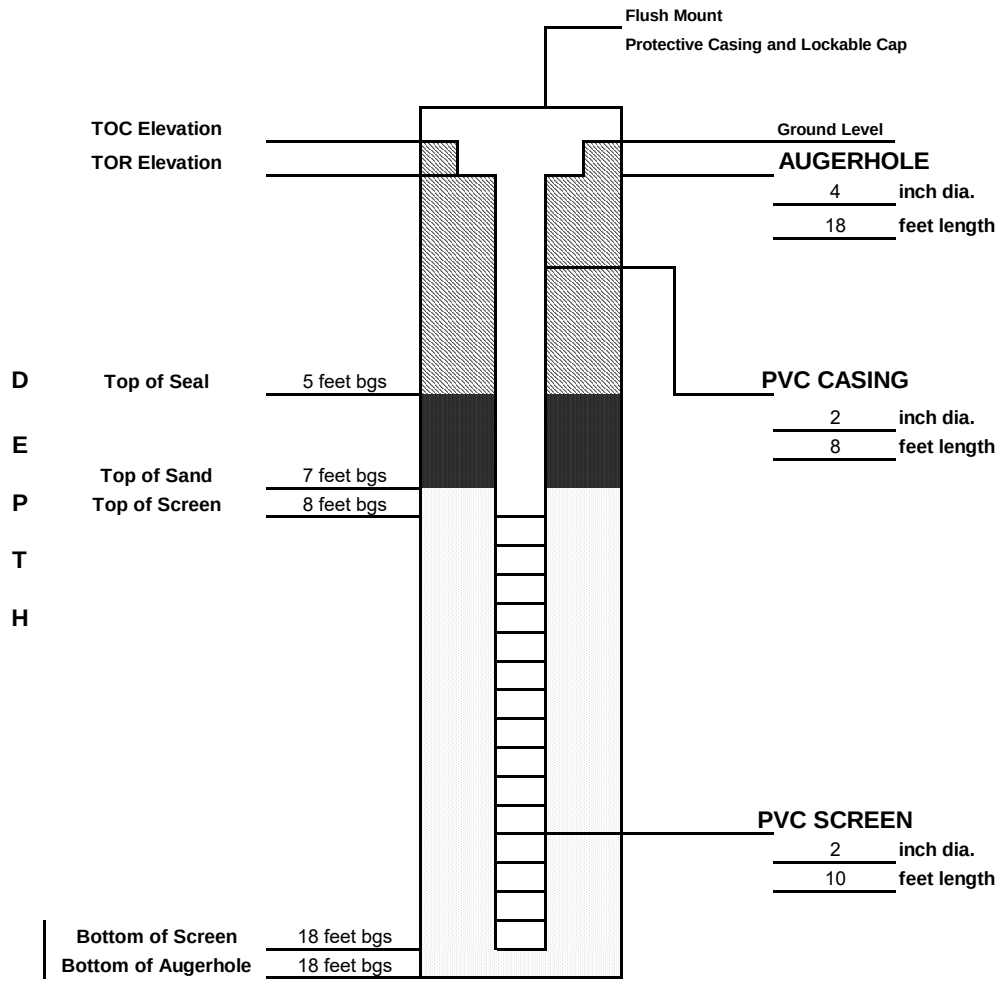
Model: Truck Mounted Rig, Hollow Stem Auger

Date: 8/27/2018

GEOLOGIC LOG

Depth(ft.)	Description
	N/A

WELL DESIGN



CASING MATERIAL	*SCREEN MATERIAL*	*FILTER MATERIAL*
Surface: Steel grade box	**Type:** 2" PVC	**Type:** #0 Sand
	Slot Size: 0.010"	**Setting:** 7 to 18 feet bgs
Monitor: N/A	**Setting:** 8 to 18 feet bgs	***SEAL MATERIAL***
		Type: Bentonite Chips
		Setting: 7 to 5 feet bgs

COMMENTS:

LEGEND

- Cement Grout
- Bentonite Seal
- Silica Sandpack

Client: 31 Tonawanda St, LLC

Location: 31 Tonawanda St, Buffalo, NY

Project No.:

BE3 CORP PANAMERICAN ENVIRONMENT • ENGINEERING • ENERGY

MONITORING WELL CONSTRUCTION DETAILS

Well Number: 31-MW-3

DRILLING SUMMARY

Geologist/Technician: Alex Brennen

Drilling Company: Nature's Way

Driller: Brett

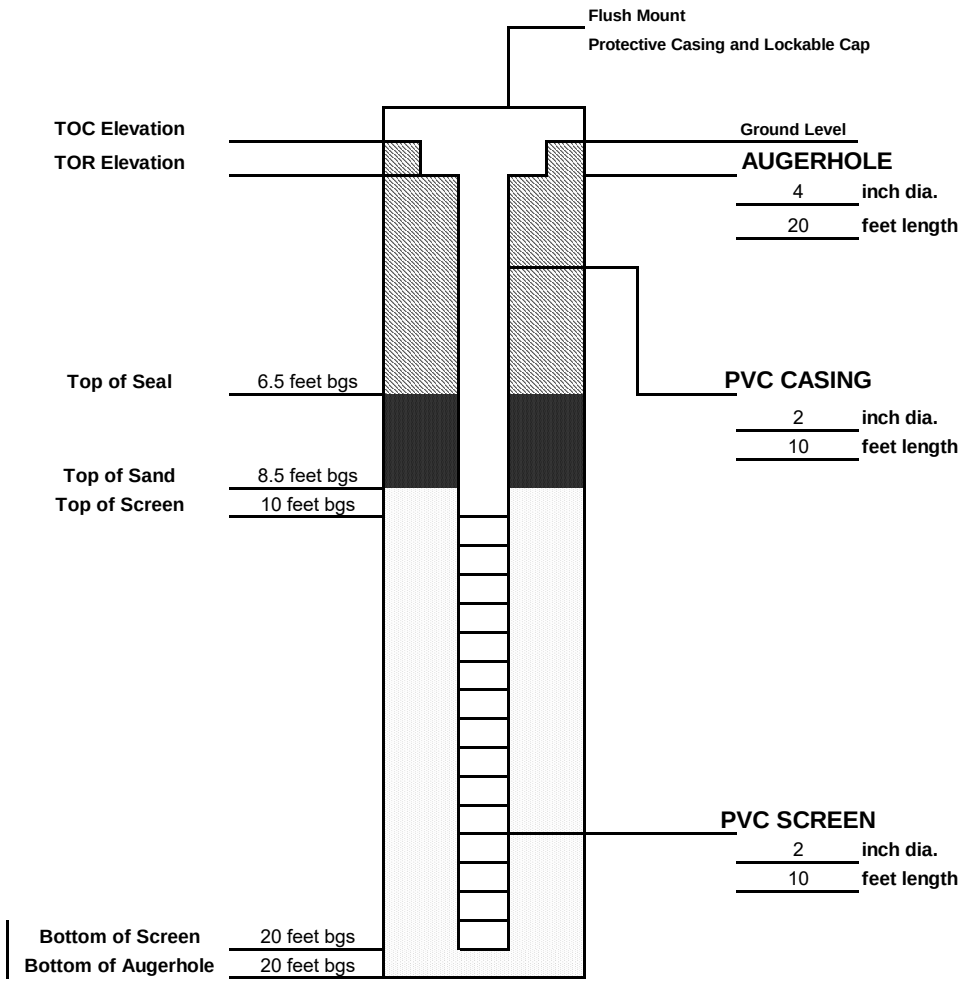
Model: Truck Mounted Rig, Hollow Stem Auger

Date: 8/28/2018

GEOLOGIC LOG

Depth(ft.)	Description
	N/A

WELL DESIGN



CASING MATERIAL	*SCREEN MATERIAL*	*FILTER MATERIAL*
Surface: Steel grade box	**Type:** 2" PVC	**Type:** #0 Sand
	Slot Size: 0.010"	**Setting:** 8.5 to 20 feet bgs
Monitor: N/A	**Setting:** 10 to 20 feet bgs	***SEAL MATERIAL***
		Type: Bentonite Chips
		Setting: 8.5 to 6.5 feet bgs

COMMENTS:

LEGEND

- Cement Grout
- Bentonite Seal
- Silica Sandpack

Client: 31 Tonawanda St, LLC

Location: 31 Tonawanda St, Buffalo, NY

Project No.:

BE3 CORP PANAMERICAN
ENVIRONMENT • ENGINEERING • ENERGY

MONITORING WELL CONSTRUCTION DETAILS

Well Number: 31-MW-4

DRILLING SUMMARY

Geologist/Technician: Pete Gorton

Contractor: Nature's Way

Operator: Brett

Model: Truck Mounted Rig
Hollow Stem Auger

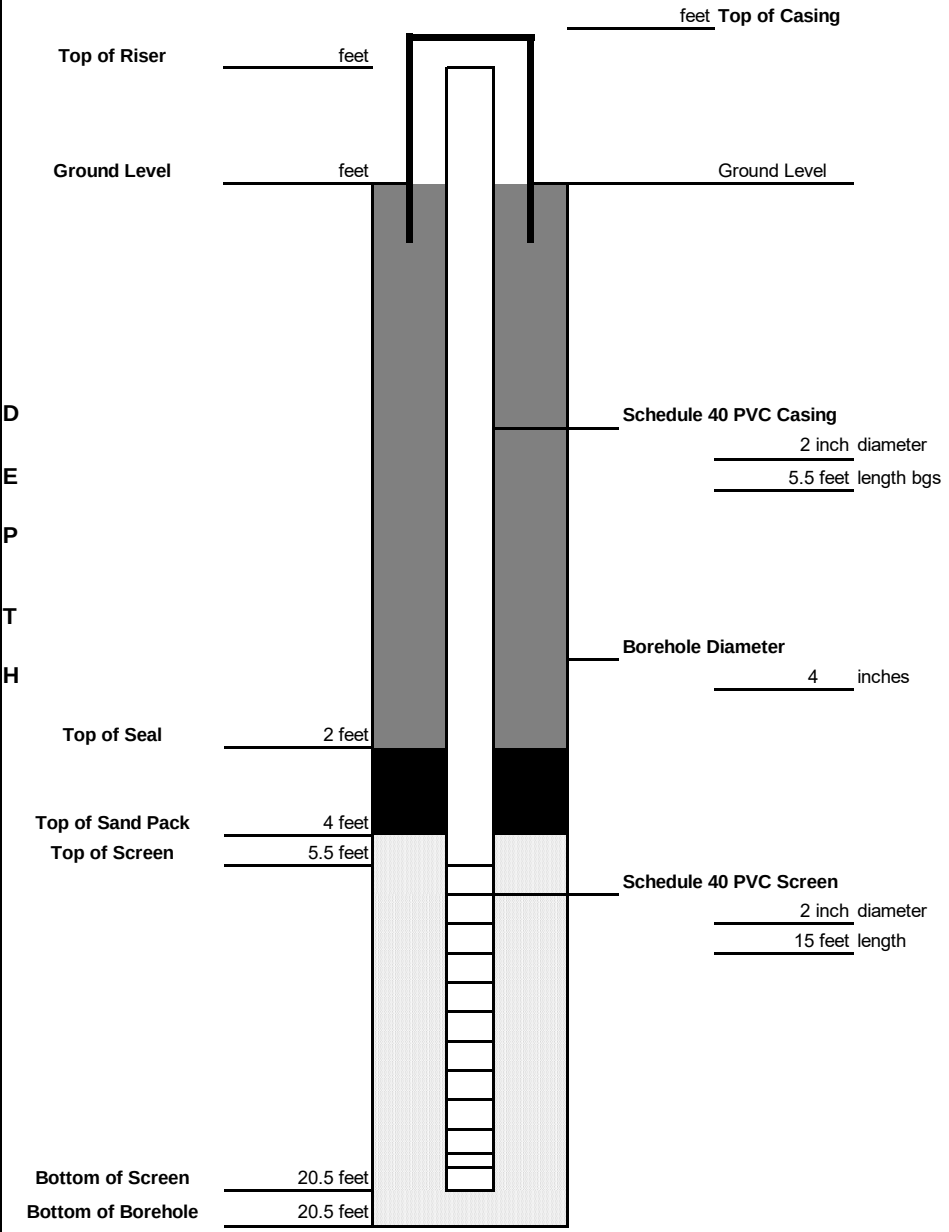
Date: 8/27/2018

GEOLOGIC LOG

Depth(ft.)	Description
4 to 6	PID Readings of cuttings 2-15 ppm

WELL DESIGN

DEPTH



CASING MATERIAL

Surface: 4" Steel protective cover (Stick Up)

Monitor: 2" Schedule 40 PVC

SCREEN MATERIAL

Type: 2" Schedule 40 PVC

Slot Size: 0.010"

Setting: 6 to 36 fbgs

FILTER MATERIAL

Type: #0 well sand

Setting: 5 to 36 fbgs

SEAL MATERIAL

Type 1: Bentonite chips

Setting: 2 to 5 fbgs

Type 1: Cement Grout

Setting: 0 to 2 fbgs

COMMENTS:

Very tight clays noted below fill. Drill depth done until clay softened to ensure water generation

LEGEND

- Cement Grout
- Bentonite Seal
- Sand Pack

Client: 31 Tonawanda St, LLC

Location: 31 Tonawanda St, Buffalo, NY

Project No.:

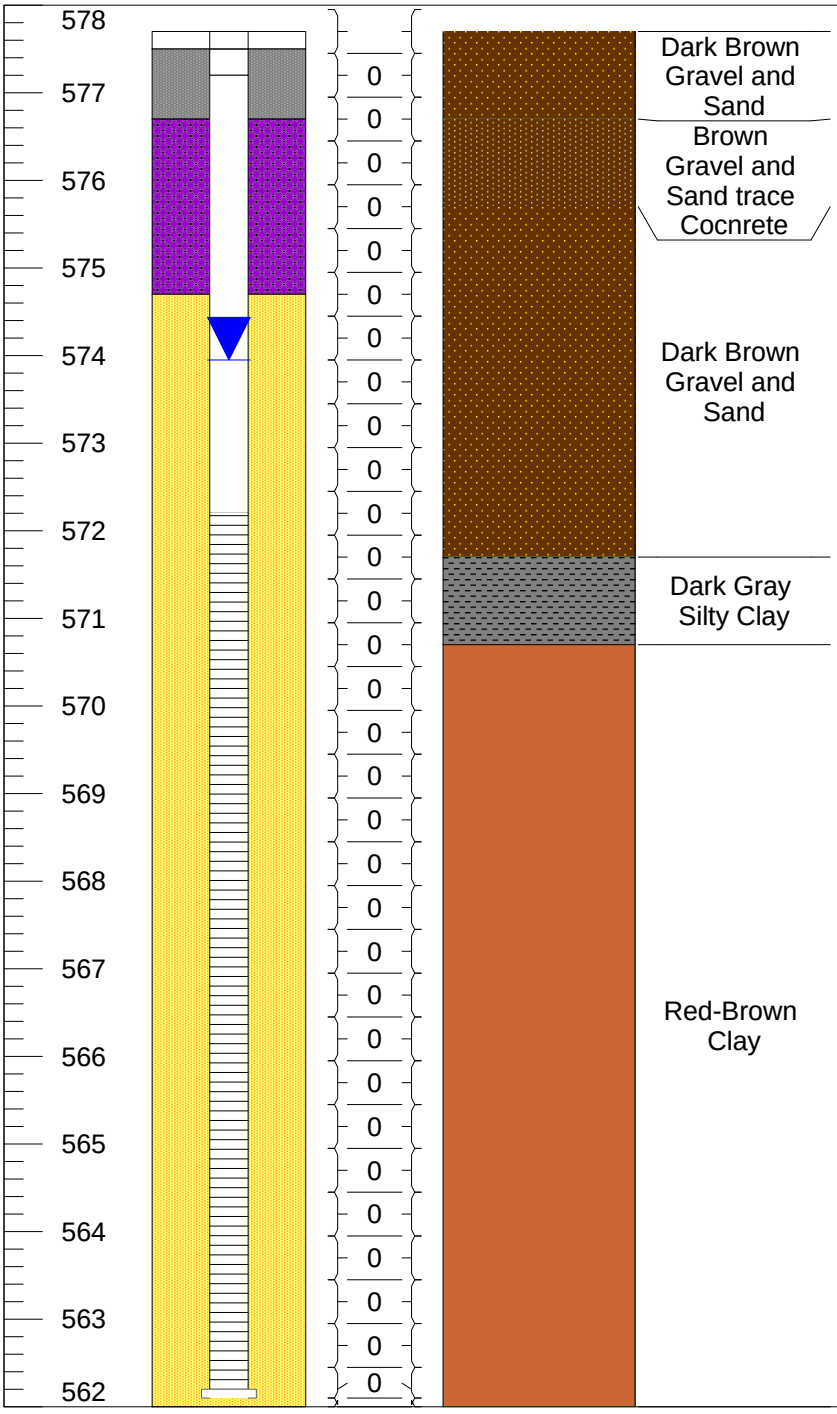
BE3 CORP PANAMERICAN ENVIRONMENT · ENGINEERING · ENERGY

MONITORING WELL CONSTRUCTION DETAILS

Well Number: 31-MW-5

Well Construction Log

Project:	Fedder Lofts	Drilling Company:	Nature's Way Contracting
Location:	57 Tonawanda Street	Drill Type:	6" Hollow Stem Auger
Client:	Eran Epstein	Driller:	Nate
Client Address:	57-71 Tonawanda Street Buffalo, NY 14211	Drill Date:	1/13/2020



LEGEND

- Cement-Bentonite Grout
- Bentonite Seal
- Silica Pack
- Static Water Level

Well Construction Properties

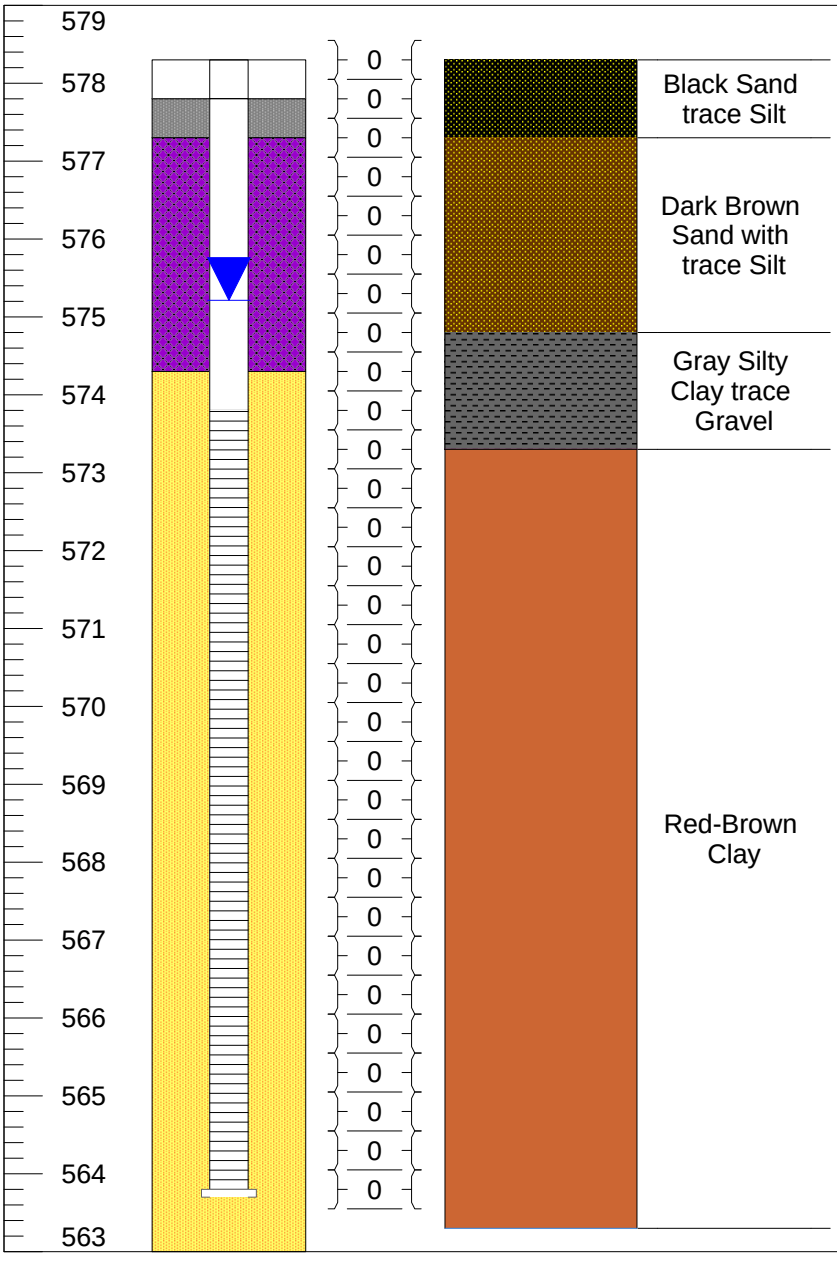
Material:	PVC
Diameter:	2"
Slot Count:	10
Slot Size:	2"
Seal Material:	Benotonite
Packing Material:	Silica-Sand
Surface Material:	Flash Mount, with Protective Casing and Bolt Lock

Monitoring Well Designation: MW-01
Latitude: 42.9304808°
Longitude: -78.8957223°
Elevation: 577.70 Feet

BE3 CORP
BRYDGES ENGINEERING IN ENVIRONMENT & ENERGY
1270 Niagara Street
Buffalo, New York, 14213

Well Construction Log

Project:	Fedder Lofts	Drilling Company:	Nature's Way Contracting
Location:	57 Tonawanda Street	Drill Type:	6" Hollow Stem Auger
Client:	Eran Epstein	Driller:	Nate
Client Address:	57-71 Tonawanda Street Buffalo, NY 14211	Drill Date:	1/13/2020



LEGEND

- Cement-Bentonite Grout
- Bentonite Seal
- Silica Pack
- Static Water Level

Well Construction Properties

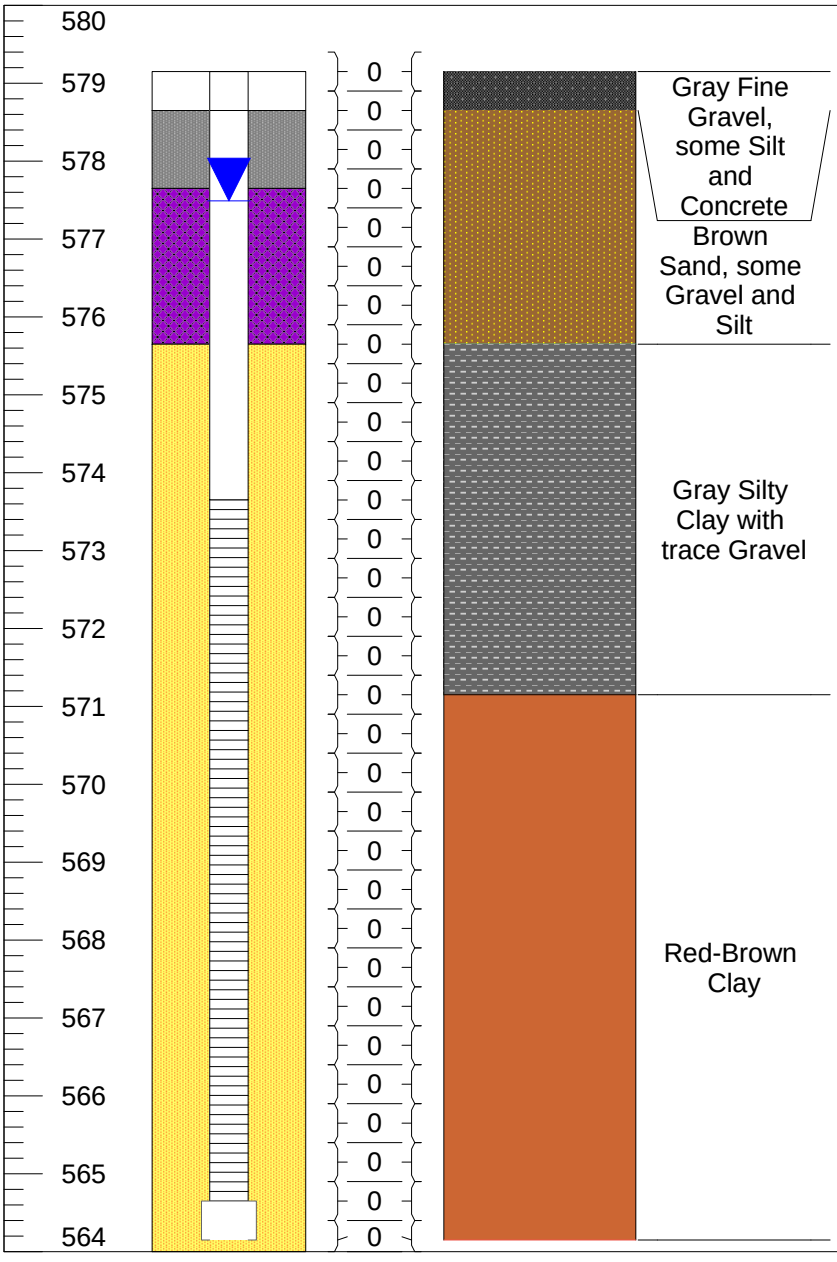
Material:	PVC
Diameter:	2"
Slot Count:	10
Slot Size:	2"
Seal Material:	Benotonite
Packing Material:	Silica-Sand
Surface Material:	Flash Mount, with Protective Casing and Bolt Lock

Monitoring Well Designation: MW-02
Latitude: 42.9308709°
Longitude: -78.8951537°
Elevation: 578.33 Feet

BE3 CORP
BRYDGES ENGINEERING IN ENVIRONMENT & ENERGY
1270 Niagara Street
Buffalo, New York, 14213

Well Construction Log

Project:	Fedder Lofts	Drilling Company:	Nature's Way Contracting
Location:	57 Tonawanda Street	Drill Type:	6" Hollow Stem Auger
Client:	Eran Epstein	Driller:	Nate
Client Address:	57-71 Tonawanda Street Buffalo, NY 14211	Drill Date:	1/13/2020



LEGEND

- Cement-Bentonite Grout
- Bentonite Seal
- Silica Pack
- Static Water Level

Well Construction Properties

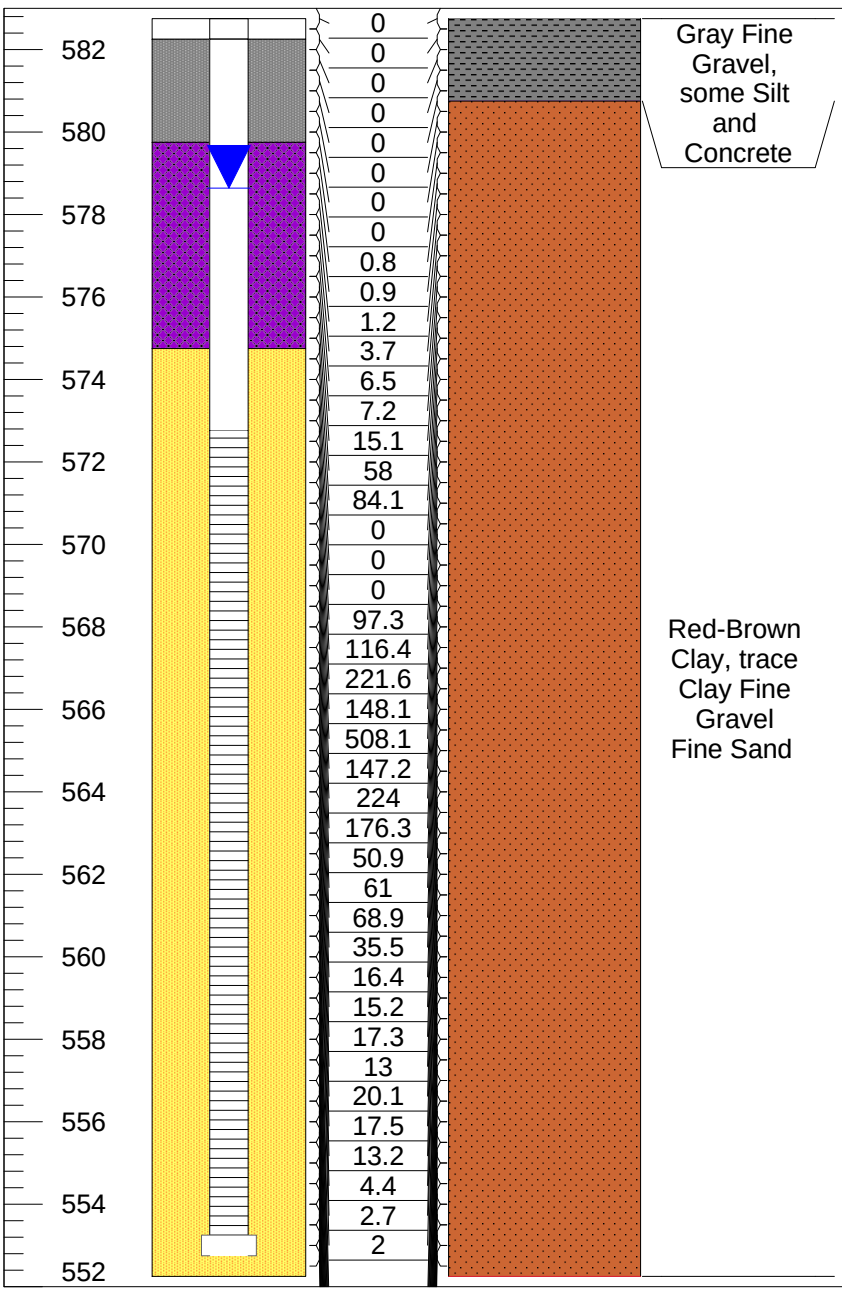
Material:	PVC
Diameter:	2"
Slot Count:	10
Slot Size:	2"
Seal Material:	Benotonite
Packing Material:	Silica-Sand
Surface Material:	Flash Mount, with Protective Casing and Bolt Lock

Monitoring Well Designatio: MW-03
Latitude: 42.9306862°
Longitude: -78.8958226°
Elevation: 579.15 Feet

BE3 CORP
BRYDGES ENGINEERING IN ENVIRONMENT & ENERGY
1270 Niagara Street
Buffalo, New York, 14213

Well Construction Log

Project:	Fedder Lofts	Drilling Company:	Nature's Way Contracting
Location:	57 Tonawanda Street	Drill Type:	6" Hollow Stem Auger
Client:	Eran Epstein	Driller:	Nate
Client Address:	57-71 Tonawanda Street Buffalo, NY 14211	Drill Date:	1/14/2020



LEGEND

- Cement-Bentonite Grout
- Bentonite Seal
- Silica Pack
- Static Water Level

Well Construction Properties

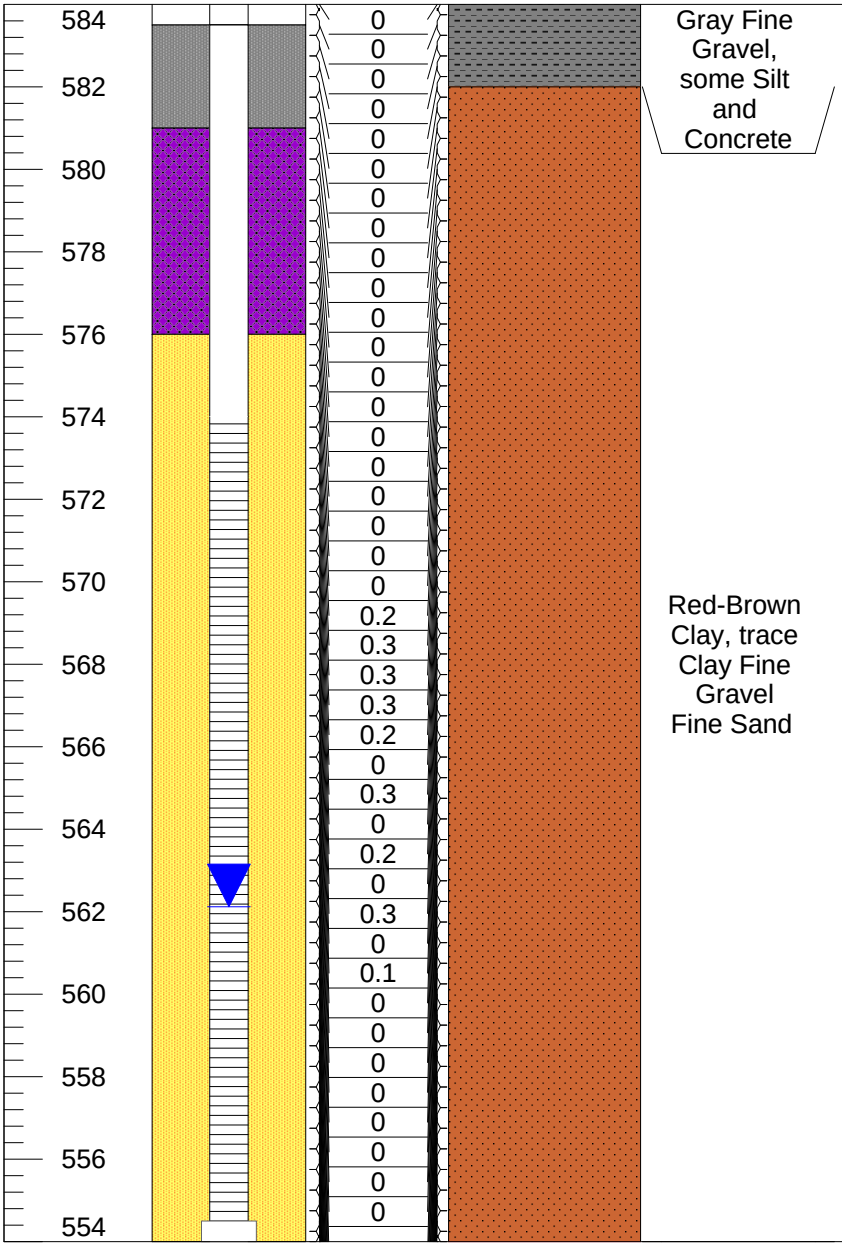
Material:	PVC
Diameter:	2"
Slot Count:	10
Slot Size:	2"
Seal Material:	Benotonite
Packing Material:	Silica-Sand
Surface Material:	Flash Mount, with Protective Casing and Bolt Lock

Monitoring Well Designation: MW-04
Latitude: 42.930726°
Longitude: -78.8971051°
Elevation: 582.75 Feet

BE3 CORP
BRYDGES ENGINEERING IN ENVIRONMENT & ENERGY
1270 Niagara Street
Buffalo, New York, 14213

Well Construction Log

Project:	Fedder Lofts	Drilling Company:	Nature's Way Contracting
Location:	57 Tonawanda Street	Drill Type:	6" Hollow Stem Auger
Client:	Eran Epstein	Driller:	Nate
Client Address:	57-71 Tonawanda Street Buffalo, NY 14211	Drill Date:	1/15/2020



LEGEND

- Cement-Bentonite Grout
- Bentonite Seal
- Silica Pack
- Static Water Level

Well Construction Properties

Material:	PVC
Diameter:	2"
Slot Count:	10
Slot Size:	2"
Seal Material:	Benotonite
Packing Material:	Silica-Sand
Surface Material:	Flash Mount, with Protective Casing and Bolt Lock

Monitoring Well Designation: MW-05
Latitude: 42.9311924°
Longitude: -78.8970434°
Elevation: 584.0 Feet

BE3 CORP
BRYDGES ENGINEERING IN ENVIRONMENT & ENERGY
1270 Niagara Street
Buffalo, New York, 14213

DRILLING SUMMARY

Geologist:
Steve Moeller

Drilling Company:
TREC Environmental

Driller:
Eric Hamman

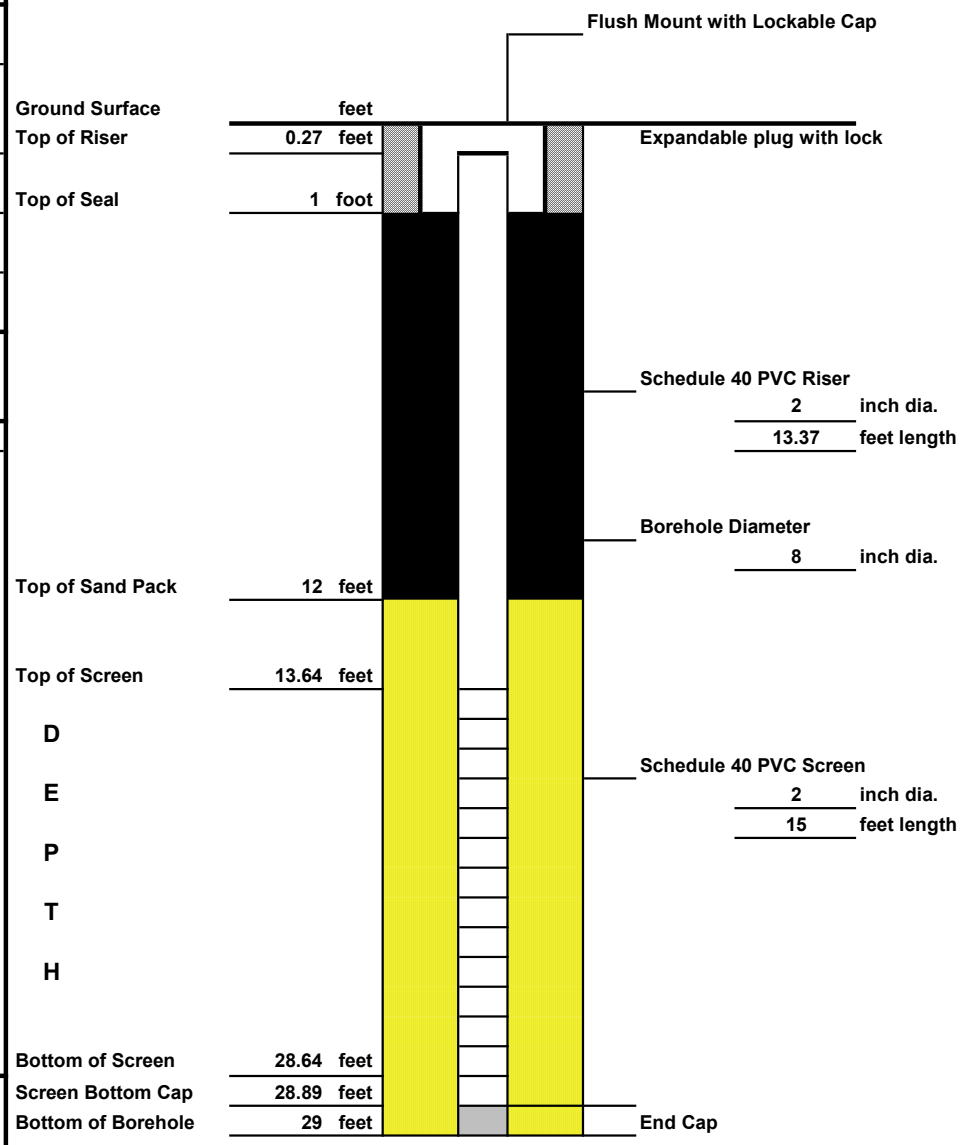
Rig Make/Model:
Geoprobe 6620 DT

Date:
September 24, 2020

GEOLOGIC LOG

Depth(ft.)	Description
	See log for soil boring SB-100.

WELL DESIGN



SURFACE COMPLETION

Surface: 8-inch diameter steel flush-mount with 12-inch skirt

RISER MATERIAL

Type: 2-inch ID Schedule 40 PVC threaded with O-ring seal

Setting: 0.27 - 13.64 feet bgs

SCREEN MATERIAL

Type: 2-inch ID Schedule 40 PVC, threaded with O-ring seal

Slot Size: 0.010" machine slotted

Setting: 13.64 - 28.64 feet bgs

FILTER MATERIAL

Type: Filpro #00N Silica Sand

Setting: 12 - 29 feet bgs

SEAL MATERIAL

Type: CETCO PureGold Medium Bentonite Chips

Setting: 1 - 12 feet bgs

COMMENTS:

Well is located west of the bike path in a grass strip adjacent to the 57-71 Tonawanda Street BCP Site, Site No. C915024. This well is at the base of the bike path ramp near an asphalt pad.

LEGEND

- Cement
- Bentonite Chip Seal
- Silica Sandpack

Client: NYSDEC

Location: Bike Path off West Ave. Buffalo, NY

Site Name: 31 Tonawanda Off Site
Site No.: C915299A

NEW YORK STATE OF OPPORTUNITY | Department of Environmental Conservation

MONITORING WELL CONSTRUCTION DETAILS

Well Number: MW-100

DRILLING SUMMARY

Geologist:
Steve Moeller

Drilling Company:
TREC Environmental

Driller:
Eric Hamman

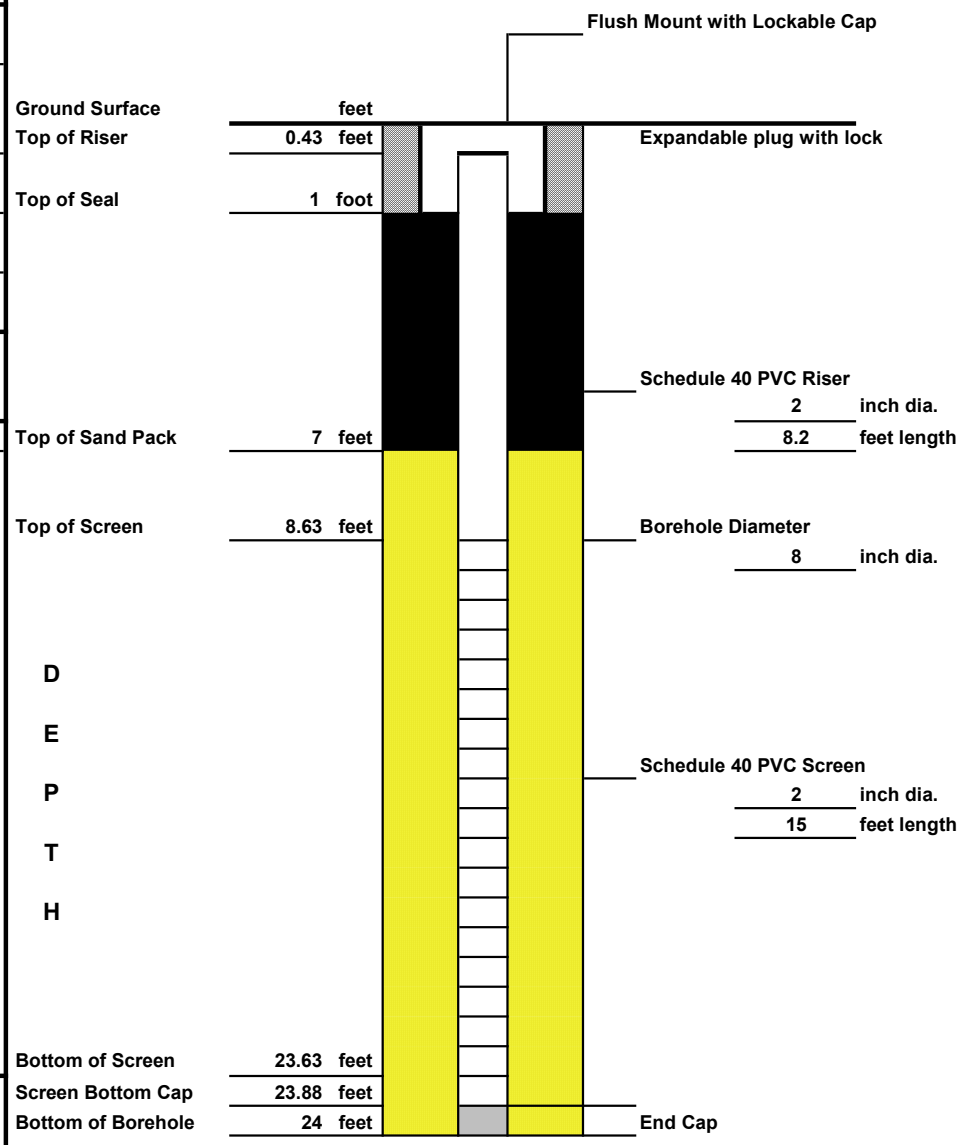
Rig Make/Model:
Geoprobe 6620 DT

Date:
September 23, 2020

GEOLOGIC LOG

Depth(ft.)	Description
	See log for soil boring SB-103.

WELL DESIGN



SURFACE COMPLETION
Surface: 8-inch diameter steel flush-mount with 12-inch skirt

RISER MATERIAL
Type: 2-inch ID Schedule 40 PVC threaded with O-ring seal
Setting: 0.43 - 8.63 feet bgs

SCREEN MATERIAL
Type: 2-inch ID Schedule 40 PVC, threaded with O-ring seal
Slot Size: 0.010" machine slotted
Setting: 8.63 - 23.63 feet bgs

FILTER MATERIAL
Type: Filpro #00N Silica Sand
Setting: 7 - 24 feet bgs

SEAL MATERIAL
Type: CETCO PureGold Medium Bentonite Chips
Setting: 1 - 7 feet bgs

COMMENTS:
Well is located west of the bike path in a grass strip adjacent to the 57-71 Tonawanda Street BCP Site, Site No. C915024.

LEGEND
- Cement
- Bentonite Chip Seal
- Silica Sandpack

Client: NYSDEC

Location: Bike Path off West Ave. Buffalo, NY

Site Name: 31 Tonawanda Off Site
Site No.: C915299A

NEW YORK STATE OF OPPORTUNITY Department of Environmental Conservation

MONITORING WELL CONSTRUCTION DETAILS

Well Number: MW-103

DRILLING SUMMARY

Geologist:
Steve Moeller

Drilling Company:
TREC Environmental

Driller:
Eric Hamman

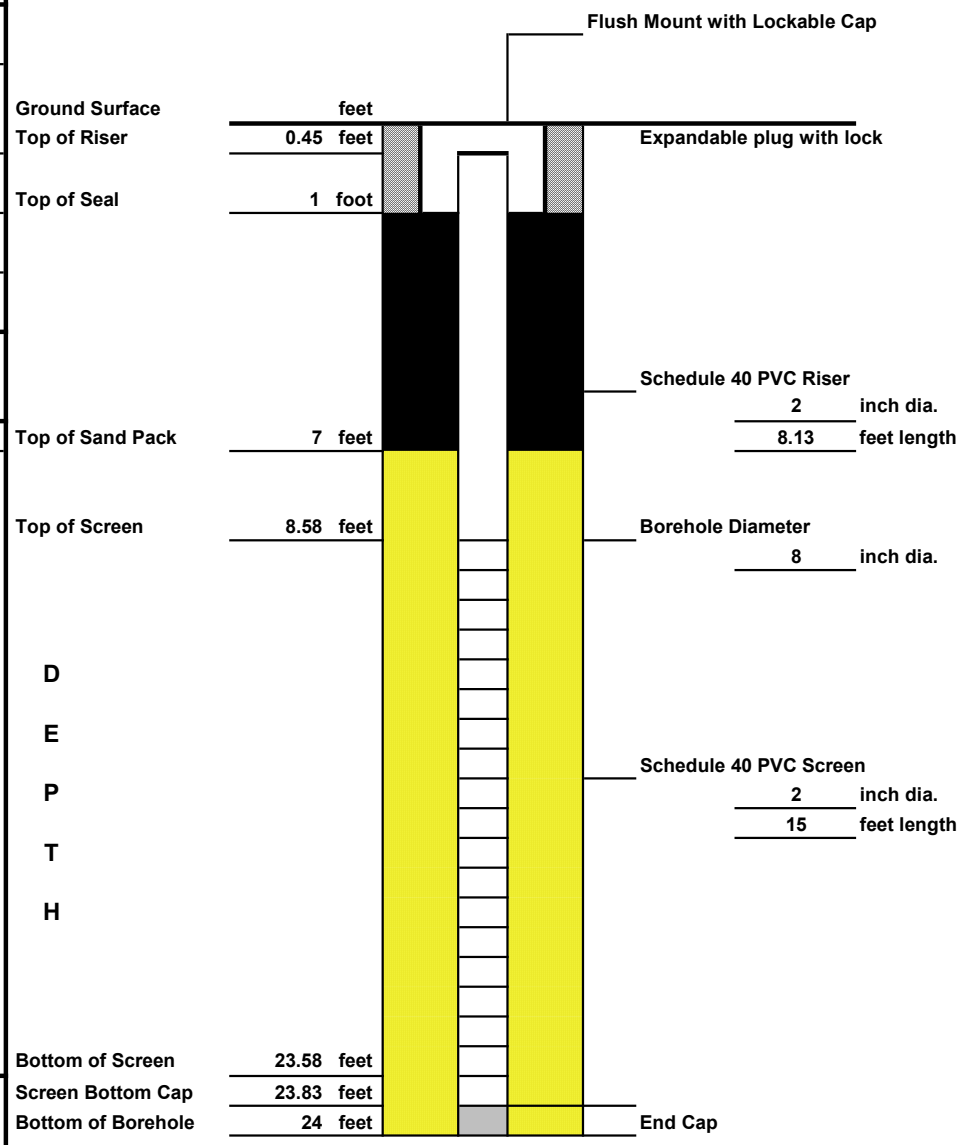
Rig Make/Model:
Geoprobe 6620 DT

Date:
September 23, 2020

GEOLOGIC LOG

Depth(ft.)	Description
	See log for soil boring SB-106.

WELL DESIGN



SURFACE COMPLETION

Surface: 8-inch diameter steel flush-mount with 12-inch skirt

RISER MATERIAL

Type: 2-inch ID Schedule 40 PVC threaded with O-ring seal

Setting: 0.45 - 8.58 feet bgs

SCREEN MATERIAL

Type: 2-inch ID Schedule 40 PVC, threaded with O-ring seal

Slot Size: 0.010" machine slotted

Setting: 8.58 - 23.58 feet bgs

FILTER MATERIAL

Type: Filpro #00N Silica Sand

Setting: 7 - 24 feet bgs

SEAL MATERIAL

Type: CETCO PureGold Medium Bentonite Chips

Setting: 1 - 7 feet bgs

COMMENTS:

Well is located west of the bike path in a grass strip adjacent to the former Pratt & Lambert Paint Company, Site No. 915251, at 75 Tonawanda Street. The well is close to where the Scajaquada Expressway crosses the bike path.

LEGEND

Cement

Bentonite Chip Seal

Silica Sandpack

Client: NYSDEC

Location: Bike Path off West Ave. Buffalo, NY

Site Name: 31 Tonawanda Off Site
Site No.: C915299A

NEW YORK STATE OF OPPORTUNITY Department of Environmental Conservation

MONITORING WELL CONSTRUCTION DETAILS

Well Number: MW-106

DRILLING SUMMARY

Geologist:
Steve Moeller

Drilling Company:
TREC Environmental

Driller:
Eric Hamman

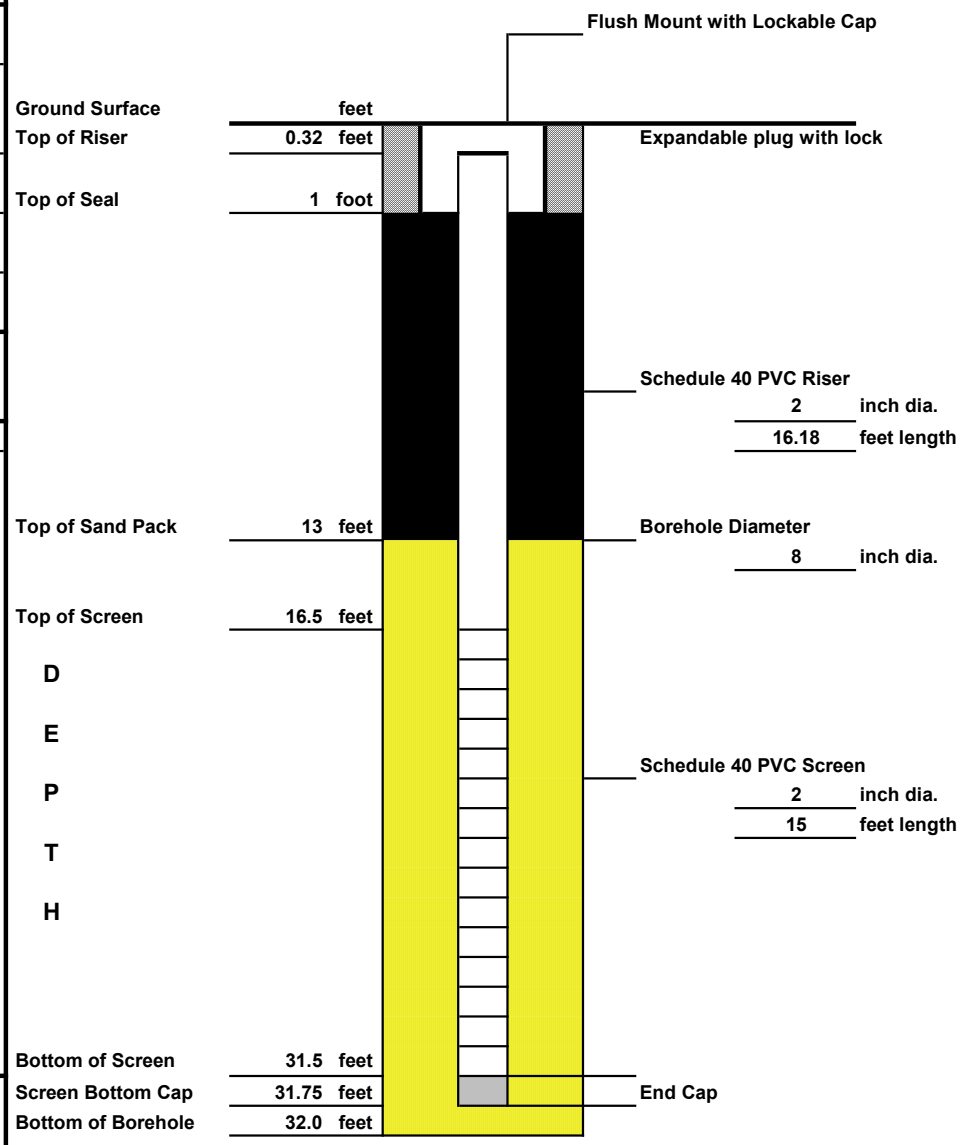
Rig Make/Model:
Geoprobe 6620 DT

Date:
September 15, 2020

GEOLOGIC LOG

Depth(ft.)	Description
	See log for soil boring 1675-SB-1.

WELL DESIGN



SURFACE COMPLETION

Surface: 8-inch diameter steel flush-mount with 12-inch skirt

RISER MATERIAL

Type: 2-inch ID Schedule 40 PVC threaded with O-ring seal

Setting: 0.32 - 16.5 feet bgs

SCREEN MATERIAL

Type: 2-inch ID Schedule 40 PVC, threaded with O-ring seal

Slot Size: 0.010" machine slotted

Setting: 16.5 - 31.5 feet bgs

FILTER MATERIAL

Type: Filpro #00N Silica Sand

Setting: 13 -32 feet bgs

SEAL MATERIAL

Type: CETCO PureGold Medium Bentonite Chips

Setting: 1 - 13 feet bgs

COMMENTS:

Well is located in front of the easternmost portion of the building in a grass strip between the building and sidewalk.

LEGEND

Cement

Bentonite Chip Seal

Silica Sandpack

Client: NYSDEC

Location: 1675 Niagara Street Buffalo, NY

Site Name: 1660 Niagara Street
Site No.: C915311

NEW YORK STATE OF OPPORTUNITY Department of Environmental Conservation

MONITORING WELL CONSTRUCTION DETAILS

Well Number: 1675-MW-1

DRILLING SUMMARY

Geologist:
Steve Moeller

Drilling Company:
TREC Environmental

Driller:
Eric Hamman

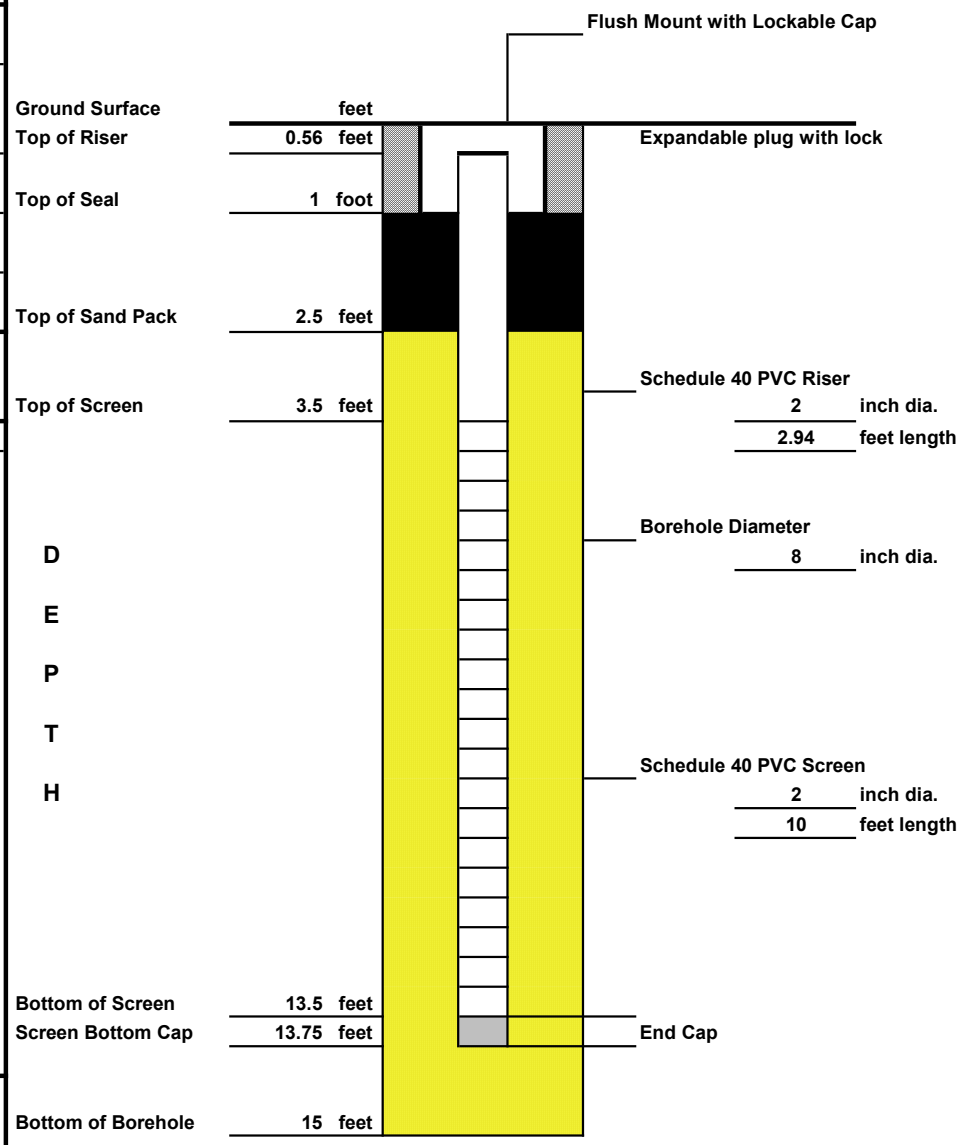
Rig Make/Model:
Geoprobe 6620 DT

Date:
September 15, 2020

GEOLOGIC LOG

Depth(ft.)	Description
	See log for soil boring 1675-SB-2.

WELL DESIGN



SURFACE COMPLETION

Surface: 8-inch diameter steel flush-mount with 12-inch skirt

RISER MATERIAL

Type: 2-inch ID Schedule 40 PVC threaded with O-ring seal

Setting: 0.56 - 3.5 feet bgs

SCREEN MATERIAL

Type: 2-inch ID Schedule 40 PVC, threaded with O-ring seal

Slot Size: 0.010" machine slotted

Setting: 3.5 - 13.5 feet bgs

FILTER MATERIAL

Type: Filpro #00N Silica Sand

Setting: 2.5 - 15 feet bgs

SEAL MATERIAL

Type: CETCO PureGold Medium Bentonite Chips

Setting: 1 - 2.5 feet bgs

COMMENTS:

Well is located in asphalt parking lot just south of easternmost overhead door.

LEGEND

- Cement
- Bentonite Chip Seal
- Silica Sandpack

Client: NYSDEC

Location: 1675 Niagara Street Buffalo, NY

Site Name: 1660 Niagara Street
Site No.: C915311

NEW YORK STATE OF OPPORTUNITY Department of Environmental Conservation

MONITORING WELL CONSTRUCTION DETAILS

Well Number: 1675-MW-2

DRILLING SUMMARY

Geologist:
Steve Moeller

Drilling Company:
TREC Environmental

Driller:
Eric Hamman

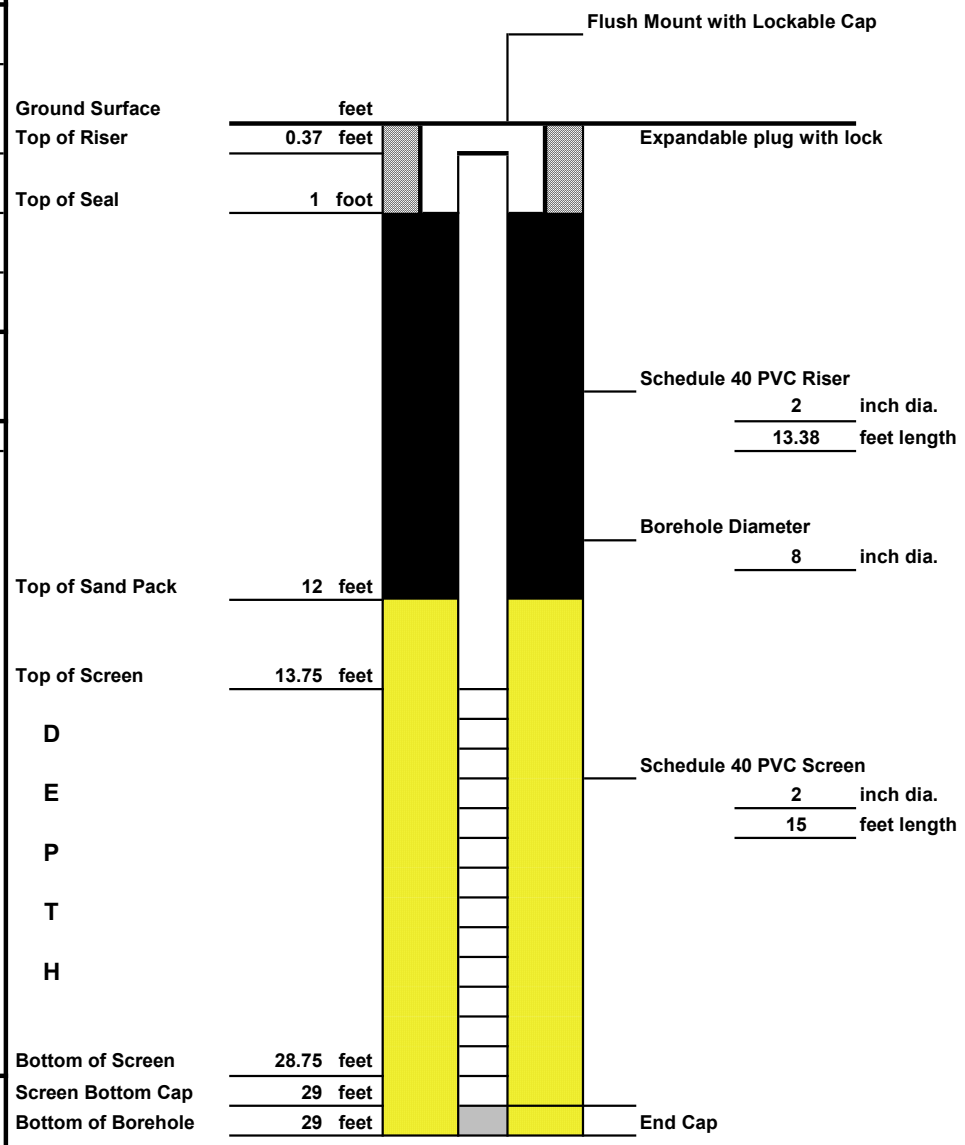
Rig Make/Model:
Geoprobe 6620 DT

Date:
September 22, 2020

GEOLOGIC LOG

Depth(ft.)	Description
	See log for soil boring SB-3R.

WELL DESIGN



SURFACE COMPLETION

Surface: 8-inch diameter steel flush-mount with 12-inch skirt

RISER MATERIAL

Type: 2-inch ID Schedule 40 PVC threaded with O-ring seal

Setting: 0.27 - 13.75 feet bgs

SCREEN MATERIAL

Type: 2-inch ID Schedule 40 PVC, threaded with O-ring seal

Slot Size: 0.010" machine slotted

Setting: 13.75 - 28.75 feet bgs

FILTER MATERIAL

Type: Filpro #00N Silica Sand

Setting: 12 - 29 feet bgs

SEAL MATERIAL

Type: CETCO PureGold Medium Bentonite Chips

Setting: 1 - 12 feet bgs

COMMENTS:

Well is a replacement for original well MW-3, and is in the north-central portion of the site.

LEGEND

Cement

Bentonite Chip Seal

Silica Sandpack

Client: NYSDEC

Location: 1660 Niagara Street Buffalo, NY

Site Name: 1660 Niagara Street
Site No.: C915311

NEW YORK STATE OF OPPORTUNITY | Department of Environmental Conservation

MONITORING WELL CONSTRUCTION DETAILS

Well Number: MW-3R

DRILLING SUMMARY

Geologist:
Steve Moeller

Drilling Company:
TREC Environmental

Driller:
Eric Hamman

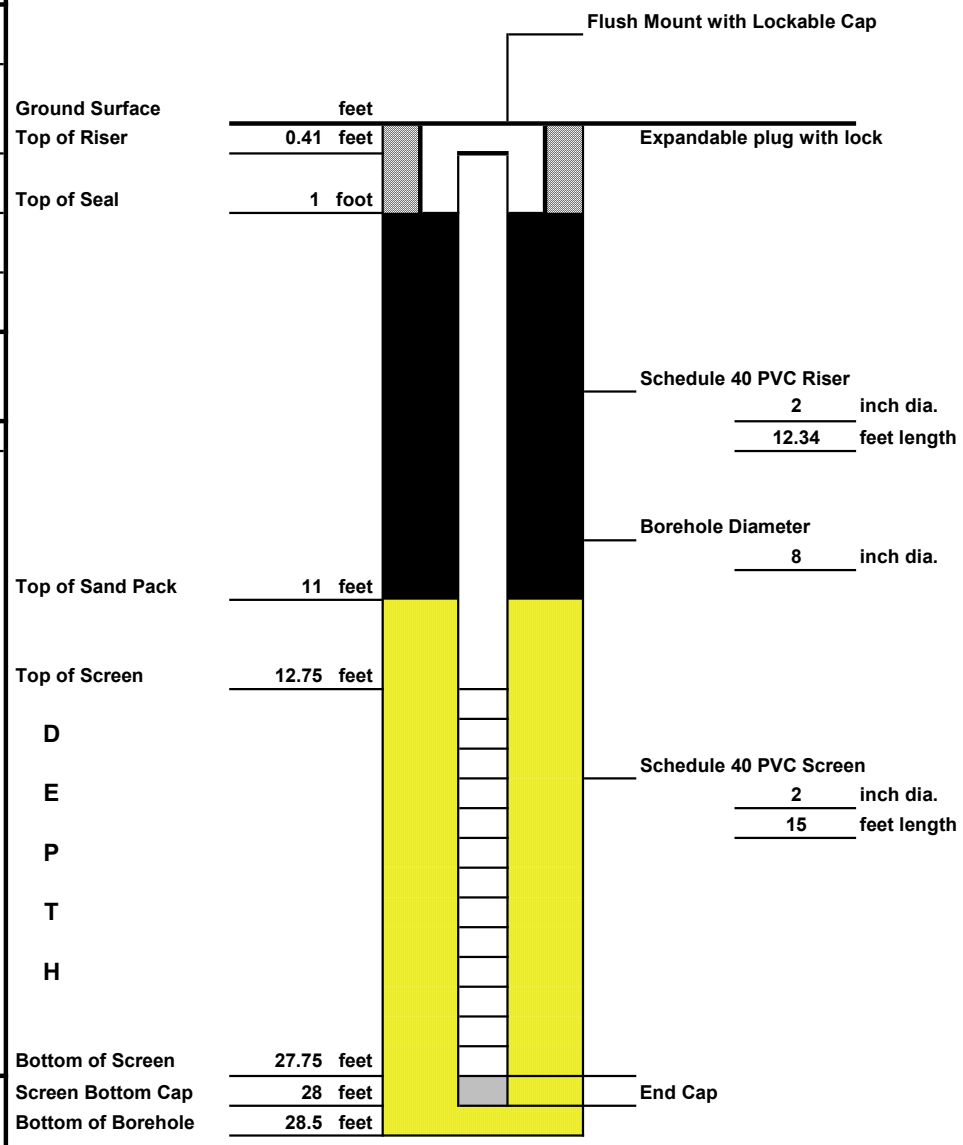
Rig Make/Model:
Geoprobe 6620 DT

Date:
September 21, 2020

GEOLOGIC LOG

Depth(ft.)	Description
	See log for soil boring SB-3R.

WELL DESIGN



SURFACE COMPLETION

Surface: 8-inch diameter steel flush-mount with 12-inch skirt

RISER MATERIAL

Type: 2-inch ID Schedule 40 PVC threaded with O-ring seal

Setting: 0.41 - 12.75 feet bgs

SCREEN MATERIAL

Type: 2-inch ID Schedule 40 PVC, threaded with O-ring seal

Slot Size: 0.010" machine slotted

Setting: 12.75 - 27.75 feet bgs

FILTER MATERIAL

Type: Filpro #00N Silica Sand

Setting: 12 - 28.5 feet bgs

SEAL MATERIAL

Type: CETCO PureGold Medium Bentonite Chips

Setting: 1 - 11 feet bgs

COMMENTS:

Well is a replacement for original well MW-5, and is in the northern portion of the site about 15 feet west of the site divider fence.

LEGEND

- Cement
- Bentonite Chip Seal
- Silica Sandpack

Client: NYSDEC

Location: 1660 Niagara Street Buffalo, NY

Site Name: 1660 Niagara Street
Site No.: C915311

NEW YORK STATE OF OPPORTUNITY Department of Environmental Conservation

MONITORING WELL CONSTRUCTION DETAILS

Well Number: MW-5R

DRILLING SUMMARY

Geologist:
Steve Moeller

Drilling Company:
TREC Environmental

Driller:
Eric Hamman

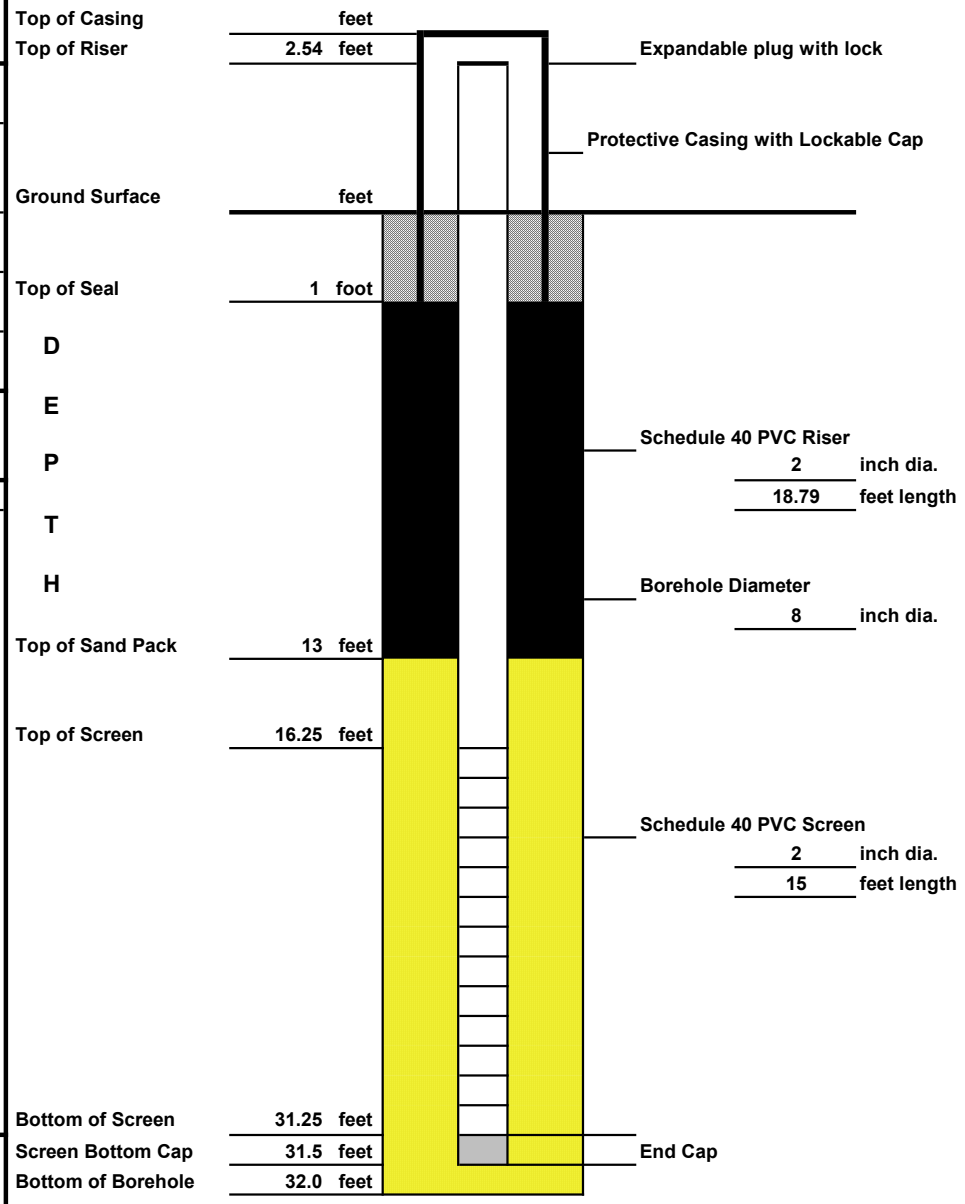
Rig Make/Model:
Geoprobe 6620 DT

Date:
September 17, 2020

GEOLOGIC LOG

Depth(ft.)	Description

WELL DESIGN



SURFACE COMPLETION

Surface: 8-inch diameter steel flush-mount with 12-inch skirt

RISER MATERIAL

Type: 2-inch ID Schedule 40 PVC threaded with O-ring seal

Setting: -2.54 - 16.25 feet bgs

SCREEN MATERIAL

Type: 2-inch ID Schedule 40 PVC, threaded with O-ring seal

Slot Size: 0.010" machine slotted

Setting: 16.25 - 31.5 feet bgs

FILTER MATERIAL

Type: Filpro #00N Silica Sand

Setting: 13 - 32 feet bgs

SEAL MATERIAL

Type: CETCO PureGold Medium Bentonite Chips

Setting: 1 - 13 feet bgs

COMMENTS:

Well is located in the southeastern portion of the site about 15 feet west of the the BSA pump house building.

LEGEND

- Cement/Bentonite Grout
- Bentonite Seal
- Silica Sandpack

Client: NYSDEC

Location: 1660 Niagara Street Buffalo, NY

Site Name: 1660 Niagara Street
Site No.: C915311

NEW YORK STATE OF OPPORTUNITY | Department of Environmental Conservation

MONITORING WELL CONSTRUCTION DETAILS

Well Number: MW-8

BH-31
BH-64
BH-32
BH-65
BH-33
190

SM 282 E 12/02

STATE OF NEW YORK
DEPARTMENT OF TRANSPORTATION
GEOTECHNICAL ENGINEERING BUREAU
SUBSURFACE EXPLORATION LOG

UNOFFICIAL LOG

PSN 253 BORNUM 31
REGION 5
COUNTY ERIE
PIN
PROJECT SCAJAQUADA CREEK EXPRESSWAY DELAWARE PARK
SCALED COORDINATES (N) 1,067,466.670 (E) 1,063,681.400 **DATUM** NAD83
DATE START 12-NOV-1956 **DATE FINISH** 20-NOV-1956

HOLE DH-B-C-3
LINE Baseline
STA 454+61
OFFSET 340 ft LEFT
SURF. ELEV. 571.11
DEPTH TO WATER SEE NOTE

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18 **in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18 **in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	0.0							(0.00) Water - Black Rock Channel
WOH	5.0	J1	WOH	WOH	WOH			(4.00) Black SILT , Some Sand And Organic Muck (W-NPL)
WOH WOH WOH WOH WOH	10.0	J2	WOH	WOH	WOH			
WOH WOH WOH WOH WOH	15.0	J3	7	8	9			(15.00) Red SAND , Trace Silt And Clay (W-LPL)
24 35 35 22 19	20.0							(18.00) Red SILT , Some Clay, Trace Gravel And Fine Sand (M-PL)
27 30 40 18		J4	5	6	8			(23.00) Red CLAY , Some Silt (M-PL)
18	25.0	J5	1	1				

The subsurface information shown here was obtained for design and estimate purposes. It is made available so that users may have access to the same information available to the State. It is presented in good faith. By the nature of the exploration process, the information represents only a small fraction of the total volume of the material at the site. Interpolation between data samples may not be indicative of the actual material encountered.

CONTRACT _____ **CONTRACTOR** Westville Engineering

DRILL RIG OPERATOR
SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER UNOFFICIAL LOG
DATE APPROVED
RESIDENT ENGINEER
STRUCTURE NAME Route 198 Ramp over I-190
B.I.N. 103989C

SM 282 E 12/02

STATE OF NEW YORK
DEPARTMENT OF TRANSPORTATION
GEOTECHNICAL ENGINEERING BUREAU
SUBSURFACE EXPLORATION LOG

UNOFFICIAL LOG

PSN 253 BORNUM 31
REGION 5
COUNTY ERIE
PIN
PROJECT SCAJAQUADA CREEK EXPRESSWAY DELAWARE PARK
SCALED COORDINATES (N) 1,067,466.670 (E) 1,063,681.400 **DATUM** NAD83
DATE START 12-NOV-1956 **DATE FINISH** 20-NOV-1956

HOLE DH-B-C-3
LINE Baseline
STA 454+61
OFFSET 340 ft LEFT
SURF. ELEV. 571.11
DEPTH TO WATER SEE NOTE

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18 **in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18 **in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	25.0				1			(25.00) Red CLAY , Some Silt (M-PL)
19								
20								
16								
16								
		J6	1					
15	30.0			1				
					1			
19								
23								
21								
33								
		J7	1					
21	35.0			1				
					2			
35								
41								
41								
54								(38.00) Red SILT , Some Clay, Trace Gravel And Fine Sand (M-PL)
		J8	1					
31	40.0			2				
					2			
37								
42								
71								
71								
		J9	2					
55	45.0			2				
					3			
61								
78								
85								
80								
		J10	2					
65	50.0			3				

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CONTRACT _____ **CONTRACTOR** Westville Engineering

DRILL RIG OPERATOR
SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER UNOFFICIAL LOG
DATE APPROVED
RESIDENT ENGINEER
STRUCTURE NAME Route 198 Ramp over I-190
B.I.N. 103989C

SM 282 E 12/02

STATE OF NEW YORK
DEPARTMENT OF TRANSPORTATION
GEOTECHNICAL ENGINEERING BUREAU
SUBSURFACE EXPLORATION LOG

UNOFFICIAL LOG

PSN 253 BORNUM 31
REGION 5
COUNTY ERIE
PIN
PROJECT SCAJAQUADA CREEK EXPRESSWAY DELAWARE PARK
SCALED COORDINATES (N) 1,067,466.670 (E) 1,063,681.400 **DATUM** NAD83
DATE START 12-NOV-1956 **DATE FINISH** 20-NOV-1956

HOLE DH-B-C-3
LINE Baseline
STA 454+61
OFFSET 340 ft LEFT
SURF. ELEV. 571.11
DEPTH TO WATER SEE NOTE

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18	**in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18	**in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	50.0				3			(50.00) Red SILT , Some Clay, Trace Gravel And Fine Sand (M-PL)
85								
96								
100								
110								
		J11	4					
115	55.0			7	11			
195								
165								
177								
245								(58.00) Red CLAY , Some Silt (M-PL)
		J12	2					
195	60.0			5	5			
219								
221								
211								(62.00) Red SILT , Some Clay (M-PL)
213								
		J13	2					
145	65.0			4	4			
128								
115								
185								(67.00) Gray SILT , Some Gravel And Sand, Trace Clay (D-LW)
245								
		J14	13					
115	70.0			31	55			
250								
550								
410								
340								
		R1						(73.66) Run #1 Drilled from 73'8" To 78'8" LIMESTONE , Fractured And Broken
	75.0							REC. 31" 51.7% 25

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CONTRACT _____ **CONTRACTOR** Westville Engineering

DRILL RIG OPERATOR
SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER **UNOFFICIAL LOG**
DATE APPROVED
RESIDENT ENGINEER
STRUCTURE NAME **B.I.N.** 103989C
Route 198 Ramp over I-190

SM 282 E 12/02

PSN 253 BORNUM 31
REGION 5
COUNTY ERIE
PIN
PROJECT SCAJAQUADA CREEK EXPRESSWAY DELAWARE PARK
SCALED COORDINATES (N) 1,067,466.670 (E) 1,063,681.400 **DATUM** NAD83

STATE OF NEW YORK
DEPARTMENT OF TRANSPORTATION
GEOTECHNICAL ENGINEERING BUREAU
SUBSURFACE EXPLORATION LOG

UNOFFICIAL LOG

HOLE DH-B-C-3
LINE Baseline
STA 454+61
OFFSET 340 ft LEFT
SURF. ELEV. 571.11
DEPTH TO WATER SEE NOTE

DATE START 12-NOV-1956 **DATE FINISH** 20-NOV-1956

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18 **in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18 **in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	75.0							
		R2						(78.66) Run #2 Drilled from 78'8" To 83'8" LIMESTONE , Fractured And Broken REC. 35" 58.3% 38
	80.0							

BOTTOM OF HOLE AT 83.66 ft

Ground elevation is the surface of the Black Rock Channel.
Groundwater elevation is the surface of the Black Rock Channel.
Artesian condition encountered at 83.67 feet, flow of approximately 1 gal/min, continued until all casing was pulled.

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CONTRACT _____ **CONTRACTOR** Westville Engineering

DRILL RIG OPERATOR
SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER **UNOFFICIAL LOG**
DATE APPROVED
RESIDENT ENGINEER
STRUCTURE NAME Route 198 Ramp over I-190 **B.I.N.** 103989C

SM 282 E 12/02

STATE OF NEW YORK
DEPARTMENT OF TRANSPORTATION
GEOTECHNICAL ENGINEERING BUREAU
SUBSURFACE EXPLORATION LOG

UNOFFICIAL LOG

PSN 253 BORNUM 32
REGION 5
COUNTY ERIE
PIN
PROJECT SCAJAQUADA CREEK EXPRESSWAY DELAWARE PARK
SCALED COORDINATES (N) 1,067,403.730 (E) 1,063,686.550 **DATUM** NAD83

HOLE DH-B-C-4
LINE Baseline
STA 454+09
OFFSET 332 ft LEFT
SURF. ELEV. 571.82
DEPTH TO WATER SEE NOTE

DATE START 07-NOV-1956 **DATE FINISH** 12-NOV-1956

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18 **in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18 **in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	0.0							(0.00) Water - Black Rock Channel
WOH		J1	WOH	WOH				(3.50) Black SILT , Some Clay And Organic Matter, Trace Sand (W-PL)
WOH	5.0				WOH			
WOH								
1								
1								
1								
1	10.0							
1		J2	WOH	WOH				
1					WOH			
3								
2								
4	15.0							
17		J3	3	3				(15.00) Red CLAY , Some Silt (M-PL)
16					10			
24								
33								
37	20.0							
29		J4	3	3				
33					4			
33								
26								
22	25.0							

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CONTRACT _____ **CONTRACTOR** Westville Engineering

DRILL RIG OPERATOR
SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER UNOFFICIAL LOG
DATE APPROVED
RESIDENT ENGINEER
STRUCTURE NAME Route 198 Ramp over I-190
B.I.N. 103989C

SM 282 E 12/02

STATE OF NEW YORK
DEPARTMENT OF TRANSPORTATION
GEOTECHNICAL ENGINEERING BUREAU
SUBSURFACE EXPLORATION LOG

UNOFFICIAL LOG

PSN 253 BORNUM 32
REGION 5
COUNTY ERIE
PIN
PROJECT SCAJAQUADA CREEK EXPRESSWAY DELAWARE PARK
SCALED COORDINATES (N) 1,067,403.730 (E) 1,063,686.550 **DATUM** NAD83
DATE START 07-NOV-1956 **DATE FINISH** 12-NOV-1956

HOLE DH-B-C-4
LINE Baseline
STA 454+09
OFFSET 332 ft LEFT
SURF. ELEV. 571.82
DEPTH TO WATER SEE NOTE

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18	**in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18	**in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	25.0	J5	1	2	4			(25.00) Red CLAY , Some Silt (M-PL)
14								
13								
								(27.00) Red SILT , Some Clay, Trace Gravel And Fine Sand (M-PL)
23								
22								
26	30.0							
		J6	1	1	2			
21								
28								
32								
32								
36	35.0							
		J7	2	2	3			
25								
31								
36								
36								
37	40.0							
		J8	2	3	4			
32								
32								
37								
								(43.00) Red CLAY , Some Silt (M-PL)
43								
48	45.0							
		J9	2	4	5			
55								
70								
67								
78								
90	50.0							

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CONTRACT _____ **CONTRACTOR** Westville Engineering

DRILL RIG OPERATOR
SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER UNOFFICIAL LOG
DATE APPROVED
RESIDENT ENGINEER
STRUCTURE NAME Route 198 Ramp over I-190
B.I.N. 103989C

SM 282 E 12/02

STATE OF NEW YORK
DEPARTMENT OF TRANSPORTATION
GEOTECHNICAL ENGINEERING BUREAU
SUBSURFACE EXPLORATION LOG

UNOFFICIAL LOG

PSN 253 BORNUM 32
REGION 5
COUNTY ERIE
PIN
PROJECT SCAJAQUADA CREEK EXPRESSWAY DELAWARE PARK
SCALED COORDINATES (N) 1,067,403.730 (E) 1,063,686.550 **DATUM** NAD83
DATE START 07-NOV-1956 **DATE FINISH** 12-NOV-1956

HOLE DH-B-C-4
LINE Baseline
STA 454+09
OFFSET 332 ft LEFT
SURF. ELEV. 571.82
DEPTH TO WATER SEE NOTE

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18	**in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18	**in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	50.0	J10	2	4	4			(50.00) Red SILT , Some Clay, Trace Gravel And Fine Sand (M-PL)
40								
44								
44								
83								
		J11	3	5	5			
81	55.0							
85		J11A	3	4	6			
87								
109								
170								
		J12	3	6	5			
105	60.0							
115		J12A	3	4	4			
181								
185								
145								(63.00) Red CLAY , Some Silt (M-PL)
		J13	2	3	3			
115	65.0							
136								
138								
175								
182								
		J14	3	4	5			
195	70.0							
173								
239								(71.00) Gray SAND , Some Gravel And Broken Limestone, Trace Silt (D-NW)
200								
205								
		J15	13	17				
145	75.0							

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SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER UNOFFICIAL LOG
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RESIDENT ENGINEER
STRUCTURE NAME Route 198 Ramp over I-190
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SCALED COORDINATES (N) 1,067,403.730 (E) 1,063,686.550 **DATUM** NAD83
DATE START 07-NOV-1956 **DATE FINISH** 12-NOV-1956

HOLE DH-B-C-4
LINE Baseline
STA 454+09
OFFSET 332 ft LEFT
SURF. ELEV. 571.82
DEPTH TO WATER SEE NOTE

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18 **in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18 **in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	75.0	R1			17			(75.00) Gray SAND , Some Gravel And Broken Limestone, Trace Silt (D-NW)
185								(75.08) Run #1 Drilled from 75'1" To 80'1" LIMESTONE , Fractured And Broken REC. 23" 38.3% 34
	80.0	R2						(80.08) Run #2 Drilled from 80'1" To 85'1" LIMESTONE , Fractured And Broken REC. 59" 98.3% 47
	85.0							BOTTOM OF HOLE AT 85.08 ft

Ground elevation is the surface of the Black Rock Channel.
Groundwater elevation is the surface of the Black Rock Channel.
No recovery for samples 11, 12 and 12A.

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PSN 253 BORNUM 33
REGION 5
COUNTY ERIE
PIN
PROJECT SCAJAQUADA CREEK EXPRESSWAY DELAWARE PARK
SCALED COORDINATES (N) 1,067,254.590 (E) 1,063,727.820 **DATUM** NAD83
DATE START 20-NOV-1956 **DATE FINISH** 26-NOV-1956

HOLE DH-B-C-6
LINE Baseline
STA 452+97
OFFSET 288 ft LEFT
SURF. ELEV. 575.29
DEPTH TO WATER SEE NOTE

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18 **in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18 **in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	0.0							(0.00) Black SAND , Some Gravel, Cinders And Rubble - Fill Material (D-NW)
8								
6								
3								
3								
4	5.0							
		J1	5					
141				101				
19					200			
								(7.00) Gray SILT , Some Sand And Organic Matter, Trace Gravel (W-NPL)
5								Muck Seepage From Black Rock Channel
7								
5	10.0							
		J2	1					
3				2				
5					2			
5								
5								
105	15.0							
		J3	WOH					
21				WOH				
16					WOH			
26								
21								
								(19.00) Red CLAY , Some Silt (M-PL)
26	20.0							
		J4	2					
22				3				
28					5			
29								
31								
								(24.00) Red SILT , Some Clay, Trace Gravel And Fine Sand (M-PL)
39	25.0							

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SCALED COORDINATES (N) 1,067,254.590 (E) 1,063,727.820 **DATUM** NAD83

HOLE DH-B-C-6
LINE Baseline
STA 452+97
OFFSET 288 ft LEFT
SURF. ELEV. 575.29
DEPTH TO WATER SEE NOTE

DATE START 20-NOV-1956 **DATE FINISH** 26-NOV-1956

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18 **in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18 **in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	25.0	J5	5	7	42			(25.00) Red SILT , Some Clay, Trace Gravel And Fine Sand (M-PL)
37								
140								
								(27.00) Red CLAY , Some Silt (M-PL)
45								
46								
57	30.0							
		J6	8	7	9			
44								
46								
58								
62								
59	35.0							
		J7	4	4	7			
61								
69								
								(37.00) Red SILT , Some Clay, Trace Gravel And Fine Sand (M-PL)
80								
82								
82	40.0							
		J8	7	8	9			
92								
99								
81								
83								
74	45.0							
		J9	6	8	11			
57								
65								
76								
79								
81	50.0							

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HOLE DH-B-C-6
LINE Baseline
STA 452+97
OFFSET 288 ft LEFT
SURF. ELEV. 575.29
DEPTH TO WATER SEE NOTE

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18	**in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18	**in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	50.0	J10	4	4	5			(50.00) Red SILT , Some Clay, Trace Gravel And Fine Sand (M-PL)
69								
88		J10A	3	5	6			
97								
117								
136	55.0							
		J11	5	6	7			
101								
105								
123								(57.00) Red CLAY , Some Silt (M-PL)
128								
122	60.0							
115		J12	4	4	7			
120								
129								
152								
155	65.0							
109		J13	3	4	7			
107								
119								
123								
127	70.0							
166		J14	6	13	52			(70.00) Red CLAY , Some Silt, Trace Gravel (M-PL)
473								
		R1						(72.83) Run #1 Drilled from 72'10" To 75'1" LIMESTONE , Fractured And Broken REC. 13" 48.1% 8
	75.0							

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SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER UNOFFICIAL LOG
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RESIDENT ENGINEER
STRUCTURE NAME Route 198 Ramp over I-190
B.I.N. 103989C

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SCALED COORDINATES (N) 1,067,254.590 (E) 1,063,727.820 **DATUM** NAD83

HOLE DH-B-C-6
LINE Baseline
STA 452+97
OFFSET 288 ft LEFT
SURF. ELEV. 575.29
DEPTH TO WATER SEE NOTE

DATE START 20-NOV-1956 **DATE FINISH** 26-NOV-1956

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18 **in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18 **in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	75.0							(75.08) Red SILT , Some Gravel And Clay (M-PL)
		R2						(76.33) Run #2 Drilled from 76'4" To 81'4" LIMESTONE , Fractured And Broken REC. 45" 75% 34
	80.0							

BOTTOM OF HOLE AT 81.33 ft

Insufficient recovery for sample 10.
Lost water while coring from 74.67 to 81.33 feet.

DATE	TIME	DEPTH ft			ARTESIAN HEAD HEIGHT ABOVE GROUND	FILLED WITH WATER AT END OF DAY
		HOLE	CASING	WATER		
26-Nov-56	15:00	81.33	72.00	7.00		

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SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER **UNOFFICIAL LOG**
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STRUCTURE NAME Route 198 Ramp over I-190 **B.I.N.** 103989C

SM 282 E 12/02

PSN 253 BORNUM 64
REGION 5
COUNTY ERIE
PIN
PROJECT SCAJAQUADA CREEK EXPRESSWAY DELAWARE PARK

STATE OF NEW YORK
DEPARTMENT OF TRANSPORTATION
GEOTECHNICAL ENGINEERING BUREAU
SUBSURFACE EXPLORATION LOG

UNOFFICIAL LOG

HOLE PH-B-F-2
LINE Baseline
STA 455+25
OFFSET 182 ft LEFT
SURF. ELEV. 585.87
DEPTH TO WATER SEE NOTE

SCALED COORDINATES (N) 1,067,482.060 (E) 1,063,852.640 **DATUM** NAD83

DATE START 12-NOV-1956 **DATE FINISH** 13-NOV-1956

CASING O. D. 2 7/8 in **I. D.** 2 3/8 in **WT OF HAMMER-CASING** 300 lb **HAMMER FALL-CASING** 18 in
SAMPLER O. D. in **I. D.** in **WT OF HAMMER-SAMPLER** lb **HAMMER FALL-SAMPLER** in

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	0.0							(0.00) Cased Probing - No Samples Taken
55								
29								
21								
18								
11	5.0							
10								
6								
9								
14								
25	10.0							
27								
35								
31								
30								
27	15.0							
37								
50								
50								
39								
35	20.0							
43								
48								
49								
49								
51	25.0							

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DRILL RIG OPERATOR
SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER **UNOFFICIAL LOG**
DATE APPROVED
RESIDENT ENGINEER
STRUCTURE NAME **B.I.N.** 103989A
Route 198 Ramp over Scajaquada Creek

SM 282 E 12/02

STATE OF NEW YORK
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DATE START 12-NOV-1956 **DATE FINISH** 13-NOV-1956

HOLE PH-B-F-2
LINE Baseline
STA 455+25
OFFSET 182 ft LEFT
SURF. ELEV. 585.87
DEPTH TO WATER SEE NOTE

CASING O. D. 2 7/8 in **I. D.** 2 3/8 in **WT OF HAMMER-CASING** 300 lb **HAMMER FALL-CASING** 18 in
SAMPLER O. D. in **I. D.** in **WT OF HAMMER-SAMPLER** lb **HAMMER FALL-SAMPLER** in

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	25.0							(25.00) Cased Probing - No Samples Taken
59								
55								
66								
77								
95	30.0							
112								
140								
159								
179								
197	35.0							
210								
260								
255								
257								
260	40.0							
350								
323								
240								
250								
200	45.0							
216								
145								
212								
193								
205	50.0							

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SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER UNOFFICIAL LOG
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RESIDENT ENGINEER
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SM 282 E 12/02

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HOLE PH-B-F-2
LINE Baseline
STA 455+25
OFFSET 182 ft LEFT
SURF. ELEV. 585.87
DEPTH TO WATER SEE NOTE

DATE START 12-NOV-1956 **DATE FINISH** 13-NOV-1956

CASING	**O. D.**	2 7/8 in	**I. D.**	2 3/8 in	**WT OF HAMMER-CASING**	300 lb	**HAMMER FALL-CASING**	18	in
SAMPLER	**O. D.**	in	**I. D.**	in	**WT OF HAMMER-SAMPLER**	lb	**HAMMER FALL-SAMPLER**		in

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	50.0							(50.00) Cased Probing - No Samples Taken
210								
239								
200								
141								
150	55.0							
161								
154								
150								
157								
172	60.0							
213								
212								
200								
223								
208	65.0							
178								
183								
185								
186								
200	70.0							
194								
220								
222								
210								
212	75.0							

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HOLE PH-B-F-2
LINE Baseline
STA 455+25
OFFSET 182 ft LEFT
SURF. ELEV. 585.87
DEPTH TO WATER SEE NOTE

CASING **O. D.** 2 7/8 in **I. D.** 2 3/8 in **WT OF HAMMER-CASING** 300 lb **HAMMER FALL-CASING** 18 in
SAMPLER **O. D.** in **I. D.** in **WT OF HAMMER-SAMPLER** lb **HAMMER FALL-SAMPLER** in

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	75.0							(75.00) Cased Probing - No Samples Taken
226								
223								
220								
229								
262	80.0							
271								
251								
276								
297								
314	85.0							
375								
800								
		R1						(87.42) Run #1 Drilled from 87'5" To 92'5" LIMESTONE , Fractured And Broken REC. 53.5" 89.2% 41
	90.0							

BOTTOM OF HOLE AT 92.42 ft

DATE	TIME	DEPTH ft			ARTESIAN HEAD HEIGHT ABOVE GROUND	FILLED WITH WATER AT END OF DAY
		HOLE	CASING	WATER		
13-Nov-56	15:00	92.42	86.50	15.00		

The subsurface information shown here was obtained for design and estimate purposes. It is made available so that users may have access to the same information available to the State. It is presented in good faith. By the nature of the exploration process, the information represents only a small fraction of the total volume of the material at the site. Interpolation between data samples may not be indicative of the actual material encountered.

CONTRACT **CONTRACTOR** Westville Engineering

DRILL RIG OPERATOR
SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER UNOFFICIAL LOG
DATE APPROVED
RESIDENT ENGINEER
STRUCTURE NAME **B.I.N.** 103989A
Route 198 Ramp over Scajaquada Creek
SHEET 4 OF 4 HOLE PH-B-F-2

SM 282 E 12/02

STATE OF NEW YORK
DEPARTMENT OF TRANSPORTATION
GEOTECHNICAL ENGINEERING BUREAU
SUBSURFACE EXPLORATION LOG

UNOFFICIAL LOG

PSN 253 BORNUM 65
REGION 5
COUNTY ERIE
PIN
PROJECT SCAJAQUADA CREEK EXPRESSWAY DELAWARE PARK
SCALED COORDINATES (N) 1,067,341.450 (E) 1,063,913.330 **DATUM** NAD83
DATE START 12-NOV-1956 **DATE FINISH** 15-NOV-1956

HOLE DH-B-F-4
LINE Baseline
STA 454+06
OFFSET 120 ft LEFT
SURF. ELEV. 587.05
DEPTH TO WATER SEE NOTE

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18 **in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18 **in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
23	0.0							(0.00) Black SAND , Some Gravel, Cinders And Rubble, Trace Silt And Clay - Fill Material (D-LW)
35								
16								
13								
15	5.0							
31		J1	5	7	7			
20								
20								
23								
30	10.0							
20		J2	4	5				
31		J2A	5					(11.00) Red SILT , Some Clay (D-PW)
32								
35								(13.00) Red SILT , Some Clay (M-PL)
37	15.0							
36		J3	2	3	6			
39								
36								
37								
40	20.0							
41		J4	2	8	5			
53								
47								
41								
52	25.0							

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CONTRACT _____ **CONTRACTOR** Westville Engineering

DRILL RIG OPERATOR
SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER UNOFFICIAL LOG
DATE APPROVED
RESIDENT ENGINEER
STRUCTURE NAME Route 198 Ramp over Scajaquada Creek
B.I.N. 103989A

SM 282 E 12/02

STATE OF NEW YORK
DEPARTMENT OF TRANSPORTATION
GEOTECHNICAL ENGINEERING BUREAU
SUBSURFACE EXPLORATION LOG

UNOFFICIAL LOG

PSN 253 BORNUM 65
REGION 5
COUNTY ERIE
PIN
PROJECT SCAJAQUADA CREEK EXPRESSWAY DELAWARE PARK
SCALED COORDINATES (N) 1,067,341.450 (E) 1,063,913.330 **DATUM** NAD83
DATE START 12-NOV-1956 **DATE FINISH** 15-NOV-1956

HOLE DH-B-F-4
LINE Baseline
STA 454+06
OFFSET 120 ft LEFT
SURF. ELEV. 587.05
DEPTH TO WATER SEE NOTE

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18	**in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18	**in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	25.0	J5	3	5	5			(25.00) Red SILT , Some Clay (M-PL)
31								
49								
51								
51								
53	30.0							
		J6	2	4	5			
55								
53								
62								
65								
73	35.0							
		J7	2	3	3			
46								
56								
73								
70								
72	40.0							
		J8	3	4	7			(40.00) Red SILT , Some Clay, Trace Gravel And Fine Sand (M-PL)
72								
116								
121								
109								
112	45.0							
		J9	4	6	7			
103								
137								
148								
150								
127	50.0							

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CONTRACT _____ **CONTRACTOR** Westville Engineering

DRILL RIG OPERATOR
SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER UNOFFICIAL LOG
DATE APPROVED
RESIDENT ENGINEER
STRUCTURE NAME Route 198 Ramp over Scajaquada Creek
B.I.N. 103989A

SM 282 E 12/02

STATE OF NEW YORK
DEPARTMENT OF TRANSPORTATION
GEOTECHNICAL ENGINEERING BUREAU
SUBSURFACE EXPLORATION LOG

UNOFFICIAL LOG

PSN 253 BORNUM 65
REGION 5
COUNTY ERIE
PIN
PROJECT SCAJAQUADA CREEK EXPRESSWAY DELAWARE PARK
SCALED COORDINATES (N) 1,067,341.450 (E) 1,063,913.330 **DATUM** NAD83

HOLE DH-B-F-4
LINE Baseline
STA 454+06
OFFSET 120 ft LEFT
SURF. ELEV. 587.05
DEPTH TO WATER SEE NOTE

DATE START 12-NOV-1956 **DATE FINISH** 15-NOV-1956

CASING	**O. D.**	2 7/8 **in**	**I. D.**	2 3/8 **in**	**WT OF HAMMER-CASING**	300 **lb**	**HAMMER FALL-CASING**	18 **in**
SAMPLER	**O. D.**	2 **in**	**I. D.**	1 3/8 **in**	**WT OF HAMMER-SAMPLER**	300 **lb**	**HAMMER FALL-SAMPLER**	18 **in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	50.0	J10	4	5	7			(50.00) Red SILT , Some Clay, Trace Gravel And Fine Sand (M-PL)
141								
183								
173								
187								
178	55.0							
		J11	3	4	8			
165								
260								
323								
301								
326	60.0							
		J12	5	8	10			
221								
270								
290								
270								
271	65.0							
		J13	4	5	7			
250								
321								
320								
375								(68.00) Red SILT , Some Clay (M-PL)
372	70.0							
		J14	4	5				
333								
409								
492								
529								
445	75.0							

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CONTRACT _____ **CONTRACTOR** Westville Engineering

DRILL RIG OPERATOR
SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER UNOFFICIAL LOG
DATE APPROVED
RESIDENT ENGINEER
STRUCTURE NAME Route 198 Ramp over Scajaquada Creek **B.I.N.** 103989A

SM 282 E 12/02

STATE OF NEW YORK
DEPARTMENT OF TRANSPORTATION
GEOTECHNICAL ENGINEERING BUREAU
SUBSURFACE EXPLORATION LOG

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PSN 253 BORNUM 65
REGION 5
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PIN
PROJECT SCAJAQUADA CREEK EXPRESSWAY DELAWARE PARK
SCALED COORDINATES (N) 1,067,341.450 (E) 1,063,913.330 **DATUM** NAD83
DATE START 12-NOV-1956 **DATE FINISH** 15-NOV-1956

HOLE DH-B-F-4
LINE Baseline
STA 454+06
OFFSET 120 ft LEFT
SURF. ELEV. 587.05
DEPTH TO WATER SEE NOTE

CASING	**O. D.**	2 7/8	**in**	**I. D.**	2 3/8	**in**	**WT OF HAMMER-CASING**	300	**lb**	**HAMMER FALL-CASING**	18 **in**
SAMPLER	**O. D.**	2	**in**	**I. D.**	1 3/8	**in**	**WT OF HAMMER-SAMPLER**	300	**lb**	**HAMMER FALL-SAMPLER**	18 **in**

CASING BLOWS/ft	DEPTH ft BELOW SURFACE	SAMPLE NO.	BLOWS ON SAMPLER in 0/6	6/12	12/18	18/24	MOIST. CONT. (%)	DESCRIPTION OF SOIL AND ROCK
	75.0	J15	2	4				(75.00) Red SILT , Some Clay (M-PL)
294								
317								
340								
								(78.00) Gray SILT , Some Gravel And Sand, Trace Clay (M-LPL)
381								
369	80.0	J16	18	14	13			
329								
314								
300								
								(83.00) Gray GRAVEL , Some Clay, Trace Silt (M-NPL)
336								Gravel Consists Largely Of Broken Limestone
371	85.0	J17	9	5	5			
483								
342		J18	5	13				
482								
		R1						(87.75) Run #1 Drilled from 87'9" To 92'9" LIMESTONE , Fractured And Broken
								REC. 46" 76.7% 23
	90.0							

BOTTOM OF HOLE AT 92.75 ft

DATE	TIME	DEPTH ft			ARTESIAN HEAD HEIGHT ABOVE GROUND	FILLED WITH WATER AT END OF DAY
		HOLE	CASING	WATER		
15-Nov-56	15:00	92.75	87.75	15.00		

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CONTRACT _____ **CONTRACTOR** Westville Engineering

DRILL RIG OPERATOR
SOIL & ROCK DESCRIPTION H. Daniels
REG GEOTECHNICAL ENGINEER **UNOFFICIAL LOG**
DATE APPROVED
RESIDENT ENGINEER
STRUCTURE NAME **B.I.N.** 103989A
Route 198 Ramp over Scajaquada Creek

APPENDIX C

MONITORING WELL CONSTRUCTION LOGS

LABELLA MONITORING WELL CONSTRUCTION LOGS

LABELLA Associates, D.P.C.
300 PEARL STREET, BUFFALO, NEW YORK
ENVIRONMENTAL ENGINEERING CONSULTANTS

PROJECT
Buffalo Niagara Riverkeeper
1660 Niagara Street, Buffalo, New York

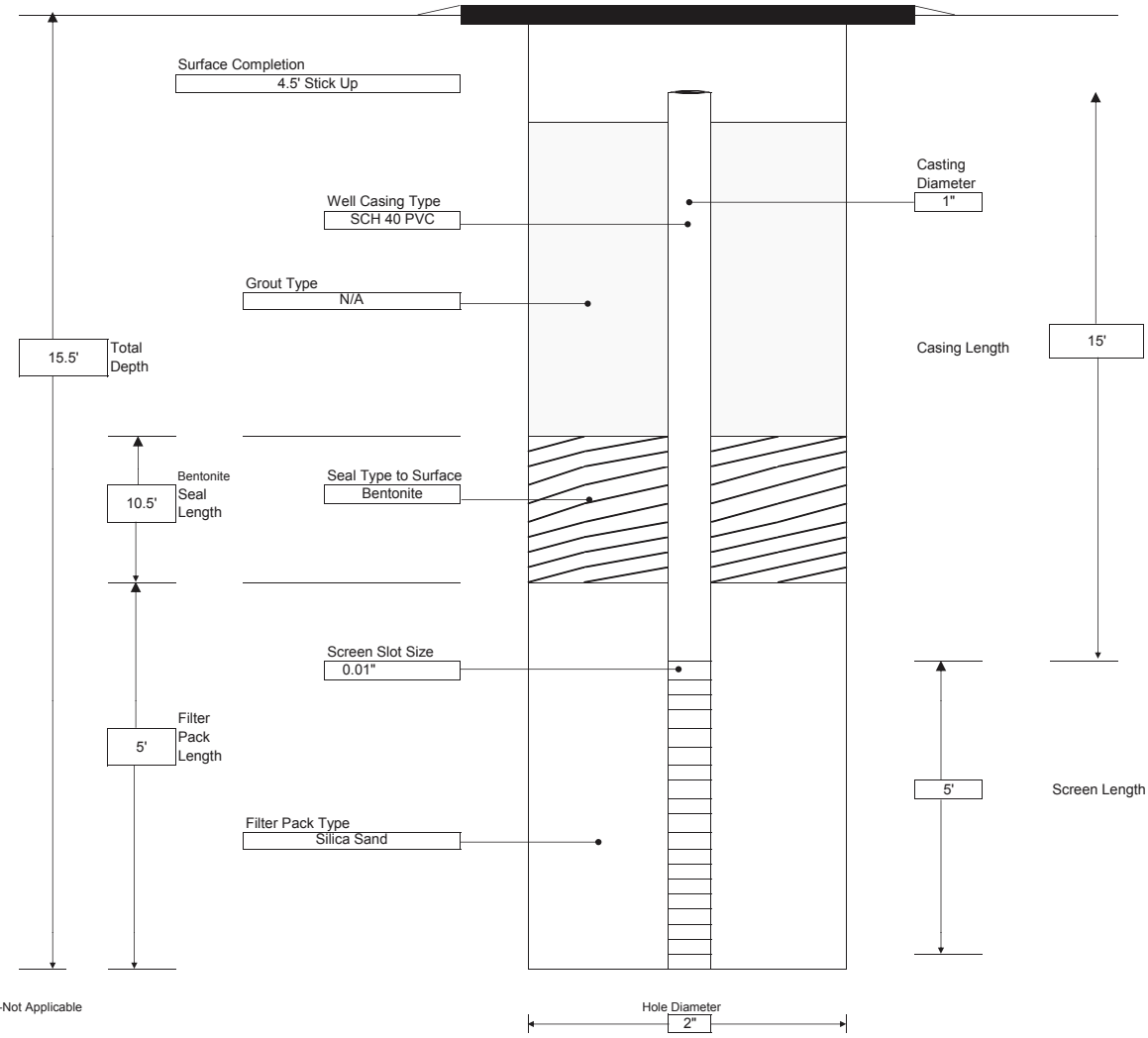
BORING: TPMW1
SHEET 1 OF 3
JOB # 2151177
CHKD. BY: CK

CONTRACTOR: Nature's Way Environmental
DRILLER:
LABELLA REPRESENTATIVE: Chris Kibler

BORING LOCATION: SB1
GROUND SURFACE ELEVATION: DATUM:
START DATE: 12/11/2015 END DATE:

TYPE OF DRILL RIG:
AUGER SIZE AND TYPE: N/A
OVERBURDEN SAMPLING METHOD: Direct-Push
ROCK DRILLING METHOD: N/A

WATER LEVEL DATA				
DATE	TIME	WATER	CASING	REMARKS



NA-Not Applicable

GENERAL NOTES:
1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL
2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER MAY OCCUR DUE TO OTHER FACTORS THAN THOSE PRESENT AT THE TIME MEASUREMENTS WERE MADE.

LABELLA Associates, DPC. 300 PEARL STREET, BUFFALO, NEW YORK ENVIRONMENTAL ENGINEERING CONSULTANTS	PROJECT Buffalo Niagara Riverkeeper 1660 Niagara Street, Buffalo, New York	BORING: TPMW2 SHEET 2 OF 3 JOB # 2151177 CHKD. BY: CK

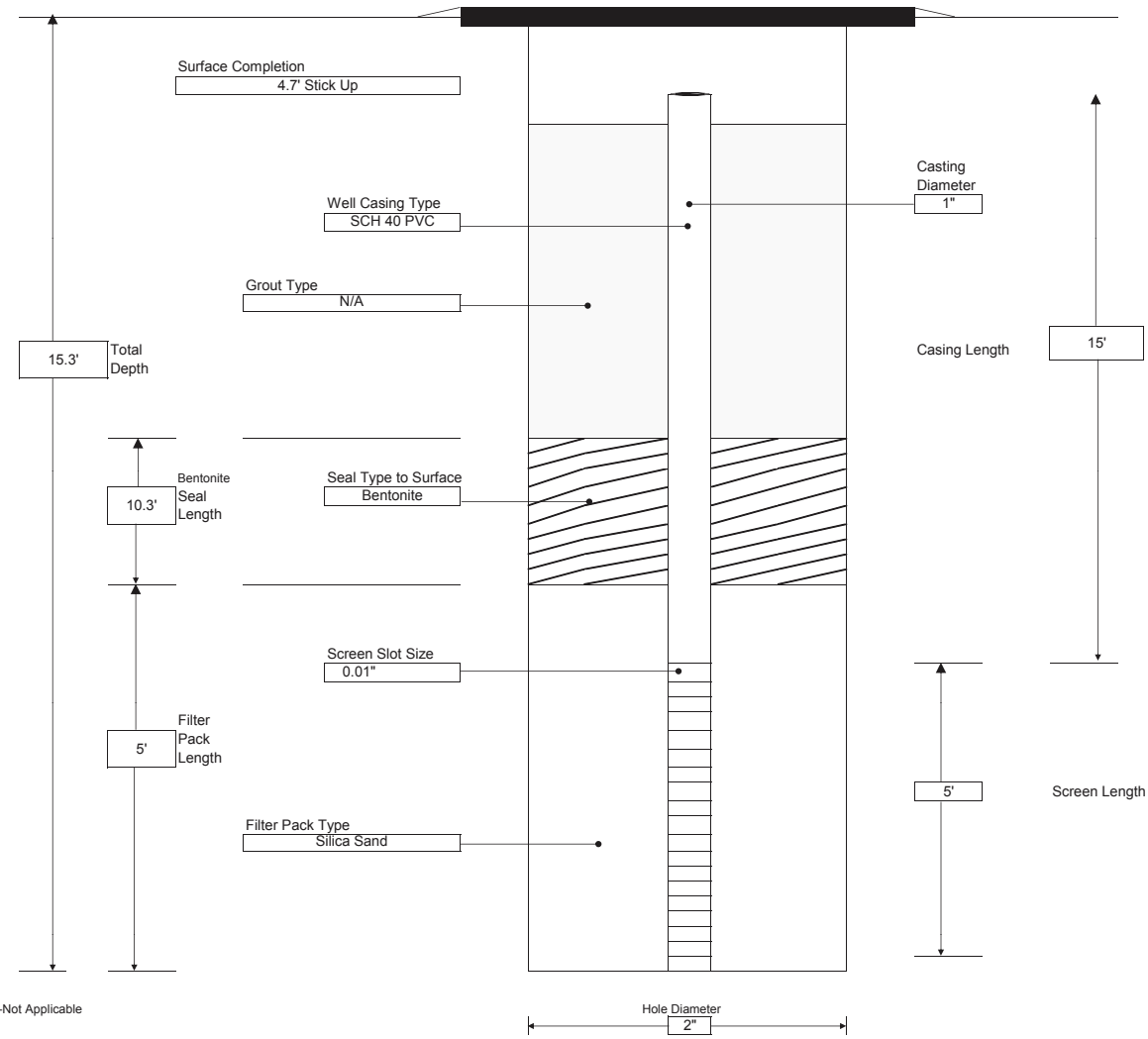
CONTRACTOR: Nature's Way Environmental
DRILLER:
LABELLA REPRESENTATIVE: Chris Kibler

BORING LOCATION: SB4
GROUND SURFACE ELEVATION: DATUM:
START DATE: 12/11/2015 END DATE:

TYPE OF DRILL RIG:
AUGER SIZE AND TYPE: N/A
OVERBURDEN SAMPLING METHOD: Direct-Push
ROCK DRILLING METHOD: N/A

WATER LEVEL DATA

DATE	TIME	WATER	CASING	REMARKS



NA-Not Applicable

GENERAL NOTES:
1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL
2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER MAY OCCUR DUE TO OTHER FACTORS THAN THOSE PRESENT AT THE TIME MEASUREMENTS WERE MADE.

LABELLA Associates, D.P.C. 300 PEARL STREET, BUFFALO, NEW YORK ENVIRONMENTAL ENGINEERING CONSULTANTS	PROJECT Buffalo Niagara Riverkeeper 1660 Niagara Street, Buffalo, New York	BORING: TPMW3 SHEET 3 OF 3 JOB # 2151177 CHKD. BY: CK

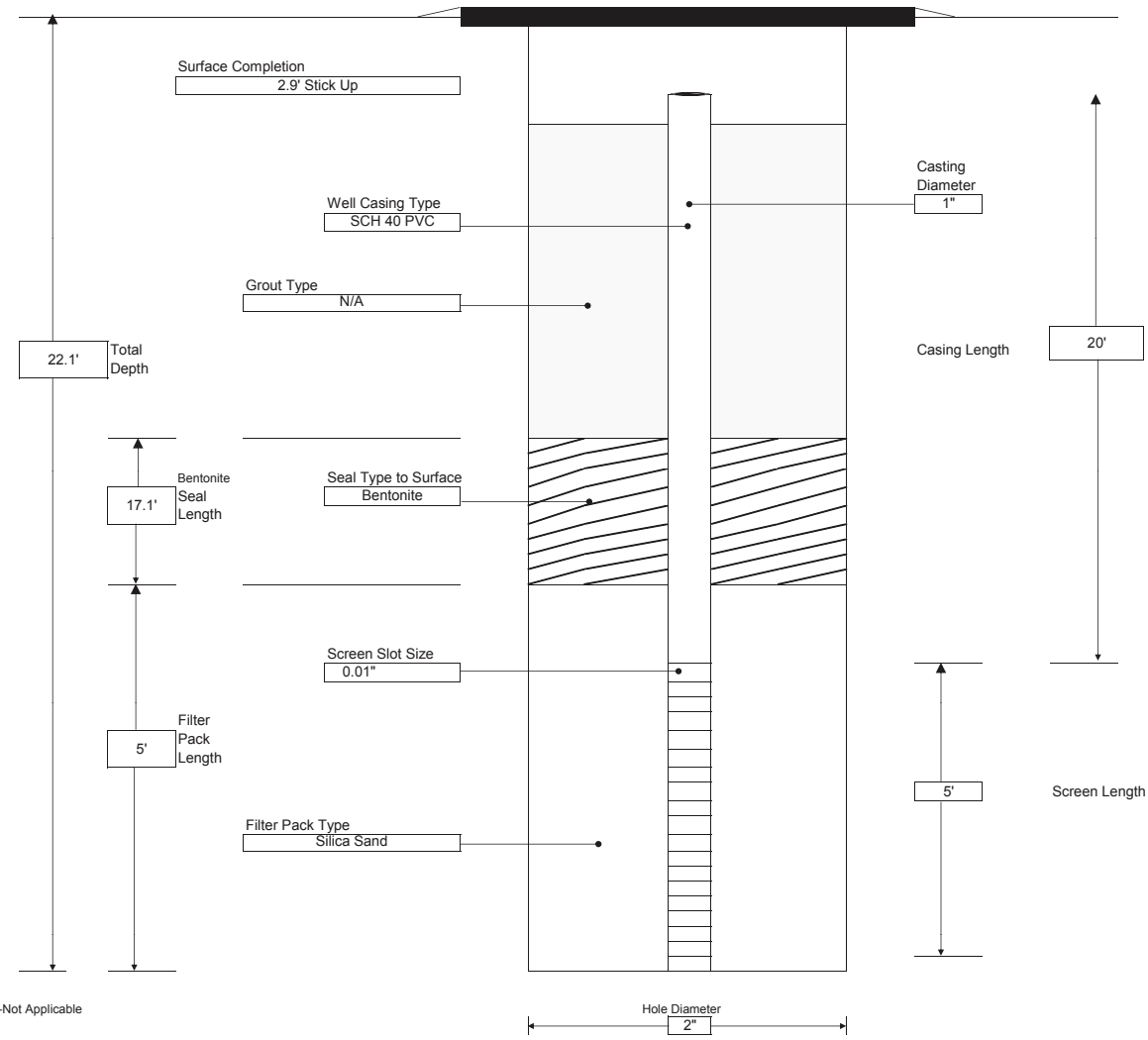
CONTRACTOR: Nature's Way Environmental
DRILLER:
LABELLA REPRESENTATIVE: Chris Kibler

BORING LOCATION: SB8
GROUND SURFACE ELEVATION: DATUM:
START DATE: 12/11/2015 END DATE:

TYPE OF DRILL RIG:
AUGER SIZE AND TYPE: N/A
OVERBURDEN SAMPLING METHOD: Direct-Push
ROCK DRILLING METHOD: N/A

WATER LEVEL DATA

DATE	TIME	WATER	CASING	REMARKS



NA-Not Applicable

GENERAL NOTES:
1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL
2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER MAY OCCUR DUE TO OTHER FACTORS THAN THOSE PRESENT AT THE TIME MEASUREMENTS WERE MADE.

LABELLA Associates, D.P.C.
300 PEARL STREET, BUFFALO, NEW YORK
ENVIRONMENTAL ENGINEERING CONSULTANTS

PROJECT
1660 Niagara Street
Buffalo, New York
Remedial Investigation

Well ID: **RI-MW-1**
SHEET 1 OF 1
JOB # 2161447
CHKD. BY:

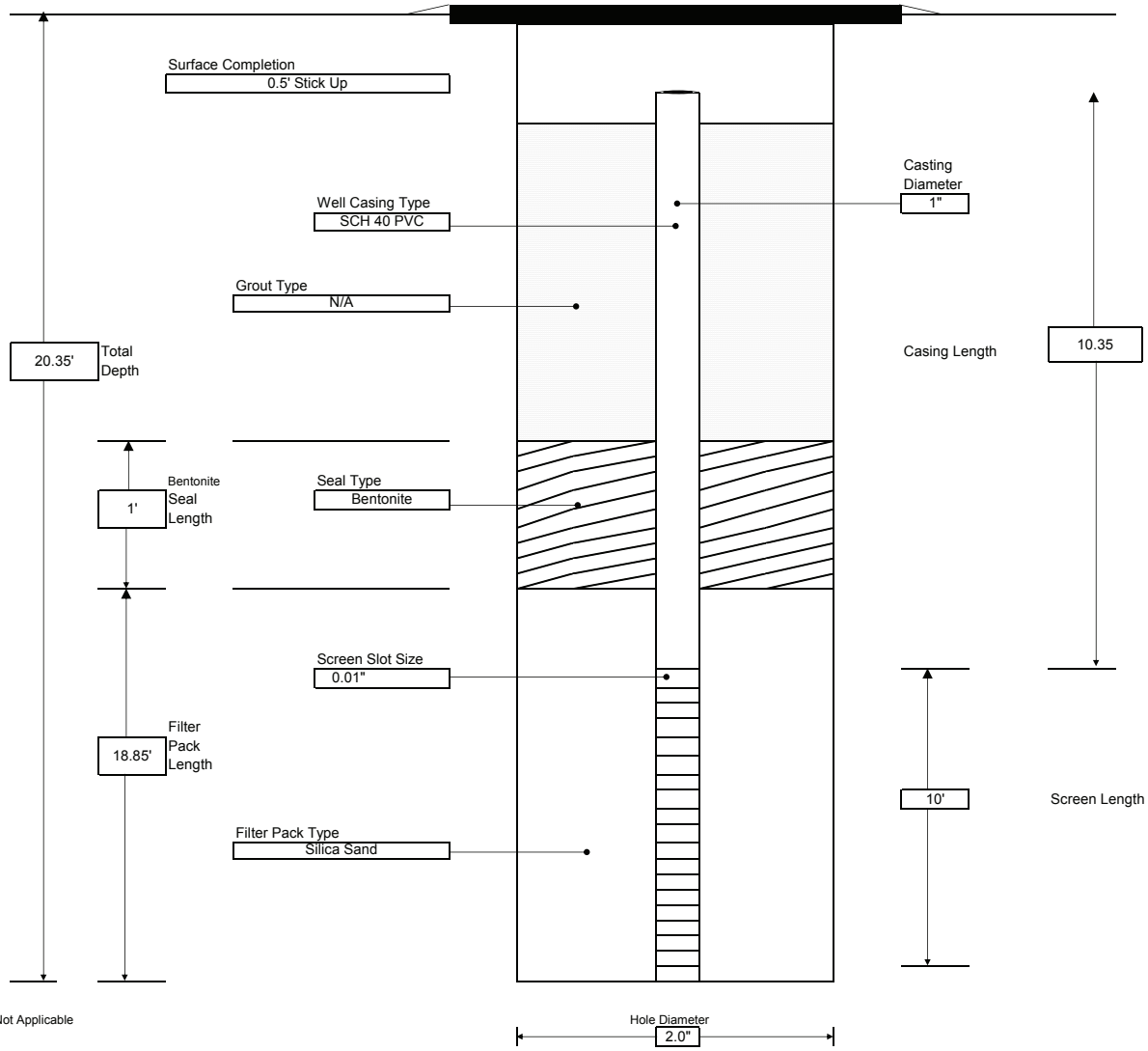
CONTRACTOR: LaBella LLC
DRILLER: Matt Pepe
LABELLA REPRESENTATIVE: Jessica Dombrowski

BORING LOCATION: RI-SB-1
GROUND SURFACE ELEVATION: DATUM: Top of Riser
START DATE: 5/30/2017 END DATE:5/30/2017

TYPE OF DRILL RIG:
AUGER SIZE AND TYPE: 2 inch Direct Push
OVERBURDEN SAMPLING METHOD:5-foot MacroCore® sampler
ROCK DRILLING METHOD: N/A

WATER LEVEL DATA

DATE	TIME	WATER	CASING	REMARKS
6/1/2017	1610	7.9'		



NA-Not Applicable

GENERAL NOTES:
1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL
2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER MAY OCCUR DUE TO OTHER FACTORS THAN THOSE PRESENT AT THE TIME MEASUREMENTS WERE MADE.

LABELLA Associates, D.P.C.
300 PEARL STREET, BUFFALO, NEW YORK
ENVIRONMENTAL ENGINEERING CONSULTANTS

PROJECT
1660 Niagara Street
Buffalo, New York
Remedial Investigation

Well ID: **RI-MW-2**
SHEET 1 OF 1
JOB # 2161447
CHKD. BY:

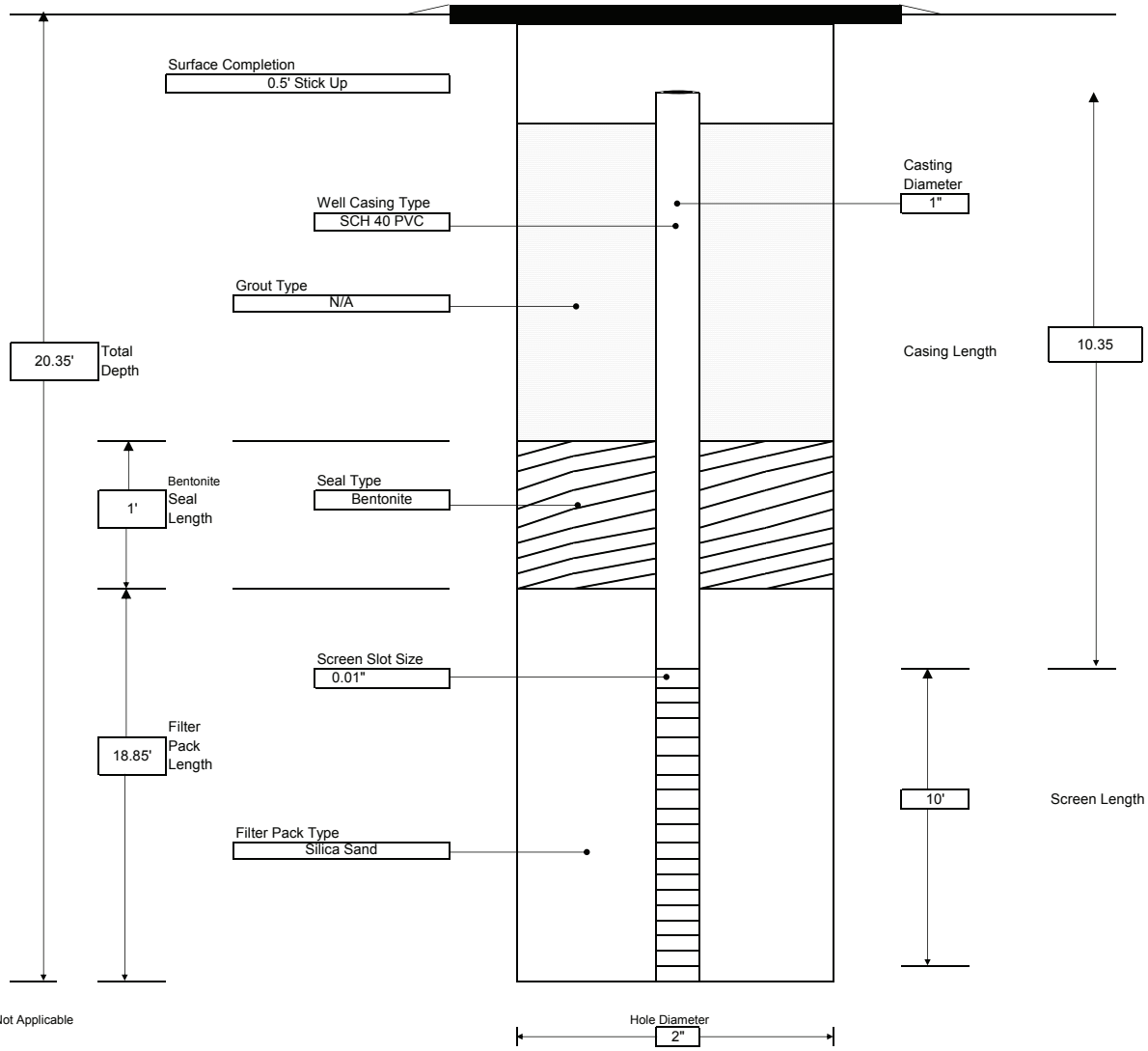
CONTRACTOR: LaBella LLC
DRILLER: Matt Pepe
LABELLA REPRESENTATIVE: Jessica Dombrowski

BORING LOCATION: RI-SB-2
GROUND SURFACE ELEVATION: DATUM: Top of Riser
START DATE: 5/30/2017 END DATE:5/30/2017

TYPE OF DRILL RIG:
AUGER SIZE AND TYPE: 2 inch Direct Push
OVERBURDEN SAMPLING METHOD:5-foot MacroCore® sampler
ROCK DRILLING METHOD: N/A

WATER LEVEL DATA

DATE	TIME	WATER	CASING	REMARKS
6/1/2017	1515	8.3'		



NA-Not Applicable

GENERAL NOTES:
1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL
2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER MAY OCCUR DUE TO OTHER FACTORS THAN THOSE PRESENT AT THE TIME MEASUREMENTS WERE MADE.

LABELLA Associates, D.P.C.
300 PEARL STREET, BUFFALO, NEW YORK
ENVIRONMENTAL ENGINEERING CONSULTANTS

PROJECT
1660 Niagara Street
Buffalo, New York
Remedial Investigation

Well ID: **RI-MW-3**
SHEET 1 OF 1
JOB # 2161447
CHKD. BY:

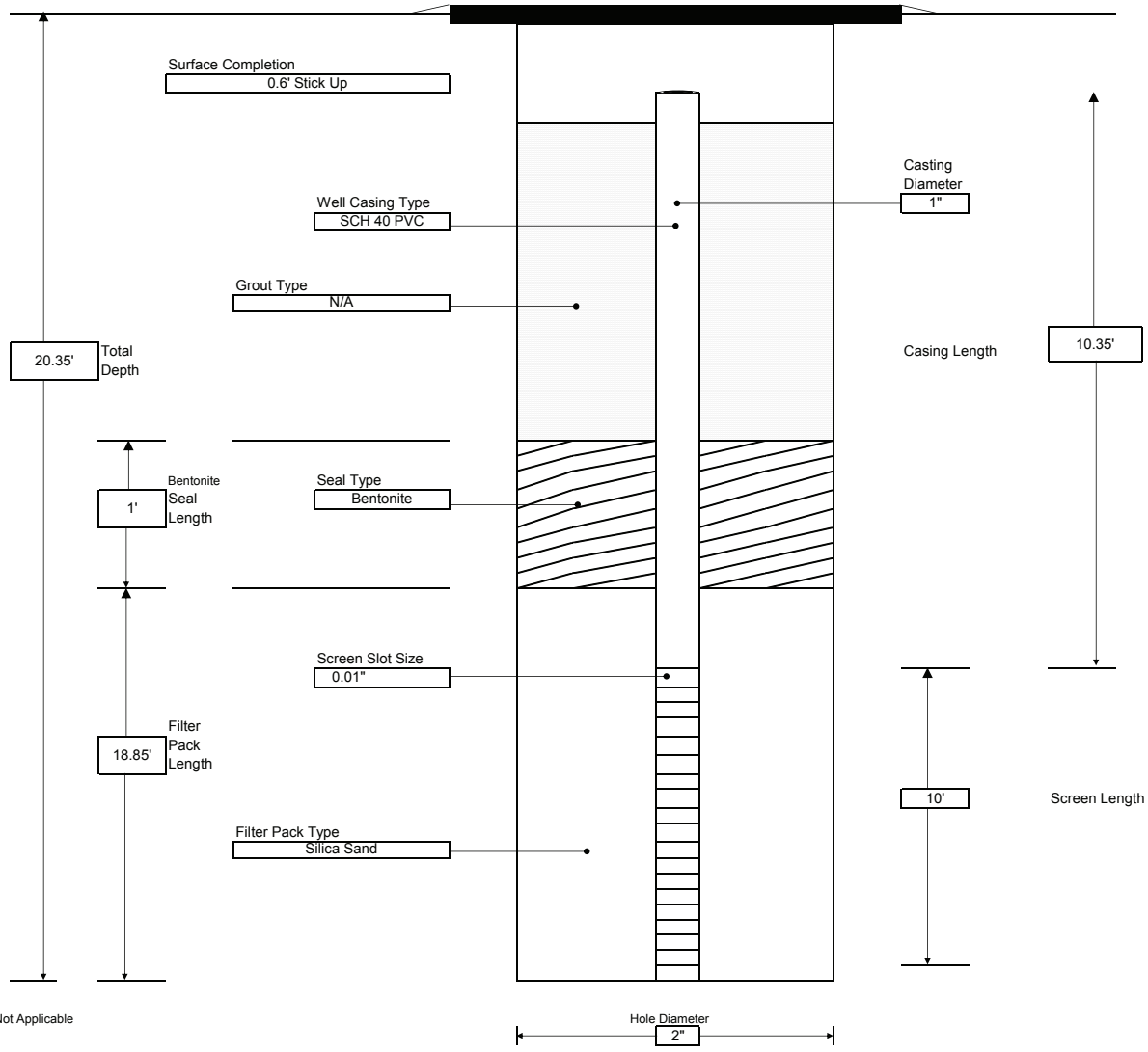
CONTRACTOR: LaBella LLC
DRILLER: Matt Pepe
LABELLA REPRESENTATIVE: Jessica Dombrowski

BORING LOCATION: RI-SB-3
GROUND SURFACE ELEVATION: DATUM: Top of Riser
START DATE: 5/30/2017 END DATE:5/30/2017

TYPE OF DRILL RIG:
AUGER SIZE AND TYPE: 2 inch Direct Push
OVERBURDEN SAMPLING METHOD:5-foot MacroCore® sampler
ROCK DRILLING METHOD: N/A

WATER LEVEL DATA

DATE	TIME	WATER	CASING	REMARKS
6/1/2017	1350	5.6'		



NA-Not Applicable

GENERAL NOTES:
1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL
2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER MAY OCCUR DUE TO OTHER FACTORS THAN THOSE PRESENT AT THE TIME MEASUREMENTS WERE MADE.

LABELLA Associates, D.P.C.
300 PEARL STREET, BUFFALO, NEW YORK
ENVIRONMENTAL ENGINEERING CONSULTANTS

PROJECT
1660 Niagara Street
Buffalo, New York
Remedial Investigation

Well ID: **RI-MW-4**
SHEET 1 OF 1
JOB # 2161447
CHKD. BY:

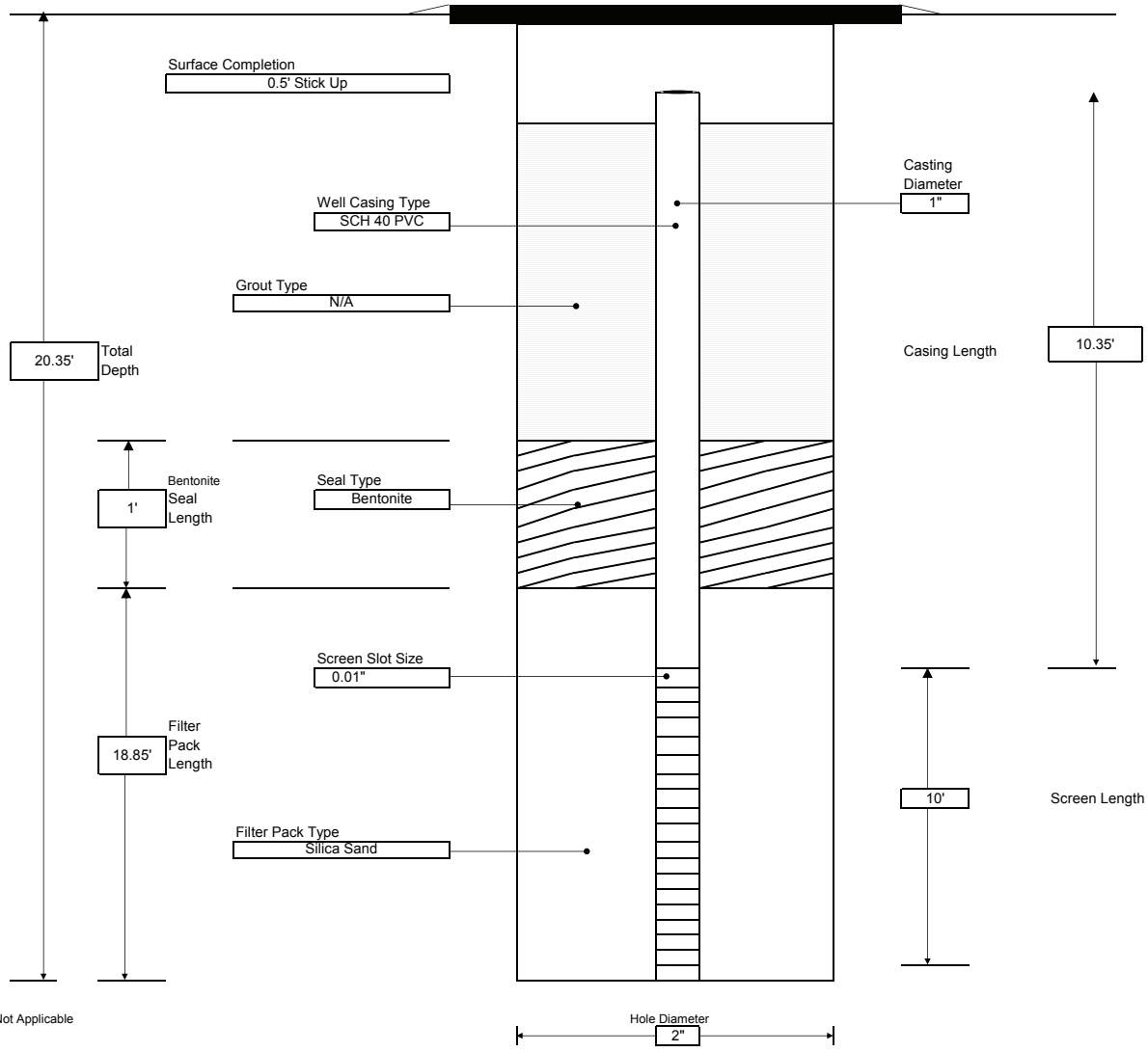
CONTRACTOR: LaBella LLC
DRILLER: Matt Pepe
LABELLA REPRESENTATIVE: Jessica Dombrowski

BORING LOCATION: RI-SB-4
GROUND SURFACE ELEVATION: DATUM: Top of Riser
START DATE: 5/30/2017 END DATE:5/30/2017

TYPE OF DRILL RIG:
AUGER SIZE AND TYPE: 2 inch Direct Push
OVERBURDEN SAMPLING METHOD:5-foot MacroCore® sampler
ROCK DRILLING METHOD: N/A

WATER LEVEL DATA

DATE	TIME	WATER	CASING	REMARKS
6/1/2017	1200	5.1'		



NA-Not Applicable

GENERAL NOTES:
1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL
2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER MAY OCCUR DUE TO OTHER FACTORS THAN THOSE PRESENT AT THE TIME MEASUREMENTS WERE MADE.

LABELLA Associates, D.P.C.
300 PEARL STREET, BUFFALO, NEW YORK
ENVIRONMENTAL ENGINEERING CONSULTANTS

PROJECT
1660 Niagara Street
Buffalo, New York
Remedial Investigation

Well ID: **RI-MW-5**
SHEET 1 OF 1
JOB # 2161447
CHKD. BY:

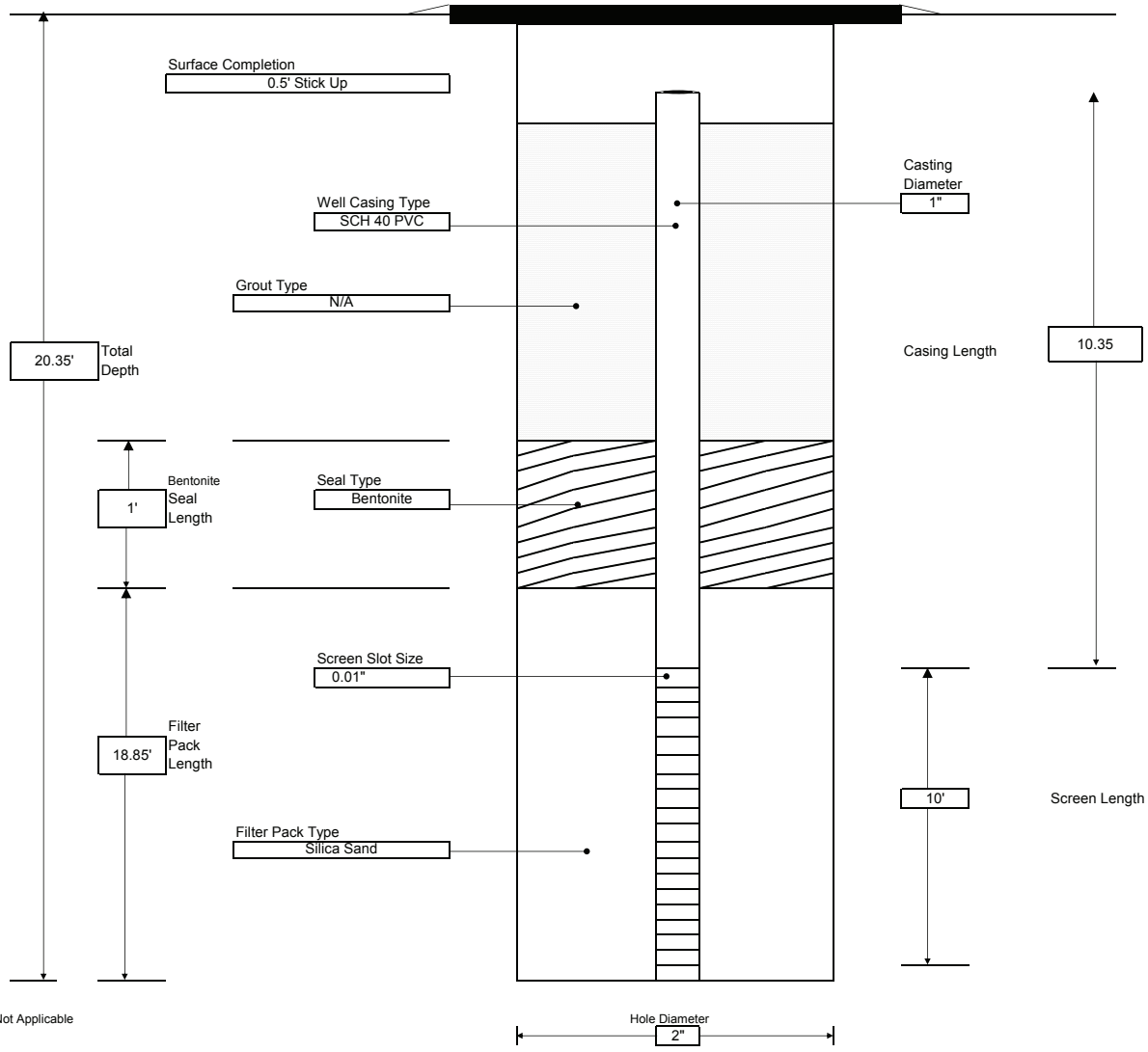
CONTRACTOR: LaBella LLC
DRILLER: Matt Pepe
LABELLA REPRESENTATIVE: Jessica Dombrowski

BORING LOCATION:RI-SB-5
GROUND SURFACE ELEVATION: DATUM: Top of Riser
START DATE: 5/30/2017 END DATE:5/30/2017

TYPE OF DRILL RIG:
AUGER SIZE AND TYPE: 2 inch Direct Push
OVERBURDEN SAMPLING METHOD:5-foot MacroCore® sampler
ROCK DRILLING METHOD: N/A

WATER LEVEL DATA

DATE	TIME	WATER	CASING	REMARKS
6/1/2017	900	10.8'		



NA-Not Applicable

GENERAL NOTES:
1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL
2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER MAY OCCUR DUE TO OTHER FACTORS THAN THOSE PRESENT AT THE TIME MEASUREMENTS WERE MADE.

LABELLA Associates, D.P.C.
300 PEARL STREET, BUFFALO, NEW YORK
ENVIRONMENTAL ENGINEERING CONSULTANTS

PROJECT
1660 Niagara Street
Buffalo, New York
Remedial Investigation

Well ID: **RI-MW-6**
SHEET 1 OF 1
JOB # 2161447
CHKD. BY:

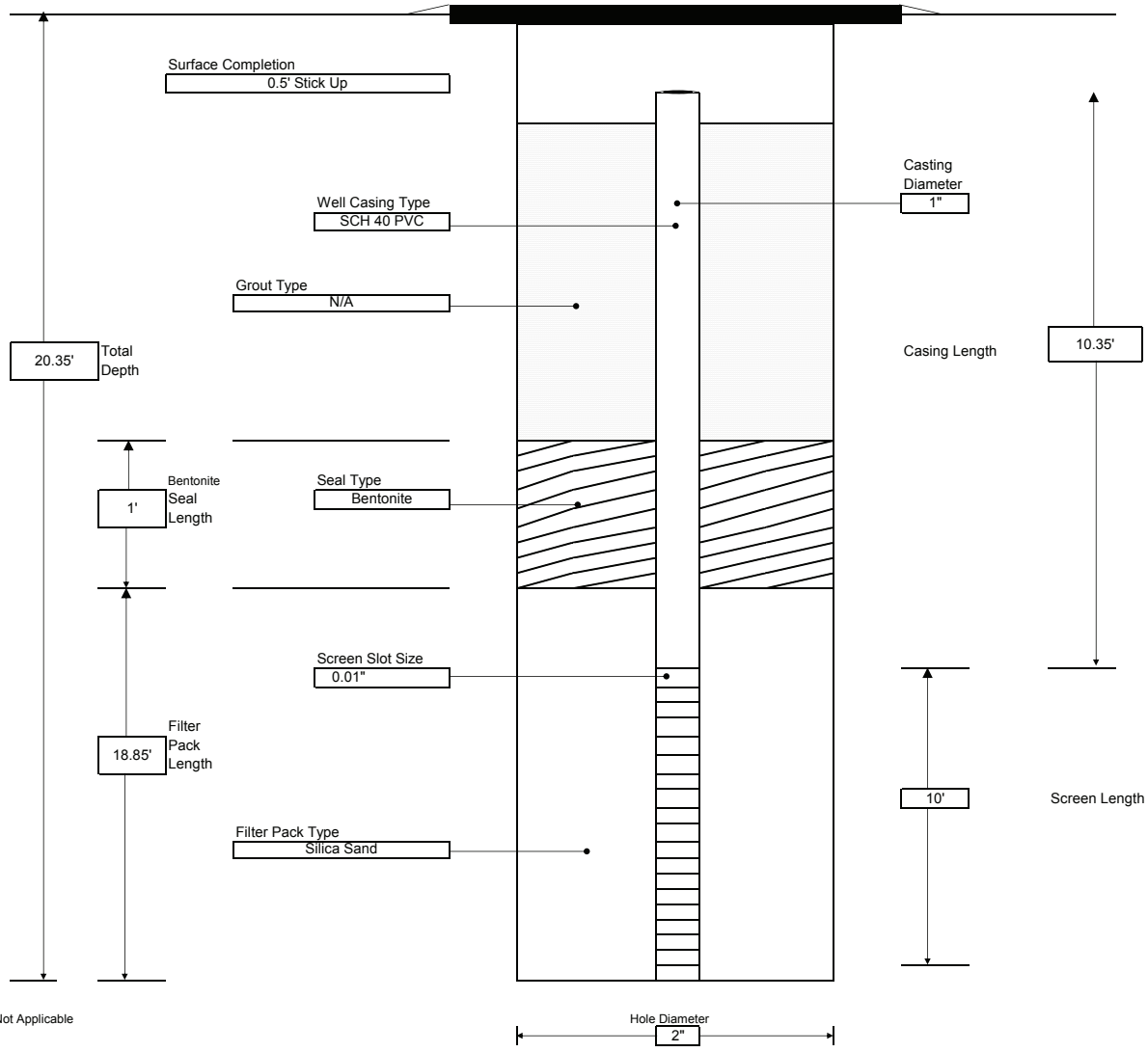
CONTRACTOR: LaBella LLC
DRILLER: Matt Pepe
LABELLA REPRESENTATIVE: Jessica Dombrowski

BORING LOCATION:RI-SB-6
GROUND SURFACE ELEVATION: DATUM: Top of Riser
START DATE: 5/30/2017 END DATE:5/30/2017

TYPE OF DRILL RIG:
AUGER SIZE AND TYPE: 2 inch Direct Push
OVERBURDEN SAMPLING METHOD:5-foot MacroCore® sampler
ROCK DRILLING METHOD: N/A

WATER LEVEL DATA

DATE	TIME	WATER	CASING	REMARKS
6/1/2017	835	6.1'		



NA-Not Applicable

GENERAL NOTES:
1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL
2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER MAY OCCUR DUE TO OTHER FACTORS THAN THOSE PRESENT AT THE TIME MEASUREMENTS WERE MADE.

LABELLA Associates, D.P.C.
300 PEARL STREET, BUFFALO, NEW YORK
ENVIRONMENTAL ENGINEERING CONSULTANTS

PROJECT
1660 Niagara Street
Buffalo, New York
Remedial Investigation

Well ID: **RI-MW-7**
SHEET 1 OF 1
JOB # 2161447
CHKD. BY:

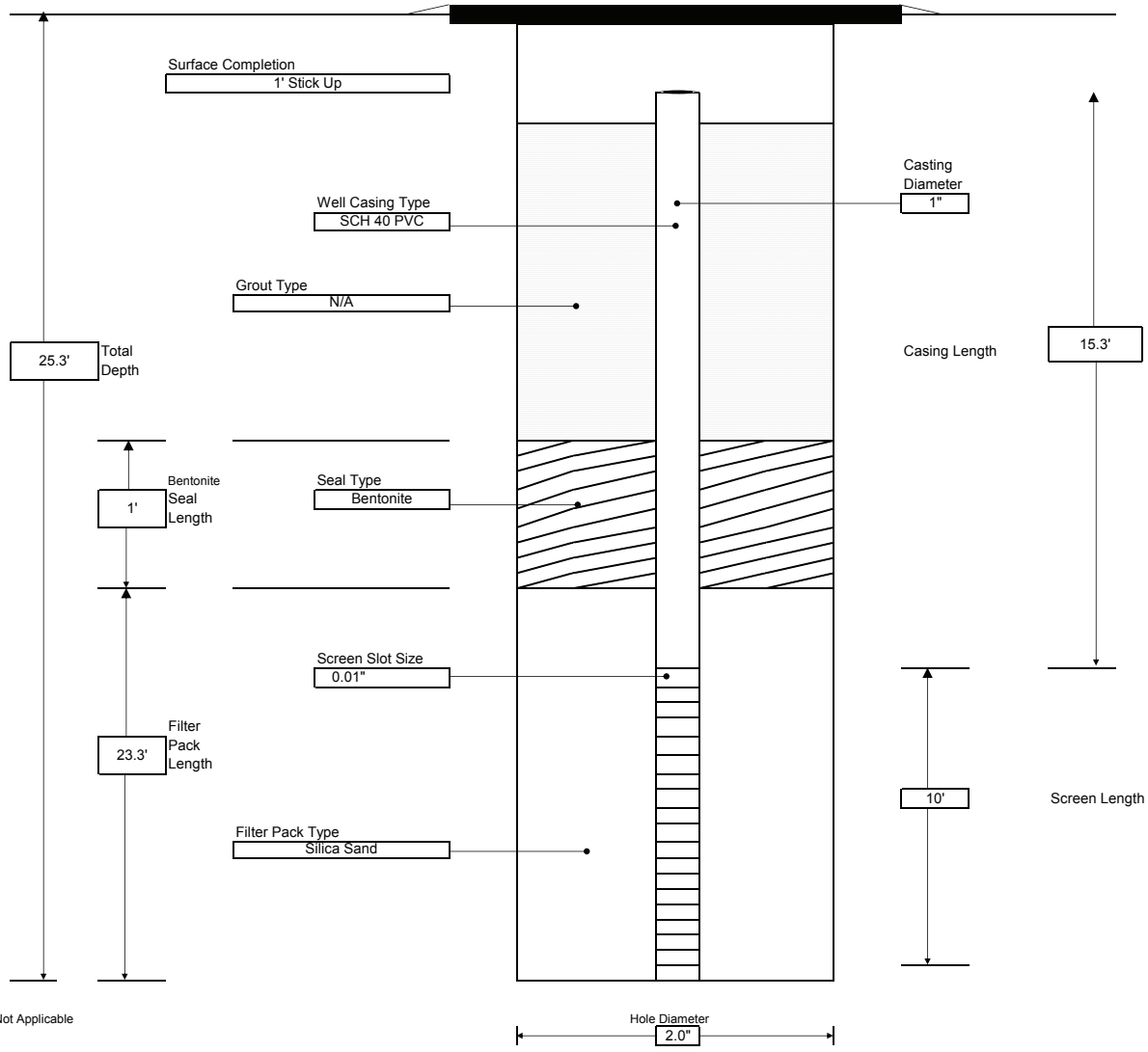
CONTRACTOR: LaBella LLC
DRILLER: Matt Pepe
LABELLA REPRESENTATIVE: Jessica Dombrowski

BORING LOCATION:RI-SB-7
GROUND SURFACE ELEVATION: DATUM: Top of Riser
START DATE: 5/30/2017 END DATE:5/30/2017

TYPE OF DRILL RIG:
AUGER SIZE AND TYPE: 2 inch Direct Push
OVERBURDEN SAMPLING METHOD:5-foot MacroCore® sampler
ROCK DRILLING METHOD: N/A

WATER LEVEL DATA

DATE	TIME	WATER	CASING	REMARKS
8/18/2017	1000	16.4'	25.3	Petroleum Odor and Sheen Observed



NA-Not Applicable

GENERAL NOTES:
1) STRATIFICATION LINES REPRESENT APPROXIMATE BOUNDARY BETWEEN SOIL TYPES, TRANSITIONS MAY BE GRADUAL
2) WATER LEVEL READINGS HAVE BEEN MADE AT TIMES AND UNDER CONDITIONS STATED, FLUCTUATIONS OF GROUNDWATER MAY OCCUR DUE TO OTHER FACTORS THAN THOSE PRESENT AT THE TIME MEASUREMENTS WERE MADE.

Appendix B.2

SC Soil Boring and Well Construction Logs

Boring Location

NORTHING: 1,067,341 **EASTING:** 1,063,933 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 582.17
LOCATION: P-Site

BORING

MW-BGL-1

Drilling Information

DATE START / END: 11/11/2021 - 11/11/2021 **TOTAL DEPTH (FT):** 28.0
CONTRACTOR: Nothnagle **DRILLER:** Tom Mangefreda **LOGGED BY:** M. Cummings
EQUIPMENT: Hand/Geoprobe 7822DT **BORING METHOD:** Hand/Geoprobe
AUGER ID/OD: 4.25 in / N/A **CASING ID/OD:** 2 in / 2 in
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft):
GENERAL NOTES: Top of Riser Elevation: 581.74'

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_P = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	GRAPHIC LOG	Sample Description & Classification
580										Hand clear to 5.0' bgs.
	5		1	SS	4 to 8	48/30		OVM=0.0		**FILL** Reddish-brown silty clay with trace fine sand and fine angular gravel. Moist throughout (reworked native material)
575			2	SS	8 to 12	48/48		OVM=0.1		**Lacustrine Clay** Reddish-brown silty clay with trace fine sand and fine angular gravel, stiff, low plasticity fines, moist throughout.
	10									
570			3	SS	12 to 16	48/48		OVM=0.0		**Lacustrine Clay**, as above, moist.
	15		4	SS	16 to 20	48/48		OVM=0.0		
565										
	20		5	SS	20 to 24	48/48		OVM=0.0		**Lacustrine Clay**, as above, moist.
560										

WELL CONSTRUCTION DETAILS

- Flush mount surface casing
- 2" dia. schedule 40 PVC well riser
- Cement/ bentonite grout
- 3/8" dia. bentonite chips
- 2" dia. schedule 40 PVC well screen #00N Filter Sand

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas
PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Boring Location

NORTHING: 1,067,341 **EASTING:** 1,063,933 **STATION:** N/A **OFFSET:** N/A

HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A

VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 582.17

LOCATION: P-Site

BORING

MW-BGL-1

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	SAMPLE INFORMATION: Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	GRAPHIC LOG	Sample Description & Classification	WELL CONSTRUCTION DETAILS
555	25		6		24 to 28	48/48		OVM=0.0			- 2' sump
										End of Boring at 28 feet Samples to Alpha Analytical: 16-20' below ground surface (566.17-562.17' elev.)	
550	30										
545	35										
540	40										
535	45										
530	50										

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas

PROJECT NAME: National Fuel Lower Scajaquada Creek

CITY/STATE: Buffalo, New York

GEI PROJECT NUMBER: 2101620

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Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Boring Location

NORTHING: 1,067,421 **EASTING:** 1,063,786 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 576.88
LOCATION: P-Site

BORING

MW-BGL-3

Drilling Information

DATE START / END: 11/9/2021 - 11/9/2021 **TOTAL DEPTH (FT):** 28.0
CONTRACTOR: Nothnagle **DRILLER:** Tom Mangefreda **LOGGED BY:** M. Cummings
EQUIPMENT: Hand/Geoprobe 7822DT **BORING METHOD:** Hand/Geoprobe
AUGER ID/OD: 4.25 in / N/A **CASING ID/OD:** 2 in / 2 in
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft):
GENERAL NOTES: Top of Riser Elevation: 579.82'

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_P = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	SAMPLE INFORMATION: Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	GRAPHIC LOG	Sample Description & Classification
575										
	5		1		4 to 8	48/48		OVM=0.5		Hand clear to 5.0' bgs. **FILL** Reddish-brown silty clay with coarse angular limestone gravel, weathered concrete pieces, moist. Locally mottled with black silty clay, firm to hard.
570			2		8 to 12	48/48		OVM=0.1		
	10		3		12 to 16	48/0		OVM=--		No recovery 12-16' interval.
565	15		4		16 to 20	48/21		OVM=20		Loose angular fine gravel with little fine sand, light to moderate diesel-like odor, light sheen, saturated.
560										**Lacustrine Clay** Reddish-brown silty clay with trace fine sand and fine angular gravel, stiff, low plasticity fines, moist throughout.
	20		5		20 to 24	48/48		OVM=0.0		
555										

WELL CONSTRUCTION DETAILS

- Flush mount surface casing
- 2" dia. schedule 40 PVC well riser
- Cement/ bentonite grout
- 3/8" dia. bentonite chips
- 2" dia. schedule 40 PVC well screen
- #00N Filter Sand
- 2' sump
- 3/8" dia. bentonite chips

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas
PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Boring Location

NORTHING: 1,067,421 **EASTING:** 1,063,786 **STATION:** N/A **OFFSET:** N/A

HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A

VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 576.88

LOCATION: P-Site

BORING

MW-BGL-3

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	SAMPLE INFORMATION: Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	GRAPHIC LOG	Sample Description & Classification	WELL CONSTRUCTION DETAILS
550	25		6		24 to 28	48/48		OVM=0.0			3/8" dia. bentonite chips
545	30									End of Boring at 28 feet Samples to Alpha Analytical: 17-19' below ground surface (559.88' to 561.88' elev.) Samples for forensics (Meta Env.) 18-19' below ground surface (560.88' to 559.88' elev.)	
540	35										
535	40										
530	45										
525	50										

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas

PROJECT NAME: National Fuel Lower Scajaquada Creek

CITY/STATE: Buffalo, New York

GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Boring Location

NORTHING: 1,067,490 **EASTING:** 1,064,648 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 585.76
LOCATION: Lower Scajaquada Creek Area

BORING

MW-LSC-1

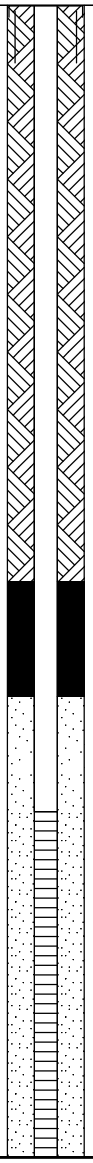
Drilling Information

DATE START / END: 11/1/2021 - 11/1/2021 **TOTAL DEPTH (FT):** 24.0
CONTRACTOR: Nothnagle **DRILLER:** Tom Mangefreda **LOGGED BY:** E. Wood
EQUIPMENT: Hand/Geoprobe 7822DT **BORING METHOD:** Hand/Geoprobe
AUGER ID/OD: 4.25 in / N/A **CASING ID/OD:** 2 in / 2 in
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft):
GENERAL NOTES: Top of Riser Elevation: 585.69'

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_P = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	GRAPHIC LOG	Sample Description & Classification
585			1		0 to 5	60/		OVM=0.4		Topsoil **FILL** Brown/dark brown, fine grained to medium grained sand, trace organics, 1'x1' sandstone blocks from 3' to 5', dry
								OVM=0.2		
								OVM=0.0		
	5		2		5 to 8	36/19				Brown silty sand, fine grained to medium grained, well rounded, well sorted, moist Medium gravel, slag, red brick fragments, some black gravel (wet, no odors)
580								OVM=0.0		
										Reddish brown silty clay, trace small gravel, dry
			3		8 to 12	48/39		OVM=0.1		4" seam of black, coarse-grained poorly-sorted, angular sand and fine gravel
										Transitions to reddish-brown silty clay, trace fine gravel Black coarse grained, poorly sorted, angular, sand and fine gravel Reddish brown silty clay, trace small gravel, dry.
	10							OVM=0.2		
575										
			4		12 to 16	48/24		OVM=0.2		Black fine grained to coarse grained sand with fine gravel, some cinders and possible coal, trace Red brick fragments, saturated, some medium gravel, angular
								OVM=0.1		
	15									
570			5		16 to 20	48/41.5		OVM=0.3		
										Native Alluvium Gray silty clay, little fine grained sand, trace wood/organic material, saturated (4"), increasing fine grained sand at 18' Gray silty clay, organic material (roots, wood, plant material) noted, saturated

WELL CONSTRUCTION DETAILS



Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas
PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Boring Location

NORTHING: 1,067,490 **EASTING:** 1,064,648 **STATION:** N/A **OFFSET:** N/A

HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A

VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 585.76

LOCATION: Lower Scajaquada Creek Area

BORING

MW-LSC-1

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	SAMPLE INFORMATION: Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	GRAPHIC LOG	Sample Description & Classification	WELL CONSTRUCTION DETAILS
565			6	X	20 to 24	48/42		OVM=0.2			
								OVM=0.1		**Lacustrine Clay** Reddish brown silty clay, little fine sand, low plasticity fines, moist, firm.	
	25									End of Boring at 24 feet	- 2' sump
560										Samples to Alpha Analytical:	- 3/8" dia. bentonite chips
	30									13-15' below ground surface (572.76 to 570.76' elev.)	
555										22-24' below ground surface (563.76' to 561.76' elev.)	
	35										
550											
	40										
545											
	45										

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas

PROJECT NAME: National Fuel Lower Scajaquada Creek

CITY/STATE: Buffalo, New York

GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
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Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Boring Location

NORTHING: 1,067,487 **EASTING:** 1,064,471 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 582.46
LOCATION: Lower Scajaquada Creek Area

BORING

MW-LSC-2

Drilling Information

DATE START / END: 11/1/2021 - 11/1/2021 **TOTAL DEPTH (FT):** 40.0
CONTRACTOR: Nothnagle **DRILLER:** Tom Mangefreda **LOGGED BY:** E. Wood
EQUIPMENT: Hand/Geoprobe 7822DT **BORING METHOD:** Hand/Geoprobe
AUGER ID/OD: 4.25 in / N/A **CASING ID/OD:** 2 in / 2 in
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft):
GENERAL NOTES: Top of Riser Elevation: 585.35'

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_P = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	SAMPLE INFORMATION: Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	GRAPHIC LOG	Sample Description & Classification	WELL CONSTRUCTION DETAILS
					0 to 5	60/				Topsoil **FILL** Organics, root material, dry, trace silty clay Gray/red brown silty clay, dry	stickup protective casing
580										Gray silt, little clay, trace fine grained gravel, well sorted, trace angular gravel, slug at 4'	
	5		1		5 to 8	36/31		OVM=0.1		Reworked native reddish brown silty clay, angular fine gravel, firm, moist	2" dia. schedule 40 PVC well riser
575			2		8 to 12	48/48		OVM=0.0		**FILL** as above, red brown reworked native to 9.5' then loose brown silty sand, bricks, angular, fine grained gravel, cinders, slag	
	10										Cement/ bentonite grout
570			3		12 to 16	48/35		OVM=0.3		Loose cindery fill, saturated, top of clay at ~15' bgs, black, slight septic-like odor, no sheen or other odors	
	15							OVM=0.3			
			4		16 to 20	48/30		OVM=0.5		**Native Alluvium** firm to hard, medium plasticity, light gray grading to gray brown at 20' bgs	
565								OVM=1.5			3/8" dia. bentonite chips
	20		5		20 to 24	48/45		OVM=0.2		Moist, firm, gray-brown	
560								OVM=0.2		Sandy silt with little low plasticity fines, firm, wood pieces, shells	#00N Filter Sand

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas
PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Boring Location

NORTHING: 1,067,487 **EASTING:** 1,064,471 **STATION:** N/A **OFFSET:** N/A

HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A

VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 582.46

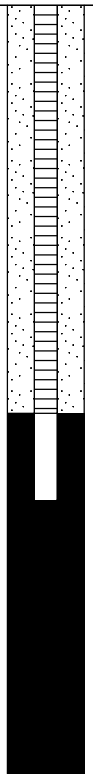
LOCATION: Lower Scajaquada Creek Area

BORING

MW-LSC-2

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	SAMPLE INFORMATION: Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	GRAPHIC LOG	Sample Description & Classification
	25		6		24 to 28	48/12		OVM=0.0		Gray silty sand with gravel, saturated top 6", becoming moist, fine to medium angular gravel, no visual impacts
555										
			7		28 to 32	48/1				Slough from above, little red-brown clay in shoe (1") with staining and moderate coal tar-like odor
	30									**Lacustrine Clay** Reddish brown silty clay, little fine sand, low plasticity fines, moist, firm, no visual impacts
550			8		32 to 36	48/38		OVM=59.4		
	35									
			9		36 to 40	48/44				
545										
	40									End of Boring at 40 feet
540										Samples to Alpha Analytical:
	45									18-20' below ground surface (564.46 to 562.46' elev.)
535										
	50									
530										

WELL CONSTRUCTION DETAILS



Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas

PROJECT NAME: National Fuel Lower Scajaquada Creek

CITY/STATE: Buffalo, New York

GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Boring Location

NORTHING: 1,067,270 **EASTING:** 1,064,263 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 584.19
LOCATION: Lower Scajaquada Creek Area

BORING

MW-LSC-3

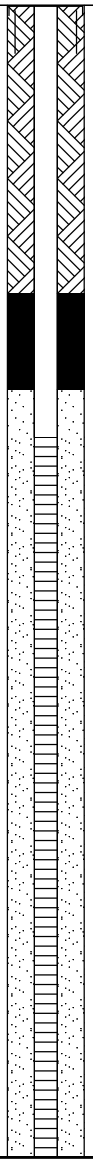
Drilling Information

DATE START / END: 11/1/2021 - 11/8/2021 **TOTAL DEPTH (FT):** 32.0
CONTRACTOR: Nothnagle **DRILLER:** Tom Mangefreda **LOGGED BY:** E. Wood
EQUIPMENT: Hand/Geoprobe 7822DT **BORING METHOD:** Hand/Geoprobe
AUGER ID/OD: 4.25 in / N/A **CASING ID/OD:** 2 in / 2 in
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft):
GENERAL NOTES: Top of Riser Elevation: 586.95'

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_P = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	GRAPHIC LOG	Sample Description & Classification
			1		0 to 4	48/				**FILL** Brown/gray silty clay, trace fine grained sand, little concrete, moist
580	5		2		4 to 8	48/26		OVM=0.2 OVM=1.7		**FILL** as above, brown silty clay with fine grained to coarse grained sand, angular limestone at 1.5', some coal in upper 1' of core, trace root material, dry
575	10		3		8 to 12	48/44		OVM=2.0 OVM=88.4		Fill: Same as above, locally saturated (4'), increasing brown silty clay dry, transitioning to dark brown, gray, and black, trace fine grained to medium grained sand in lower 1', little to small medium subrounded gravel, slight possible solvent odor in lower 1'
570	15		4		12 to 16	48/38		OVM=16.2 OVM=3.1		Dark gray to black silty clay with medium gravel and gray slug, locally saturated, transitions to brown silty clay (reworked) with trace medium rounded gravel, very slight possible solvent odor 12-14', moist
			5		16 to 20	48/48		OVM=1.1 OVM=19.1		Reddish brown silty clay, trace fine to medium sand, dry
565	20		6		20 to 24	48/42		OVM=0.1 OVM=1.1		**Native Alluvium** Black silty fine grained sand, locally saturated, septic odor, with clay, saturated Trace wood material and shell fragments in lower 2'

WELL CONSTRUCTION DETAILS



Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas
PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Boring Location

NORTHING: 1,067,270 **EASTING:** 1,064,263 **STATION:** N/A **OFFSET:** N/A

HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A

VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 584.19

LOCATION: Lower Scajaquada Creek Area

BORING

MW-LSC-3

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	SAMPLE INFORMATION: Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	GRAPHIC LOG	Sample Description & Classification	WELL CONSTRUCTION DETAILS
560			7		24 to 28	48/47		OVM=0.3		**Lacustrine Clay** Reddish brown silty clay, trace fine sand, (locally high plasticity), moist	
	25							OVM=0.6			
			8		28 to 32	48/		OVM=0.4		Same as above, little fine to coarse angular sand, trace angular small gravel, saturated	- 3/8" dia. bentonite chips
555	30							OVM=0.5			
										End of Boring at 32 feet	
550	35									Samples to Alpha Analytical: 11-12' below ground surface (573.19 to 571.19' elev.) 22-24' below ground surface (562.19 to 560.19' elev.)	(1) Longer well screen selected to monitor groundwater in fill with solvent odor and material above clay.
545	40										
540	45										
535	50										
530											

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CLIENT: National Fuel Gas

PROJECT NAME: National Fuel Lower Scajaquada Creek

CITY/STATE: Buffalo, New York

GEI PROJECT NUMBER: 2101620

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100 Sylvan Parkway
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Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Boring Location

NORTHING: 1,067,415 **EASTING:** 1,064,262 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 582.37
LOCATION: Lower Scajaquada Creek Area

BORING

MW-LSC-4

Drilling Information

DATE START / END: 11/8/2021 - 11/8/2021 **TOTAL DEPTH (FT):** 32.0
CONTRACTOR: Nothnagle **DRILLER:** Tom Mangefreda **LOGGED BY:** M. Cummings
EQUIPMENT: Hand/Geoprobe 7822DT **BORING METHOD:** Hand/Geoprobe
AUGER ID/OD: 4.25 in / N/A **CASING ID/OD:** 2 in / 2 in
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft):
GENERAL NOTES: Top of Riser Elevation: 582.13'

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_P = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	GRAPHIC LOG	Sample Description & Classification
			1		0 to 4	48/				Topsoil **FILL** Brown silt, sand, fine grained to medium grained, well rounded, rounded, gravel fine to coarse, subrounded to angular, cobbles
580			2		4 to 8	48/32		OVM=0.0		Black silty sand, fine grained to coarse grained, dry, organics, medium gravel, slag and black coal, root material
	5							OVM=0.0		
575			3		8 to 12	48/6				Brown silty clay fill, fabric or fiber materials at 4.25', brick fragments, trace small angular gravel and cinders, dry Very low recovery, some material as above (4-8'), some black to gray silty clay with fine grained sand at end 1", moist
	10									
570			4		12 to 16	48/44		OVM=0.1		**Native Alluvium** Dark gray to black silty clay, moist
								OVM=0.1		Dark gray to black, some olive color
	15									Clayey silt, trace fine grained sand, saturated at 14.5'
			5		16 to 20	48/34		OVM=0.1		Same as above, dark gray to black clayey silt, trace fine grained sand
565								OVM=0.1		Silty fine gravel, sand with gastropod shells, moist
	20		6		20 to 24	48/44		OVM=0.2		Silty fine to coarse grained sand, some wood material upper 6", gastropod shells throughout, saturated
560								OVM=0.4		Root organics at 22'

WELL CONSTRUCTION DETAILS

- Flush mount surface casing
- 2" dia. schedule 40 PVC well riser
- Cement/ bentonite grout
- 3/8" dia. bentonite chips
- 2" dia. schedule 40 PVC well screen
- #00N Filter Sand

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

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GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Boring Location

NORTHING: 1,067,415 **EASTING:** 1,064,262 **STATION:** N/A **OFFSET:** N/A

HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A

VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 582.37

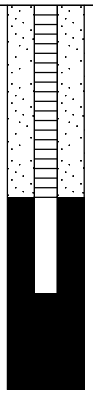
LOCATION: Lower Scajaquada Creek Area

BORING

MW-LSC-4

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	SAMPLE INFORMATION: Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	GRAPHIC LOG	Sample Description & Classification
555	25		7		24 to 28	48/47		OVM=55.6		Grades to small gravel, angular and more coarse grained sand at 24'
								OVM=80.4		Dark brown fine to coarse grained sand and gravel, silt, coal tar-like odors throughout, angular small to medium grained, liquid tar-like material present, saturated
			8		28 to 32	48/19		OVM=1.9		Lacustrine Clay Reddish brown silty clay with trace fine angular gravel, dry,tar-like material impacts and odors on outside of clay only.
	30							OVM=1.7		Reddish brown silty clay as above, trace fine grained sand, low plasticity fines, stiff, moist
550										End of Boring at 32 feet
	35									Samples to Alpha Analytical:
										20-22' below ground surface (562.37' to 560.37' elev.)
545										24-28' below ground surface (558.37' to 556.37' elev.)
	40									Samples for forensics (Meta Env.)
										24-25' below ground surface (558.37' to 557.37' elev.)
540										
	45									
535										
	50									
530										

WELL CONSTRUCTION DETAILS



Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas

PROJECT NAME: National Fuel Lower Scajaquada Creek

CITY/STATE: Buffalo, New York

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GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Boring Location

NORTHING: 1,067,445 **EASTING:** 1,064,113 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 582.72
LOCATION: Lower Scajaquada Creek Area

BORING

MW-LSC-5

Drilling Information

DATE START / END: 1/4/2022 - 1/4/2022 **TOTAL DEPTH (FT):** 42.0
CONTRACTOR: Nothnagle **DRILLER:** Tom Mangefreda **LOGGED BY:** M. Cummings
EQUIPMENT: Hand/Geoprobe 7822DT **BORING METHOD:** Hand/Geoprobe
AUGER ID/OD: 4.25 in / N/A **CASING ID/OD:** 2 in / 2 in
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft):
GENERAL NOTES: Top of Riser Elevation: 582.46'

ABBREVIATIONS: ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length; bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample; U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core; WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter; Q_P = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	Sample Description & Classification
									Hand clear to 11' bgs, bricks, gravel, concrete. Advance augers to 5.0' without sampling.
580	5		1		5 to 8	36/3			**FILL** Loose angular crushed limestone gravel, moist
575									Steady grinding at 8.5' bgs (likely concrete basement flooring) grind through 6" then advance HSA to10 bgs without sampling.
	10		2		10 to 14	48/13		OVM=0.0	Native Alluvium Gray to light gray silty fine sand, little black wood pieces, firm, moist throughout
570			3		14 to 18	48/37		OVM=0.0	Gray fine sand and silt with little low plasticity fines, moist throughout, rootlets bottom 6", firm
	15								
565			4		18 to 22	48/42		OVM=0.0	Gray to dark gray clayey silt with trace fine sand, shell fragments (white) at 21' with few shells, scattered throughout, little peat and cobbles
	20								Gray silty fine sand, saturated, soft, slight petroleum odor
560			5		22 to 26	48/48		OVM=2.7	

WELL CONSTRUCTION DETAILS

- Flush mount surface casing
- 2" dia. schedule 40 PVC well riser
- Cement/ bentonite grout
- 3/8" dia. bentonite chips

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas
PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Boring Location

NORTHING: 1,067,445 **EASTING:** 1,064,113 **STATION:** N/A **OFFSET:** N/A

HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A

VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 582.72

LOCATION: Lower Scajaquada Creek Area

BORING

MW-LSC-5

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	SAMPLE INFORMATION: Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blow Count or RQD	Field Test Data	GRAPHIC LOG	Sample Description & Classification	WELL CONSTRUCTION DETAILS
555	25		6		26 to 30	48/32		OVM=2.7 OVM=26		As above, saturated, then 6", wood fragments from 28.5-30', coarse fine sand, silt, slight coal-tar like odor, gray clay in shoe with tar-like material staining, firm, 1' red clay in very bottom of shoe	2" dia. schedule 40 PVC well screen #00N Filter Sand
550	30		7		30 to 34	48/24		OVM=0.4		Silty red clay with trace fine angular gravel, firm, medium to high plasticity, slight coal-tar like odor in top 1.5'	
545	35		8		34 to 38	48/48		OVM=0.0		As above, little coal tar present on shoe only, likely resulting from withdrawing sampling assembly through impacted interval above, little red clay in sampler	2' sump
540	40		9		38 to 42	48/2		OVM=0.0		Poor recovery, little red clay in sampler	3/8" dia. bentonite chips
535	45									End of Boring at 42 feet Samples to Alpha Analytical: 18-22' below ground surface (564.72 to 560.72 elev.) 26-30' below ground surface (556.72 to 552.72 elev.)	
530	50										

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CLIENT: National Fuel Gas

PROJECT NAME: National Fuel Lower Scajaquada Creek

CITY/STATE: Buffalo, New York

GEI PROJECT NUMBER: 2101620

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GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/15/22

Appendix B.3

SC Sediment Boring Logs

Boring Location

NORTHING: 1,067,369 **EASTING:** 1,063,785 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 563.2
LOCATION:

BORING

SED-BGL-1

Drilling Information

DATE START / END: 11/10/2021 - 11/10/2021 **TOTAL DEPTH (FT):** 11.5
CONTRACTOR: Nothnagle **DRILLER:** Kevin Busch **LOGGED BY:** E. Wood
EQUIPMENT: Barge/Geoprobe **BORING METHOD:** Geoprobe
AUGER ID/OD: N/A / N/A **CASING ID/OD:** 1.5 in / N/A
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft): Surface water elevation 573.0'
GENERAL NOTES: Barge measuring point 1.2' above surface water- 574.2'

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_p = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Lab Sample Depth	Field Test Data	GRAPHIC LOG	Sample Description & Classification
			1		0 to 4	48/24	Alpha Alpha	OVM=0.3		**Recent Fluvial Deposits** Black silt and organics, mussels shells, septic odor, saturated.
560								OVM=0.4		**Lacustrine Clay** Reddish-brown silty clay, trace to little fine to medium subrounded gravel throughout, moist
	5		2		4 to 8	48/48		OVM=0.2		As above, slightly more sand content, moist throughout.
555								OVM=0.3		
	10		3		8 to 11.5	42/48		OVM=0.2		
								OVM=0.1		probe refusal at 11.5' bgs in lacustrine clay unit.
550										End of Boring at 11.5 feet
	15									
545										
	20									
540										

Water Depth: 9.8'

Samples to Alpha Analytical:

0-0.5' below top of sediment (563.2' to 562.7' elev.)

12-14' below MP (562.2' to 560.2' elev.)

No forensics collected

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas
PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/11/22

Boring Location

NORTHING: 1,067,768 **EASTING:** 1,063,771 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 567.6
LOCATION:

BORING

SED-LSC-1

Drilling Information

DATE START / END: 11/9/2021 - 11/9/2021 **TOTAL DEPTH (FT):** 22.5
CONTRACTOR: Nothnagle **DRILLER:** Kevin Busch **LOGGED BY:** E. Wood
EQUIPMENT: Barge/Geoprobe **BORING METHOD:** Geoprobe
AUGER ID/OD: N/A / N/A **CASING ID/OD:** 1.5 in / N/A
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft): Surface water elevation 572.9'
GENERAL NOTES: Barge measuring point 1.2' above surface water- 574.1

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_p = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Lab Sample Depth	Field Test Data	GRAPHIC LOG	Sample Description & Classification
			1		0 to 2.5	30/3	Alpha	OVM=0.4 OVM=0.1		**Recent Fluvial Deposits** Black, very soft silt, very slight suspect petroleum-like odor, some fine grained to medium grained sand, subrounded, saturated, no sheen
565			2		2.5 to 6.5	48/5		OVM=23.8		Same as above, no gravel present, suspect petroleum odors still present
	5						Alpha	OVM=43.1		
			3		6.5 to 10.5	48/28		OVM=27.3		Same as above, some fine grained to coarse grained sand, trace medium angular/subangular gravel, black, stronger petroleum-like odor, saturated with sheen
560								OVM=18.8		**Historic Alluvial Deposits** Transition to reddish/gray silt with clay, some fine sand in bottom 3", very slight suspect petroleum odor
	10		4		10.5 to 14.5	48/22		OVM=35.1		Dark brown to gray sand, fine grained to coarse grained, subrounded to angular with silt and small medium angular gravel, saturated, petroleum sheen and odors present
555								OVM=13.6		Gray silty sand with fine grained to coarse grained sand and gravel, odors, saturated, dark brown staining and suspect tar-like material in core catcher
	15		5		14.5 to 18.5	48/33	Meta	OVM=2.7		3" coarse alluvium (fine gravel and coarse sand) **Lacustrine Clay** Reddish brown silty clay, trace fine grained to coarse grained sand and fine gravel, subrounded, moist
550								OVM=1.4		
			6		18.5 to 22.5	48/		OVM=0.8		Same as above, slight septic/organic odor.
	20							OVM=0.6		
545										End of Boring at 22.5 feet

Water Depth: 5.3'

Samples to Alpha Analytical:

0-0.5' below top of sediment (567.6'-567.1' elev.)

11-15' below MP (563.1'-559.1' elev.)

Samples for forensics (Meta Env.)

20-22' below MP (554.1' to 553.1' elev.)

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

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CITY/STATE: Buffalo, New York
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GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/11/22

Boring Location

NORTHING: 1,067,693 **EASTING:** 1,063,840 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 568.0
LOCATION:

BORING

SED-LSC-2

PAGE 1 of 1

Drilling Information

DATE START / END: 11/9/2021 - 11/9/2021 **TOTAL DEPTH (FT):** 20.0
CONTRACTOR: Nothnagle **DRILLER:** Kevin Busch **LOGGED BY:** E. Wood
EQUIPMENT: Barge/Geoprobe **BORING METHOD:** Geoprobe
AUGER ID/OD: N/A / N/A **CASING ID/OD:** 1.5 in / N/A
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft): Surface water elevation 572.9'
GENERAL NOTES: Barge measuring point 1.2' above surface water- 574.1'

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_p = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	SAMPLE INFORMATION: Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Lab Sample Depth	Field Test Data	GRAPHIC LOG	Sample Description & Classification
			1		0 to 5	60/2	Alpha			**Recent Fluvial Deposits** Single piece of wood, black, no odors.
565	5		2		5 to 8	36/15		OVM=108.2		Wood material in upper 6" Black sand, fine grained to coarse grained, subangular, subrounded, small subangular gravel Grading to silty fine to coarse sand, some clay, black, some sandy subangular gravel, strong petroleum (suspect gasoline) odors and brown liquid
							Meta	OVM=199.4		
560			3		8 to 12	48/31	Alpha	OVM=101.6		**Historic Alluvial Deposits** Reddish brown silty clay with brown staining on exterior, NAPL blebs within core
	10							OVM=20.7		
			4		12 to 16	48/18				as above, suspect tar-like material at base of core. Gray/reddish brown, moist, petroleum odors, staining
555										**Lacustrine Clay** Reddish brown silty clay, stiff, moist, no odors/staining
	15		5		16 to 20	48/48		OVM=0.4		Same as above, no evidence of impacts, moist
550								OVM=0.2		
	20									End of Boring at 20 feet
545										

Water Depth: 4.8'

Samples to Alpha Analytical:

0-0.5' below top of sediment (568.0' to 567.5' elev.)

14-16' below MP (560.1' to 558.1' elev.)

Samples for forensics (Meta Env.)

13-14' below MP (561.1' to 560.1' elev.)

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas
PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/11/22

Boring Location

NORTHING: 1,067,522 **EASTING:** 1,063,919 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 566.3
LOCATION:

BORING

SED-LSC-3

Drilling Information

DATE START / END: 11/9/2021 - 11/9/2021 **TOTAL DEPTH (FT):** 21.5
CONTRACTOR: Nothnagle **DRILLER:** Kevin Busch **LOGGED BY:** E. Wood
EQUIPMENT: Barge/Geoprobe **BORING METHOD:** Geoprobe
AUGER ID/OD: N/A / N/A **CASING ID/OD:** 1.5 in / N/A
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft): Surface water elevation 572.9'
GENERAL NOTES: Barge measuring point 1.2' above surface water- 574.1'

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_p = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Lab Sample Depth	Field Test Data	GRAPHIC LOG	Sample Description & Classification
565	5		1		0 to 5.5	66/5	Alpha	OVM=2.4		**Recent Fluvial Deposits** Black silt, trace gravel and fine grained sand, subrounded, saturated with sheen and slight petroleum-like odor
			2		5.5 to 9.5	48/15	Met	OVM=29.8 OVM=30.9		Same as above Grades to silty sand, black, fine grained to coarse grained, subrounded with small gravel, coal tar-like odor, saturated with sheen
560	10		3		9.5 to 13.5	48/13	pha	OVM=398		Same as above
555	15		4		13.5 to 17.5	48/48		OVM=100.2 OVM=8.2 OVM=27.2		**Lacustrine Clay** Reddish brown silty clay, stiff, moist, moderate coal tar-like odor, no staining, slight sheen on water in core. Same as above, slightly gray color with slight coal tar-like odor
550	20		5		17.5 to 21.5	48/48		OVM=1.2 OVM=1.1		Same as above, moist
545										End of Boring at 21.5 feet

Water Depth: 6.6'

Samples to Alpha Analytical:

0-0.5' below top of sediment (567.5' to 567.0' elev.)

13-16' below MP (561.1 to 558.1' elev.)

Samples for forensics (Meta Env.)

14-15' below MP (560.1 to 559.1' elev.)

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas
PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/11/22

Boring Location

NORTHING: 1,067,408 **EASTING:** 1,063,987 STATION: N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 GROUND SURFACE ELEVATION (FT): 564.7
LOCATION:

BORING

SED-LSC-4

Drilling Information

DATE START / END: 11/10/2021 - 11/10/2021 **TOTAL DEPTH (FT):** 16.0
CONTRACTOR: Nothnagle **DRILLER:** Kevin Busch **LOGGED BY:** E. Wood
EQUIPMENT: Barge/Geoprobe **BORING METHOD:** Geoprobe
AUGER ID/OD: N/A / N/A **CASING ID/OD:** 1.5 in / N/A
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft): Surface water elevation 572.9'
GENERAL NOTES: Barge measuring point 1.2' above surface water- 574.1'

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_p = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Lab Sample Depth	Field Test Data	GRAPHIC LOG	Sample Description & Classification
			1		0 to 4	48/9	Alpha	OVM=0.1		**Recent Fluvial Deposits** Black silt, organic material with fine grained subangular sand and small subangular gravel Grades to fine to coarse subangular, subrounded gravel with silty sand, saturated with coal tar-like odors, staining
560	5		2		4 to 8	48/11	Meta Alpha	OVM=4.4		3" same as above, sheen, saturated **Lacustrine Clay** Reddish brown silty clay, moist, sheen on exterior of core, coal tar-like odor, no staining within core
555	10		3		8 to 12	48/48		OVM=0.4		Same as above, coal tar-like odors and sheen on water on exterior of core but no evidence within core, moist Same as above, no sheen/odors, moist
			4		12 to 16	48/48		OVM=12.0 OVM=0.2		
550	15							OVM=1.1		Little fine grained to medium grained well rounded sand (6") End of Boring at 16 feet
545	20									

Water Depth: 8.3'

Samples to Alpha Analytical:

0-0.5' below top of sediment (564.7' to 564.2' elev.)

14-16' below MP (560.0' to 558.0' elev.)

Samples for forensics (Meta Env.)

13-14' below MP (559.0 to 560.0' elev.)

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas
PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/11/22

Boring Location

NORTHING: 1,067,332 **EASTING:** 1,064,081 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 565.2
LOCATION:

BORING

SED-LSC-5

Drilling Information

DATE START / END: 11/10/2021 - 11/10/2021 **TOTAL DEPTH (FT):** 17.0
CONTRACTOR: Nothnagle **DRILLER:** Kevin Busch **LOGGED BY:** E. Wood
EQUIPMENT: Barge/Geoprobe **BORING METHOD:** Geoprobe
AUGER ID/OD: N/A / N/A **CASING ID/OD:** 1.5 in / N/A
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft): Surface water elevation 572.9'
GENERAL NOTES: Barge measuring point 1.2' above surface water- 574.1'

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_p = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength
NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Lab Sample Depth	Field Test Data	GRAPHIC LOG	Sample Description & Classification
565			1		0 to 1	12/8	Alpha	OVM=8.3		
			2		1 to 5	48/16		OVM=10.6		**Recent Fluvial Deposits-** Black to reddish brown silty clay with fine grained to coarse grained sand, subangular, small subangular gravel, saturated
								OVM=14.0		Same as above, slight coal tar-like odor and sheen, decreasing sand and gravel content.
560	5		3		5 to 9	48/34	Meta / Alpha	OVM=55.3		Blebs of tar-like material and strong coal tar -like odor within sample core, saturated with sheen Black fine to medium silty sand, trace clay, sheen, saturated Same as above,tar-like material and coal tar-like odors
			4		9 to 13	48/48		OVM=1.0		**Lacustrine Clay-** Reddish brown silty clay, moist, slight gray seaming (6") with strong coal tar-like odors, sheen on exterior of core, slight coal tar odors on sampler and water likely from passing through impacted interval above. Trace small gravel at 10'. (dropstone?)
555	10									No apparent impact.
			5		13 to 17	48/48		OVM=0.0		As above, with little fine grained to medium grained sand, moist
550	15							OVM=7.6		
										End of Boring at 17 feet
545	20									

Water Depth: 7.8'

Samples to Alpha Analytical:

0-0.5' below top of sediment (565.2' to 564.7' elev.)

14-16' below MP (560.0' to 558.0' elev.)

Samples for forensics (Meta Env.)

14-15' below MP (560.0' to 559.0' elev.)

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas
PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

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Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/11/22

Boring Location

NORTHING: 1,067,382 **EASTING:** 1,064,317 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 564.7
LOCATION:

BORING

SED-LSC-6

Drilling Information

DATE START / END: 11/3/2021 - 11/3/2021 **TOTAL DEPTH (FT):** 16.0
CONTRACTOR: SJB **DRILLER:** Art Koske **LOGGED BY:** E. Wood
EQUIPMENT: Barge/Geoprobe **BORING METHOD:** Geoprobe
AUGER ID/OD: N/A / N/A **CASING ID/OD:** 3 in / N/A
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft): Surface water elevation 573.2'
GENERAL NOTES: Measuring point is water surface

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_p = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Lab Sample Depth	Field Test Data	GRAPHIC LOG	Sample Description & Classification
			1		0 to 2	24/1	Alpha	OVM=0		**Recent Fluvial Deposits** little black angular gravel, mussel shells, loose.
			2		2 to 4	24/2		OVM=8.7		as above, slight coal tar-like odor.
560	5		3		4 to 6	24/10	Meta	OVM=13		black silty fine to medium sand with fine angular gravel, suspect tar-like material staining and strong coal tar-like odor.
			4		6 to 8	24/6		OVM=18		medium to coarse grey sand with medium subangular gravel, loose, NAPL blebs, moderate coal tar-like odor, saturated.
			5		8 to 10	24/6		OVM=37		**Historic Alluvial Deposits** fine gray sand, loose, trace silt. Suspect tar-like material staining, blebs and moderate coal tar-like odor, saturated.
555	10		6		10 to 12	24/12	Alpha	OVM=18.3		as above to 11.5' bgs with tar-like material staining.
			7		12 to 14	24/8		OVM=--		**Lacustrine Clay** reddish brown silty clay, firm. Coal tar on outside of sampler from passing through impacted interval above.
550	15		8		14 to 16	24/0		OVM=--		
										End of Boring at 16 feet
545	20									

Water Depth: 8.5'

Samples to Alpha Analytical:

0-0.5' below top of sediment (564.7' to 564.2 elev.)

12-19' below MP (561.2' to 553.7' elev.)

Samples for forensics (Meta Env.)

12-19' below MP (561.2' to 553.7' elev.)

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas
PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

GEI Consultants, Inc.
100 Sylvan Parkway
Suite 400
Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/11/22

Boring Location

NORTHING: 1,067,564 **EASTING:** 1,064,457 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 565.8
LOCATION:

BORING

SED-LSC-7

Drilling Information

DATE START / END: 11/2/2021 - 11/2/2021 **TOTAL DEPTH (FT):** 16.0
CONTRACTOR: SJB **DRILLER:** Art Koske **LOGGED BY:** M. Cummings
EQUIPMENT: Barge/Geoprobe **BORING METHOD:** Geoprobe
AUGER ID/OD: N/A / N/A **CASING ID/OD:** 3 in / N/A
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft): Surface water elevation 573.8'
GENERAL NOTES: Measuring point is water surface

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
bpf = Blows per Foot; mpf = Minute per Foot; S = Split Spoon; DP = Direct Push Sample
U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_p = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Lab Sample Depth	Field Test Data	GRAPHIC LOG	Sample Description & Classification
565			1		0 to 2	24/6	Alpha	OVM=0.5		**Recent Fluvial Deposits** black detrital matter, sticks, zebra mussels, leaves, little medium rounded gravel, loose, light sheen 2"-6" Gray silty fine sand, firm, light petroleum-type odor, no visual impacts
			2		2 to 4	24/0				
	5		3		4 to 6	24/11		OVM=6.9		Gray/brown mottled sandy silt, grading to gray silty sand with rounded medium gravel, moist throughout, sheens throughout, light staining and coal tar-like odor
560			4		6 to 8	24/11	Meta	OVM=19.4		**Historic Fluvial Deposits-** Fine to medium gray sand with gravel (fine to medium rounded), saturated, suspect tar-like material, sheen, few NAPL blebs, strong coal tar-like odor Dark gray to black silty sand with gravel, some clay, "blebs" of brown suspect tar-like material with petroleum-like odor, heavy staining
			5		8 to 10	24/16		OVM=18		
	10		6		10 to 12	24/5		OVM=10		**Lacustrine Clay-** Reddish brown silty clay with few pieces of angular medium gravel with suspect tar-like material within upper clay unit.
555			7		12 to 14	24/12	Alpha			
			8		14 to 16	24/2				
	15									End of Boring at 16 feet
550										
	20									
545										

Water Depth: 8.0'

Samples to Alpha Analytical:

0-0.5' below top of sediment (565.8' to 565.3' elev.)

12-18' below MP (561.8' to 553.8' elev.)

Samples for forensics (Meta Env.)

14-16' below MP (559.8' to 557.8' elev.)

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

CLIENT: National Fuel Gas
PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

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Buffalo, NY 14228

GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/11/22

Boring Location

NORTHING: 1,067,750 **EASTING:** 1,064,582 **STATION:** N/A **OFFSET:** N/A
HORIZONTAL DATUM: NAD83 NY West Zone **STATION CENTERLINE:** N/A
VERTICAL DATUM: NAVD88 **GROUND SURFACE ELEVATION (FT):** 566.6
LOCATION:

BORING

SED-LSC-8

Drilling Information

DATE START / END: 11/1/2021 - 11/1/2021 **TOTAL DEPTH (FT):** 12.2
CONTRACTOR: SJB **DRILLER:** Art Koske **LOGGED BY:** M. Cummings
EQUIPMENT: Barge/Geoprobe **BORING METHOD:** Geoprobe
AUGER ID/OD: N/A / N/A **CASING ID/OD:** 3 in / N/A
HAMMER TYPE: Direct Push **HAMMER WEIGHT (lbs):** N/A **HAMMER DROP (inch):** N/A
WATER LEVEL ELEVATIONS (ft): Surface water elevation 573.4'
GENERAL NOTES: Measuring point is water surface

ABBREVIATIONS:
ID = Inside Diameter; OD = Outside Diameter; Pen. = Penetration Length; Rec. = Recovery Length
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U = Undistrubed Tube Sample; C = Rock Core; V = Field Vane Shear; SC = Sonic Core
WOR = Weight of Rods; WOH = Weight of Hammer; RQD = Rock Quality Designation; OVM = Organic Vapor Meter
Q_p = Pocket Penetrometer Strength; S_v = Pocket Torvane Shear Strength; F_v = Field Vane Shear Strength; NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Lab Sample Depth	Field Test Data	GRAPHIC LOG	Sample Description & Classification
565	0		1		0 to 2.2	26.4/8	Alpha	OVM=0.1 OVM=1.4		**Recent Fluvial Deposits** Medium angular to rounded gravel, organics, fine to coarse sand, saturated.
			2		2.2 to 4.2	24/3		OVM=24.1		black silty clay, saturated, slight petroleum-like odor.
	5		3		4.2 to 6.2	24/3	Alpha & Meta	OVM=11.1		
560			4		6.2 to 8.2	24/7		OVM=6.9		as above with petroleum-like odor.
			5		8.2 to 10.2	24/10		OVM=4.7		
	10		6		10.2 to 12.2	24/12		OVM=6.2		**Historic Alluvium** Gray clayey silt, moist, trace fine sand. **Lacustrine Clay** Reddish grey silty clay, trace find sand.
555										End of Boring at 12.2 feet
	15									
550										
	20									
545										

Water Depth: 6.8'

Samples to Alpha Analytical:

0-0.5' below top of sediment (566.6' to 566.1' elev.)

6.8 to 15.0' below MP (566.6 to 558.4' elev.)

Samples for forensics (Meta Env.)

6.8 to 15.0" below MP (566.6' to 558.4' elev.)

Strata lines represent the approximate boundaries between soil types. Actual transitions may be gradual. Water level readings have been made at times stated. Water levels may be different at other times.

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PROJECT NAME: National Fuel Lower Scajaquada Creek
CITY/STATE: Buffalo, New York
GEI PROJECT NUMBER: 2101620

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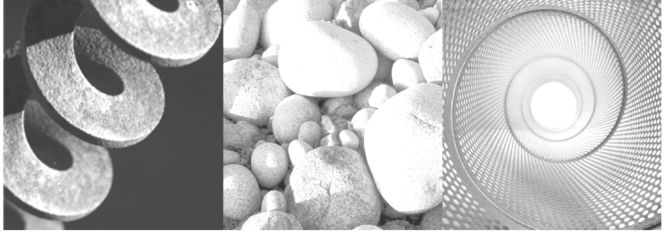
GLASTONBURY - GEOTECHNICAL BORING LOG 02 2101620_NATIONAL FUEL LOWER SCAJAQUADA CREEK SITE_EW.GPJ NEW.GDT 4/11/22

Appendix B Field Sampling Plan



GEI Consultants

Appendix B: Field Sampling Plan Remedial Investigation/Feasibility Study - GLLA Program

Lower Scajaquada Creek Operable Unit, Buffalo, New York

Submitted to:
National Fuel Gas Distribution Corporation
6363 Main Street
Williamsville, New York 14221

Submitted by:
GEI Consultants, Inc.
100 Sylvan Parkway, Suite 400
Amherst, New York 14228
716.204.7154

June 2025
Project No. 2406810

Richard H. Frappa, P.G.
Senior Professional

Michael A. Cummings, P.G.
Senior Professional

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Abbreviations and Acronyms

ASTM	American Society for Testing and Materials
bgs	below ground surface
CAMP	Community Air Monitoring Plan
COC	Constituent of Concern
DNAPL	Dense Non-Aqueous Phase Liquid
EPA	United States Environmental Protection Agency
FSP	Field Sampling Plan
GEI	GEI Consultants, Inc., P.C.
GPS	Global Positioning System
HASP	Health and Safety Plan
HSA	Hollow-Stem Auger
ID	Inner Diameter
IDW	Investigation-Derived Waste
LNAPL	Light Non-Aqueous Phase Liquid
MGP	Manufactured Gas Plant
NAPL	Non-Aqueous Phase Liquid
NTU	Nephelometric Turbidity Unit
PCB	Polychlorinated Biphenyl
PID	Photo-ionization Detector
PPE	Personal Protection Equipment
PVC	Polyvinyl Chloride
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
RI/FS	Remedial Investigation/Feasibility Study
SVOC	Semi-Volatile Organic Compound
TAL	Target Analyte List
TCL	Target Compound List
TCLP	Toxicity Characteristic Leaching Procedure
USCS	Unified Soil Classification System
VOC	Volatile Organic Compound

1. Introduction

This Field Sampling Plan (FSP) presents the methods and procedures to be used for performing field activities during the Remedial Investigation/ Feasibility Study (RI/FS) at the Lower Scajaquada Creek Operable Unit in Buffalo, New York.

1.1. Overview of Field Activities

The following field activities will be performed:

- Reconnaissance – A reconnaissance will be performed to observe site conditions.
- Utility Locating – Subsurface utilities will be identified and marked in the field with cooperation from
- Soil Boring Advancement and Soil Sampling –Soil borings will be advanced using rotary drilling methods. Soil samples will be collected using Macrocore or 2-foot split spoon samplers
- Monitoring Well Installation and Groundwater Sampling – Some of the above-described soil borings will be converted into 2-inch diameter monitoring wells installed in the overburden. Each newly-installed well will be developed after which groundwater samples will be collected.
- Sediment Borings- Sediment borings will be completed within Scajaquada Creek and the Scajaquada Creek Inlet between West Ave. and the Black Rock Canal. The borings will be advanced using either rotary or direct-push methods by a drill rig mounted on a barge.
- Surface Water Elevation Monitoring – Surface elevation data will be obtained at a few locations along Scajaquada Creek.
- Surveying – The locations and elevations of each RI/FS investigation location including soil borings, monitoring wells and sediment investigation locations will be surveyed along with relevant Site features.

2. General Field Guidelines

2.1. Site Hazards

Potential on-site surface hazards, such as traffic, uneven work surfaces, sharp objects, work on and near water and overhead power lines will be identified prior to initiation of the field work. The potential hazards at the site will be identified during a site reconnaissance by the project team on the first day of the investigation field activities. Additional safety measures to be undertaken for the work performed during the investigation are described in the site-specific Health and Safety Plan (HASP).

2.2. Underground Utilities

Underground utilities, including electric lines, gas lines, storm and sanitary sewers, and communication lines will be identified prior to initiation of drilling and other subsurface work. Underground utility location will be accomplished as follows:

- All proposed investigation locations not occurring within Scajaquada Creek will be flagged or marked-out with white paint.
- Dig Safely of New York 800-272-4480 will be contacted to initiate the locating activities. New York State law requires that Dig Safely of New York be notified at least two working days, and not more than 10 working days, before subsurface work is performed.
- As part of the Dig Safely New York utility locating process, GEI will exercise the option to request a Site visit from member utilities to clearly identify the location of buried utilities. Specific attention will be given for the potential for utilities to cross or parallel Scajaquada Creek.
- Companies and municipalities with subsurface utilities present will locate and mark-out all subsurface utility lines.

2.3. Field Log Books

All field activities will be carefully documented in field log books. Entries will be of sufficient detail that a complete daily record of significant events, observations, and measurements is developed. The field log book will provide a legal record of the activities conducted at the site. Accordingly:

- Field books will be assigned a unique identification number.
- Field books will be bound with consecutively numbered pages.
- Field books will be controlled by the Site Manager while field work is in progress.
- Entries will be written with waterproof ink.
- Entries will be signed and dated at the conclusion of each day of field work.
- Erroneous entries made while field work is in progress will be corrected by the field person that made the entries. Corrections will be made by drawing a line through the error, entering the correct information, and initialing the correction.

- Corrections necessary after departing the field will be made by the person who entered the original information. Corrections will be made by drawing a line through the error, entering the correct information, and initialing and dating the time of the correction.

At a minimum, daily field book entries will include the following information:

- Location of field activity
- Date and time of entry
- Names and titles of field team members on site and site contacts
- Names, titles of any site visitors, as well as the date and time entering and leaving the site
- Weather information, for example: temperature, cloud coverage, wind speed, and direction
- Purpose of field activity
- A detailed description of the field work conducted
- Sample media (soil, sediment, groundwater, etc.)
- Sample collection method
- Number and volume of sample(s) taken
- Description of sampling point(s)
- Volume of groundwater removed before sampling
- Preservatives used
- Analytical parameters
- Date and time of collection
- Sample identification number(s)
- Sample distribution (e.g., laboratory)
- Field observations
- All field measurements made during soil screening with the photo-ionization detector (PID), and groundwater sampling (pH, temperature, conductivity, turbidity, etc.)
- References for all maps and photographs of the sampling site(s)
- Information pertaining to sample documentation such as:
    - Dates and method of sample shipments
    - Chain-of-custody record numbers
    - Federal Express or UPS air bill numbers

3. Field Equipment Decontamination and Management of Investigation-Derived Waste

3.1. Decontamination Area

A temporary decontamination area lined with polyethylene sheeting will be constructed for personal decontamination and for decontamination of the drilling and probing equipment. Wash water collected from the decontamination activities will be pumped from the pad with a sump pump or trash pump into 55-gallon drums or a bulk tank; and managed as described in Section 3.3. For land-based soil boring and monitoring well locations, the decontamination area will be located near each investigation location. For sediment-boring locations, the temporary decontamination area will be located on the barge/support craft or on-shore, near the sediment boring transect.

3.2. Equipment Decontamination

The following procedures will be used to decontaminate equipment used during the RI/FS activities:

- All drilling equipment including the drilling rig; augers; bits; rods; tools; split-spoon samplers; soil core collection tubes and tremie pipes will be cleaned with a high-pressure washing unit before beginning work.
- Tools, drill rods, samplers, and augers will be placed on polyethylene plastic sheets following pressure washing.
- All augers, samplers, rods, and tools will be decontaminated between each land-based investigation location.
- The back of the drill rig and all tools, augers, and rods will be decontaminated at the completion of the work and prior to leaving the site.

The following lists materials and methods for decontamination.

Suggested Materials

- Potable water
- Phosphate-free detergent (such as Alconox™ or Simple Green™)
- De-ionized water
- Aluminum foil
- Plastic/polyethylene sheeting
- Plastic buckets and brushes
- Personal protective equipment (PPE) in accordance with the HASP

Procedures

- Prior to sampling, all non-dedicated sampling equipment (bowls, spoons, interface probes, etc.) will be washed with potable water and a phosphate-free detergent (such as Alconox™). Decontamination may take place at the sampling location as long as all liquids are contained in pails, buckets, etc.
- The sampling equipment will then be rinsed with potable water followed by a de-ionized water rinse.
- Between rinses, equipment will be placed on polyethylene sheets or aluminum foil, if necessary. Washed equipment will not be placed directly on the ground.
- Equipment will be wrapped in polyethylene plastic or aluminum foil for storage or transportation from the designated decontamination area to the sampling location.

3.3. Management of Investigation-Derived Waste (IDW)

Management and staging of IDW is identified in Section 6.9 of the Remedial Investigation/ Feasibility Study Work Plan. Drill cuttings, sediments and plastic used for surface containment of spoils and fluids, purge water, drill cuttings, personal protective equipment, and disposable sampling equipment will be containerized in separate 55-gallon drums.

3.3.1. Decontamination Fluids and Development and Purge Water

Hot-water pressure wash, decontamination, and development and purge water will be collected in 55-gallon drums. The storage drums will be labeled as “pending analysis – investigation-derived residual decontamination water” and temporarily staged at 1660 Niagara Street pending characterization and proper disposal.

3.3.2. Soil Cuttings and Creek Sediments

Soil and Creek sediments generated during the advancement of soil and sediment borings will be contained in 55-gallon drums. The drums will be labeled as “pending analysis – investigation-derived residual – soil from drill cuttings”. Drums will be temporarily staged at 1660 Niagara Street pending characterization and proper disposal.

3.3.3. Personal Protective Equipment

All PPE will be placed in 55-gallon drums and temporarily staged at 1660 Niagara Street pending characterization of other IDW.

3.3.4. Disposable Sampling Equipment

All used soil, sediment and groundwater sampling equipment (e.g., Macrocore liners, disposable polyethylene bailer and polypropylene tubing) will be placed in the 55-gallon drums labeled “pending analysis – investigation-derived residual – sampling equipment” and temporarily staged at 1660 Niagara Street pending characterization of other IDW.

3.3.5. NAPL Management

If quantities of NAPL are collected in 5-gallon buckets, it will be transferred to a 55-gallon drum, labeled as “pending analysis – IDW - NAPL ” with appropriate well number and temporarily staged at the location identified in the RI/FS work plan pending characterization for disposal with other IDW.

4. Subsurface Soil and Sediment Sampling Procedures

4.1. Introduction

Subsurface investigation activities to be conducted at the site consist of:

- The advancement of overburden soil borings
- The installation of overburden monitoring wells
- The advancement of sediment borings

Procedures for these activities are described in the following sections.

4.2. Soil Borings and Subsurface Soil Sampling

The following methods will be used during installation of the soil borings:

Required Equipment

- Field book
- Project plans
- PPE in accordance with the HASP
- Stakes, flagging and marking paint
- Plastic bags for soil screening samples
- Tape measure
- Decontamination supplies
- Water level indicator
- PID with a 10.2 or 10.6 eV lamp
- Camera
- Clear tape, duct tape
- Laboratory sample bottles
- Coolers and ice
- Shipping supplies

4.2.1. Overburden Drilling and Geologic Logging Methods

- Soil borings will be advanced using 4-1/4 inch inside diameter hollow stem augers or using a Geoprobe traditional or dual-tube sampling system. Each soil boring will be completed through fill and native soil into low permeability clay exhibiting no visual impact or odors.

- Soil samples will be collected continuously from the ground surface to the bottom of the borings using 4-foot long, 2-inch diameter MacroCore™ samplers, or 2-foot long by 2-inch diameter split-spoon samplers.
- Soil samples retrieved from the borehole will be visually described on the soil boring log for: 1) percent recovery, 2) soil type, 3) color, 4) moisture content, 5) texture, 6) grain size and shape, 7) consistency, 8) visible evidence of staining or other hydrocarbon-related impacts, and 9) any other relevant observations. The descriptions will be in accordance with the USCS and the American Society for Testing and Materials (ASTM) guidelines.
- Immediately after describing the core, a representative portion of the sample will be placed in a re-sealable plastic (e.g., "ziplock") bag filled approximately half full. The bag will be labeled with the boring number and interval sampled.
- After allowing the bagged soil to warm, the tip of the sample probe attached to the PID will be inserted into the bag to measure the headspace for organic vapors.
- Soil remaining after completion of sample description, collection, and field screening will be disposed of properly as IDW.
- All borings will be sealed with bentonite hole plug or cement/bentonite grout following completion.
- All drilling equipment will be decontaminated between each boring in accordance with methods specified in Section 3.2.
- The drilling rig working platform/surface will be lined with plastic such that soil is contained while performing observations, measurements, and sampling.
- The field geologist will log borehole geology and headspace measurements in the field book and the Drilling Record and any other observations (e.g., DNAPL/LNAPL impacted soil; and coal tar-like odors, staining, or sheen, etc.).
- At two locations (SB-RI-9 and SB-RI-11) the soil borings will be advanced will be beneath the area wide clay unit to evaluate extent of DNAPL. Objective and rationale for each soil boring location is summarized on Table 1 in the RI/FS Work Plan.

4.2.2. Overburden Soil Sampling

- The number and frequency of samples to be collected from each boring and the associated analytical parameters are summarized in Table 2 in the RI/FS Work Plan.
- A minimum of two soil samples will be collected from each boring and submitted for laboratory analysis. A sample will be collected from the most apparently impacted interval based on PID screening and field observations. If impacts are not observed, a sample will be collected from the 1-foot interval immediately below the water table, and a second sample will be collected from the bottom of the boring (presumed to be from the upper red-brown clay unit. The analytical program for soil borings is provided in Table 2 of the RI/FS Work Plan.
- Samples for laboratory analyses will be collected directly from the acetate liners, or split spoons. For soil and sediment samples analyzed for VOCs, either Encore or Terracore samplers will be

used to collect and preserve the appropriate sample volume. Samples will be placed into appropriate containers and compacted to minimize headspace and pore space.

- Each soil boring that is not converted into a groundwater monitoring well will be backfilled with bentonite pellets to within 1 foot of ground surface with the remainder backfilled with clean topsoil, or asphalt patch where borings are advanced through pavement.
- The sample containers will be labeled, placed in a laboratory-supplied cooler, and packed with ice. The coolers will then be shipped to the laboratory for analysis.
- If there is a delay of sample shipment due to insufficient samples to warrant overnight delivery, the samples will be stored in a cool, secure place with sufficient ice to maintain a temperature of 4℃.
- Chain-of-custody procedures will be followed as outlined in the Quality Assurance Project Plan (QAPP) using the chain-of-custody form provided by the laboratory.
- The sampling equipment will be decontaminated between samples in accordance with procedures described in Section 3.
- Soil remaining after completion of sample description, collection, and field screening will be managed as IDW described further in Section 6.9 of the RI/FS Work Plan.
- The sample locations, descriptions, and depths will be recorded on the borelogs in the field book.

4.3. Scajaquada Creek and Inlet Sediment Sampling and Probing

Sediment borings will be completed in the Scajaquada Creek main channel and Inlet along 26 transects to evaluate sediment quality. Additionally, surface soil samples will be collected from the Creek bank in the Inlet to evaluate surface soil quality where sheens were observed on surface water near the City of Buffalo Pump House. Sediment and surface soil sampling procedures are outlined below.

4.3.1. Sediment Probing

The shallow sediments at each probe point will be probed (agitated) by hand using a one-inch diameter threaded PVC pipe with a fixed plate on the bottom. Probing will involve the following:

1. Use a survey grade GPS to identify the location of probing location along the transect.
2. Measure the depth to the top of sediment using a tape or graduated rod.
3. A threaded, one-inch diameter white PVC pipe (rod) with a fixed, weighted flat-plate will be used as the probing tool. The PVC rod will be pre-marked at 10 cm intervals to help measure the depth of soft sediment penetration.
4. The probe will be lowered to the depth of the sediment surface measured at that location and gently pushed into the sediment by hand as deep as possible. Record the depth of penetration from the graduated marks on the rod.
5. Agitate the shallow sediment with the probe and observe for the presence of hydrocarbon sheen or NAPL. Sheens will be characterized using a four-point scale as trace, light, moderate, or

heavy/NAPL. Observations of sediment characteristics will be recorded (such as soft, hard, loose, cohesive, sandy, rocky, etc.). It will also be noted if deeper probing liberates sheen.

Probing is generally to be performed from a boat, but where the water is shallow (less than 3 feet deep), or the boat cannot access the probing locations, this task will be performed by wading into the creek

4.3.2. Sediment Sampling

Individual sediment sampling locations are provided on Figures 8 through 10 of the RI Work Plan. Sediment borings will be advanced by a drill-rig mounted to a barge and will be advanced using 3-inch diameters flush-jointed casing or 2-3/4-inch diameter hollow stem augers. Casing or augers are specified because vibracore methods could not achieve the required depth of penetration to assess DNAPL presence above the area-wide clay layer. Sediment samples will be collected using a 4-foot Macrocore sampler or 2" diameter split spoon samplers. A sediment sample recovery goal of 70% has been adopted for sediment borings in accordance with GLLA guidelines. If sediment recovery is below 70%, a 3-inch diameter split spoon sampler can be utilized (in 3-inch diameter casing only). Additionally, sampling methods may be modified to include adjusting the cutting shoe on the sampler, varying core-catcher type, modifying the Macrocore liner type or configuration, and modifying the rate of sampler advancement to achieve the 70% recovery goal.

Upon retrieval of the core barrel the inner liner will be capped, and the liner removed from the outer barrel. The upper portion of the liner without sediment will be carefully drained, cut, and then capped immediately above the top of the sediment. If necessary for handling purposes, the cores will cut into two sub-sections before being delivered to shore. Cores will be transferred from the coring boat to the shore where a core processing area will be established. The cores will be measured and then placed into a trough or stand where they will be cut, split, and then logged and sampled. Each core will be logged and photographs taken. All information regarding soil/sediment type, presence of impacts and intervals sampled will be summarized on the sediment boring log.

Following logging and photographing, the cores will be sampled for laboratory analysis from the target intervals and specified analytes listed in Table 4 of the RI/FS Work Plan. VOC samples will be obtained directly from the cores using laboratory-supplied collection kits. The remainder of the sediment from each sample interval will be placed into a stainless-steel bowl, homogenized, and then transferred to laboratory-supplied jars for the specified analyses. The target intervals are generally the upper two feet of sediment and the most impacted interval observed in the core.

If it is found that the texture and consistency of the surface sediment is such that it cannot be recovered reliably by the core barrel, then a supplemental surface grab sample will be obtained location using a sediment dredge such as a Ponar sampler. This sampler will sample the top 6 inches of sediment. Excess sediment not used for sample analysis will be managed as IDW as described in Section 6.9 of the RI Work Plan.

All downhole drilling equipment including casing, augers, drill rods and samplers will be decontaminated between sediment boring locations using high pressure steam methods.

4.3.3. Creek Bank Soil Sampling

Surface soil samples will be collected from 10 locations identified on Figure 8 of the RI/FS Work Plan to assess potential impact to soils adjacent to the creek in the Inlet that have historically exhibited sheens due to daily fluctuations in surface water levels. GEI surface water monitoring data using a pressure transducer/data logger show typical daily water level fluctuations of approximately two feet between September 2, 2022 and January 24, 2023 with rising trend in typical daily high-water levels (elevation range of approximately 571.5 and 573.5 feet during the monitoring timeframes. Therefore, proposed surface soil samples shown on Figure 8 of the RI/FS Work Plan will be collected from the creek at an elevation of one to two feet higher than a creek elevation of 573 feet. At each surface soil sampling location the soil will be agitated using a dedicated probe (PVC rod or similar) to evaluate for DNAPL/oil presence and the potential for generating sheens on adjacent surface water. Sample collection locations will be prioritized to those areas that exhibit the potential for sheen generation. At each location, a sample will be collected from the 0–2-inch depth interval on the creek bank that is within approximately 1-foot of the typical water's edge. Locations will be determined based on observations such as shore erosion, vegetation growth or sediment type.

Samples for VOC analysis will be collected directly from the creek bank using TerraCore (or equivalent) sampling methods. Samples for other analyses will be collected using dedicated, disposable sampling equipment and homogenized prior to placement in laboratory-supplied containers.

Each surface soil sampling location will be staked in the field and labeled following sample collection for future survey.

4.4. Monitoring Well Installation and Development

The following methods will be used for drilling, installing, and developing the monitoring wells:

Required Equipment

- Field book
- Project plans
- PPE in accordance with the HASP
- Plastic bags for soil screening samples
- Tape measure
- Decontamination supplies
- Electronic oil/water interface probe
- PID
- Camera
- Clear tape, duct tape
- Aluminum foil

- Laboratory sample bottles
- Coolers and ice
- Shipping supplies
- Clear polyethylene disposable bailers (NAPL confirmation)
- Polyethylene disposable bailers (development)
- Polypropylene rope (development)
- Waterra™ pump or other purge pump (development)
- Submersible electric pump (development)
- Stainless steel or glass beakers (development)
- Turbidity meter (development)
- Temperature, conductivity, pH meter (development)

4.4.1. Overburden Monitoring Well Installation

The monitoring wells will be installed according to the following specifications:

- The monitoring well borings will be advanced with 4 ¼-inch ID HSAs
- Wells will be constructed with 2-inch ID, threaded, flush-joint, polyvinyl chloride (PVC) casings and screens.
- Screens will be 10-feet long with 0.02-inch slot openings and will be equipped with a 2-foot long DNAPL sump at the base. In areas where DNAPL-impacted soils were identified during the 2021/2022 site characterization (SC) and no shallow wells are present, monitoring wells MW-LSC-2S-RI and MW-LSC-4S-RI will be installed. The well screens at these locations will be positioned to monitor the upper 5 feet of the saturated zone, approximately at an elevation of 565 feet or 5 feet below the observed water table during drilling. Each well will have a 5-foot screen length to evaluate groundwater quality in the shallow groundwater near the water table and will not utilize a sump below the well screen
- The annulus around the 2-foot long DNAPL sump will be filled with bentonite chips. The bentonite chips will not be placed higher than the top of the sump, so that the well screen will not be blocked.
- The annulus around the screens will be backfilled with clean silica sand having appropriate size (e.g., Morie No. 0) to a minimum height of 2 feet above the top of the screen. Auger flights will be withdrawn as sand is poured in a manner that will minimize hole collapse and bridging.
- A bentonite chip seal with a minimum thickness of 2 feet will be placed above the sand pack. The bentonite seal will be hydrated with clean, potable water before placement of grout above the seal layer.
- The remainder of the annular space will be filled with cement-bentonite grout to ground surface. The grout will be allowed to set for a minimum of 24 hours before wells are developed.

- Each monitoring well will be a flush-mounted installation with a locking cap.
- The concrete seal or pad will be sloped to channel water away from the well, and be deep enough to remain stable during freezing and thawing of the ground.
- The top of the PVC well casing and ground surface will be marked and surveyed to 0.01 foot, and the elevation will be determined relative to a fixed benchmark or datum.
- The measuring point on all wells will be on the innermost PVC casing.
- Monitoring well construction details will be recorded in the field book and on the Monitoring Well Construction Log.

4.4.2. Monitoring Well Development

- After a minimum of 48 hours after installation, each newly-installed monitoring well will be developed using methods described below. The information generated during development will be recorded on the Monitoring Well Development Log (Attachment A).
- Prior to development, the wells will be checked for NAPL presence (LNAPL and DNAPL). If NAPL is encountered, the field observations (i.e., thickness and description) will be documented on the well development forms. If NAPL is present, the monitoring well will be developed using low-flow (peristaltic) development methods to minimize contact of NAPL with the well screen and riser. Low flow development will consist of lowering a section of ¼ or 3/8-inch tubing into the well screen taking caution to keep the tubing intake at least 2-feet above the level of NAPL.
- For wells that do not contain NAPL, surging will be performed periodically, across the lengths of screen in 2-foot increments prior to, at interim periods of pumping, and immediately before the final pumping. Pumping methods may include using a centrifugal, submersible, or peristaltic pump and dedicated polyethylene tubing, using a Waterra™ positive displacement pump and dedicated polyethylene tubing, or other methods at the discretion of the field geologist.
- Water levels will be measured in each well to the nearest 0.01 foot prior to development.
- The wells will be developed until the water in the well is reasonably free of visible sediment (50 nephelometric turbidity units (NTU) or less if possible) or until pH, temperature, and specific conductivity stabilize. Field parameters are considered stable when three consecutive readings are within the stabilization criteria for that parameter. The stabilization criteria are as follows: 3% for conductivity and temperature, and 0.1 unit for pH. A portable nephelometer will be used to make the turbidity measurement.
- Development water will be collected in 5-gallon buckets and transferred to 55-gallon drums per management as described in Section 3.3.
- Following development, wells will be allowed to recover for at least 14 days before groundwater is purged and sampled. All monitoring well development will be performed or overseen by a field geologist and recorded in the field book.

4.4.3. Non-Aqueous Phase Liquid (NAPL) Gauging and Mobility Assessments

NAPL gauging and NAPL mobility assessments will be conducted following monitoring well development and sampling. In each newly-installed monitoring well, NAPL presence will be evaluated using an oil/water interface probe, a clear plastic bailer, a weighted cotton rope, or a weighted PVC rod to measure thickness (depending on whichever field technique is most suitable). NAPL presence and thickness will be documented on Monitoring Well Sampling and Development Forms (Attachment A). Accumulated NAPL will then be removed to the greatest extent possible either using a bottom loading bailer or a peristaltic pump with down-hole tubing. NAPL will be collected in a 5-gallon bucket and containerized as investigation-derived waste, described further in Section 6.9.

Once NAPL has been evacuated from the well, the well will be checked periodically for NAPL presence and/or accumulation. The NAPL will be removed during each monitoring event to assess the rate of NAPL recovery. NAPL mobility assessment checks will be completed according to the schedule in the RI/FS Work Plan. NAPL thickness and the amount of NAPL removed from each well will be recorded on the Monitoring Well Development Forms (Attachment A) for each assessment event.

5. Groundwater Sampling Procedures

5.1. Introduction

Procedures for obtaining samples of groundwater are described in this section. Groundwater samples will be collected using low-flow, low-stress purge and sampling methods.

5.2. Groundwater Sampling

The number of the samples that will be collected for laboratory analysis from each well and the analytical parameters are listed in Section 6.3.3 of the RI/FS Work Plan.

The following method will be used to collect groundwater samples from monitoring wells:

Required Equipment and Supplies

- Field book
- Project plans
- PPE in accordance with the HASP
- Electronic oil/water interface probe
- Disposable polyethylene bailers and low-flow sampling pump
- Polypropylene rope
- Temperature, conductivity, and pH meter
- Turbidity meter
- Flow-through cell
- Decontamination supplies
- Peristaltic or submersible pump capable of achieving low-flow rates (i.e., 0.5 liters per minute or less)
- Plastic tubing
- Plastic sheeting
- PID
- Clear tape, duct tape
- Coolers and ice
- Laboratory sample bottles
- Federal Express or UPS labels

5.2.1. Groundwater Sampling Method

Purging

- Prior to sampling, the static water level and thickness of any LNAPL or DNAPL will be measured to the nearest 0.01 foot from the surveyed well elevation mark on the top of the PVC casing with a decontaminated oil/water interface probe. NAPL thickness will be confirmed using a clear bailer or a weighted string. The measurement will be recorded on the Monitoring Well Sampling and Development Form (Attachment A)
- The probe will be decontaminated between uses.
- Groundwater from the well will be purged until field parameters stabilize. Field parameters are considered stable when three consecutive readings are within the stabilization criteria for that parameter. The stabilization criteria are as follows: 3% for conductivity and temperature, and 0.1 unit for pH. The target turbidity values will be <10 NTUs, if possible.
- The flow rate measurement will be approximately 0.5 liter per minute or less.
- If the parameters do not stabilize, then the next step for the sampling will be to remove up to three well volumes, or to purge wells for at least 1 hour prior to sampling.
- If a well goes dry before the required volumes are removed, it will be allowed to recover and sampled when it recovers sufficiently.
- Purge water will be managed and disposed of properly in accordance with IDW management procedures.

Sampling

- Samples will be collected using dedicated ¼- or ⅜-inch polyethylene tubing.
- Prior to filling the sample bottles, the final temperature, pH, and conductivity will be measured within a flow-through cell. Turbidity will be measured with a hand-held turbidity meter. All measurements will be recorded in the field book.
- Groundwater samples will be collected into the appropriate laboratory-provided sampling containers.
- The sample containers will be labeled, placed in a laboratory-supplied cooler, and packed on ice (to maintain a temperature of 4˚C). The cooler will be shipped overnight or delivered to the laboratory for analysis.
- Chain-of-custody procedures will be followed as outlined in the QAPP using the chain-of-custody form.
- Well sampling data will be recorded on the Monitoring Well Sampling and Development Form (Attachment A).

6. Air Monitoring

6.1. Introduction

Two types of air monitoring will be performed during the site investigation: 1) work zone monitoring for protection of the workers performing the site investigation, and 2) community air monitoring at the perimeter of the work site for protection of the local community.

6.2. Breathing Zone Air Monitoring During Drilling and Sampling

Monitoring of air in the breathing zone within the work site will be conducted periodically during all drilling and sampling activities.

- A PID will be used to monitor for VOCs or other organic vapors in the breathing zone and borehole, and to screen the samples at each investigation location.
- Additional air monitoring may be required as specified in the site-specific HASP.
- The PID readings will be recorded in the field book and on the boring log during drilling activities. The procedure for the PID operation and calibration is included in the HASP. Note that equipment calibration will be performed as often as needed to account for changing conditions or instrument readings. The minimum frequency of calibration is specified in the HASP; more frequent calibration will be performed if spurious readings are observed or there are other problems with the instruments.

6.3. Community Air Monitoring

A Community Air Monitoring Plan (CAMP, Appendix B) has been prepared for the RI/FS Work Plan and requires real-time monitoring for VOCs, particulates (i.e., dust), and MGP-related odors at the downwind perimeter of each designated work area when certain activities are in progress at impacted sites. The community air monitoring is not intended for use in establishing action levels for worker respiratory protection. Rather, its intent is to provide a measure of protection for the downwind community (i.e., any and all off-site receptors including residences and businesses and on-site workers not directly involved with the subject work activities) from potential airborne contaminant releases as a direct result of investigative and remedial work activities. The action levels for community air monitoring require increased monitoring, corrective actions to abate emissions, and/or work shutdown. Additionally, community air monitoring helps to confirm that work activities do not spread contamination off site through the air.

The procedures and action levels for community air monitoring are presented in the CAMP that has been prepared for the Site (Appendix B).

7. Field Instruments and Calibration

All field analytical equipment will be calibrated immediately prior to each day's use and more frequently if required. The calibration procedures will conform to manufacturer's standard instructions. This calibration will ensure that the equipment is functioning within the allowable tolerances established by the manufacturer and required by the project. All instrument calibrations will be documented in the project field book and in an instrument calibration log. Records of all instrument calibration will be maintained by the Field Team Leader. Copies of all of the instrument manuals will be maintained on site by the Field Team Leader. All changes to instrumentation will be noted in the field log book.

The following field instruments will be used during the investigation:

- PID
- Particulate monitors
- pH, specific conductivity, oxidation-reduction potential and temperature meter
- Turbidity meter

7.1. Portable Photo-Ionization Analyzer

- The photo-ionization analyzer will be equipped with either a 10.2 or a 10.6 eV lamp. The PID is capable of ionizing and detecting compounds with an ionization potential of less than 10.6 eV. This accounts for up to 73% of the VOCs on the United States Environmental Protection Agency (EPA) Target Compound List (TCL).
- Calibration must be performed at the beginning of each day of use with a standard calibration gas having a concentration of 100 parts per million of isobutylene. If the unit experiences abnormal perturbation or erratic readings, more frequent or additional calibration will be required.
- All calibration data must be recorded in the project field notebooks.
- A battery check must be completed at the beginning and end of each working day.
- All changes to the PID will be noted in the field notes (such as lamp or filter cleaning or replacement or change of instrument).

7.2. pH Meter

- Calibration of the pH meter must be performed at the start of each day of use, and after very high or low readings as required by this Plan, according to manufacturer's instructions.
- National Institute of Standards and Technology - traceable standard buffer solutions which bracket the expected pH range will be used. The standards will be pH of 4.0, 7.0, and 10.0 standard units.

- The use of the pH calibration must be used to set the meter to display the value of the standard being checked.
- The calibration data must be recorded in the project field book.

7.3. Specific Conductivity Meter and Temperature Probe

- Calibration checks using the conductivity standard must be performed at the start of each day of use, after five to 10 readings or after very high or low readings as required by this Plan, according to manufacturer's instructions.
- The portable conductivity meter must be calibrated using a reference solution of 200 µohms/cm (or the manufacturer's specified concentration) on a daily basis. The date and lot number of the reference solution must be recorded. Readings must be within 5% to be acceptable.
- The thermometer of the meter must be calibrated against the field thermometer on a weekly basis.

7.4. Turbidity Meter

- The calibration of the turbidity meter must be checked and reestablished at the start of each day of use according to manufacturer's instructions.

8. Environmental Sample Analyses

Collection of samples for laboratory analysis will be recorded on sample collection logs (Attachment A). The laboratory samples to be collected during the project specific to each media, including the chemical analyses to be performed and a summary of quality control samples are summarized in Table 2 and Table 4 of the RI/FS Work Plan and the Quality Assurance Project Plan (Appendix C of the RI/FS Work Plan).

Attachment A - Field Forms

DAILY FIELD LOG

Project : x
Client : x
Contractor: x

Date: Mon. Nov 5, 2015
Report No. x
Page: Page 1 of 2
GEI Proj. No. x

Time of Arrival: x a.m. **Departure:** x p.m. **Weather:** 40's F, cloudy

Persons Contacted,Company

GEI Representatives

Purpose of Site Visit: x

Observations

1. x
2. x

x

3. x

4. x
5. x
6. x

GEI Consultants

FIELD OBSERVATION REPORT

Project : x
Client : x
Contractor: x

Date: Mon. Nov 5, 2015
Report No. x
Page: Page 2 of 2
GEI Proj. No. x

By:	Reviewed By:

T:\FIELD FORMS\Field Obs Report\field obs report.doc

GEI Consultants

GROUNDWATER ELEVATION MEASUREMENT FIELD FORM

Project Name:_____ **Personnel:**_____

Location:_____ **Weather Conditions:**_____

Date:_____ **Equipment:** _____

Well ID	Time	DTW Below Measuring Point (ft.)	Comments

Boring Location

NORTHING: _____ **EASTING:** _____ **STATION:** _____ **OFFSET:** NA

HORIZONTAL DATUM: _____ **STATION CENTERLINE:** _____

VERTICAL DATUM: _____ **ESTIMATED GROUND SURFACE ELEV. (FT):** _____

LOCATION: _____

BORING _____

PAGE of

Drilling Information

DATE START / END: _____ **TOTAL DEPTH (FT):** _____

CONTRACTOR: _____ **DRILLER:** _____ **LOGGED BY (Person):** _____

EQUIPMENT: _____ **EXPLORATION TYPE/METHOD:** _____

AUGER ID/OD: _____ **CASING ID/OD:** _____ **CORE INFO:** _____

HAMMER TYPE: _____ **HAMMER WEIGHT (lbs):** _____ **HAMMER DROP (inch):** _____

WATER LEVEL DEPTHS (ft): _____

GENERAL NOTES: _____

ABBREVIATIONS:

ID = Inside Diameter	bpf = Blows per Foot	U = Undistrubed Tube Sample	WOR = Weight of Rods	Q_v = Pocket Penetrometer Strength
OD = Outside Diameter	mpf = Minute per Foot	C = Rock Core	WOH = Weight of Hammer	S_v = Pocket Torvane Shear Strength
Pen. = Penetration Length	S = Split Spoon	V = Field Vane Shear	RQD = Rock Quality Designation	F_v = Field Vane Shear Strength
Rec. = Recovery Length	DP = Direct Push Sample	SC = Sonic Core	PID = Photoionization Detector	NA, NM = Not Applicable, Not Measured

Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	SAMPLE INFORMATION						GRAPHIC LOG	Sample Description & Classification	H_20 Depth	WELL CONSTRUCTION DETAILS
			Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blows Count or RQD	Test Data				

Stratification lines represent approximate boundary between soil types, transitions may be gradual. Water level readings have been made at times and under conditions stated. Fluctuations of groundwater may occur due to other factors than those present at the time measurements were made.

LOGGED BY (Consultant): GEI Consultants

PROJECT NAME: _____

CITY/STATE: _____

GEI PROJECT NUMBER: _____

GEI Consultants

GEI Consultants, Inc.
100 Sylvan Parkway, Suite 400
Amherst, New York
14228

Boring Location

NORTHING: _____ **EASTING:** _____ **STATION:** _____ **OFFSET:** NA

HORIZONTAL DATUM: _____ **STATION CENTERLINE:** _____

VERTICAL DATUM: _____ **ESTIMATED GROUND SURFACE ELEV. (FT):** _____

LOCATION: _____

MONITORING WELL

_____

PAGE of

Drilling Information

DATE START / END: _____ **TOTAL DEPTH (FT):** _____

CONTRACTOR: _____ **DRILLER:** _____ **LOGGED BY (Person):** _____

EQUIPMENT: _____ **EXPLORATION TYPE/METHOD:** _____

AUGER ID/OD: _____ **CASING ID/OD:** _____ **CORE INFO:** _____

HAMMER TYPE: _____ **HAMMER WEIGHT (lbs):** _____ **HAMMER DROP (inch):** _____

WATER LEVEL DEPTHS (ft): _____

GENERAL NOTES: _____

ABBREVIATIONS:

ID = Inside Diameter	bpf = Blows per Foot	U = Undistrubed Tube Sample	WOR = Weight of Rods	Q_v = Pocket Penetrometer Strength
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Pen. = Penetration Length	S = Split Spoon	V = Field Vane Shear	RQD = Rock Quality Designation	F_v = Field Vane Shear Strength
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Elev. (ft)	Depth (ft)	Casing Pen. (bpf) or Core Rate (mpf)	SAMPLE INFORMATION						GRAPHIC LOG	Sample Description & Classification	H_20 Depth	WELL CONSTRUCTION DETAILS
			Sample No.	Type	Depth (ft)	Pen./ Rec. (in)	Blows Count or RQD	Test Data				

Stratification lines represent approximate boundary between soil types, transitions may be gradual. Water level readings have been made at times and under conditions stated. Fluctuations of groundwater may occur due to other factors than those present at the time measurements were made.

LOGGED BY (Consultant): GEI Consultants

PROJECT NAME: _____

CITY/STATE: _____

GEI PROJECT NUMBER: _____

GEI Consultants

GEI Consultants, Inc.
100 Sylvan Parkway, Suite 400
Amherst, New York
14228

Monitoring Well Development / Sampling Log

GEI Consultants

Site Name:	Well ID:

Date_____

Field Personnel_____

Method of Purging/ Sampling_____

Well Depth (ft btoc)_____

Depth to Water (ft btoc)_____

Casing type/dia._____

Well Volume (g)_____

Time	Total Volume (g)	Temp (°F)	pH	Cond (us/cm)	ORP (mV)	D.O. (mg/L)	Turbidity (NTU)	Comments

SAMPLE CONTROL LOG

Project Name: _____ **Laboratory:** _____

Project and Task No.: _____ Page ____ of ____

Sampling Date	Sampling Time	Sample Number (ID)	C.O.C. Number	Analyses Requested	Turnaround Time, Sample Location, Handling, Notes, Chain-of-Custody Remarks, etc. (Duplicate, Blank info, etc.)	Date Sent to Lab	Date Results Due

WELL DECOMMISSIONING RECORD

Site Name:	Well I.D.:
Site Location:	Driller:
Drilling Co.:	Inspector:
	Date:

DECOMMISSIONING DATA

(Fill in all that apply)

OVERDRILLING

Interval Drilled	
Drilling Method(s)	
Borehole Dia. (in.)	
Temporary Casing Installed? (y/n)	
Depth temporary casing installed	
Casing type/dia. (in.)	
Method of installing	

CASING PULLING

Method employed	
Casing retrieved (feet)	
Casing type/dia. (in.)	

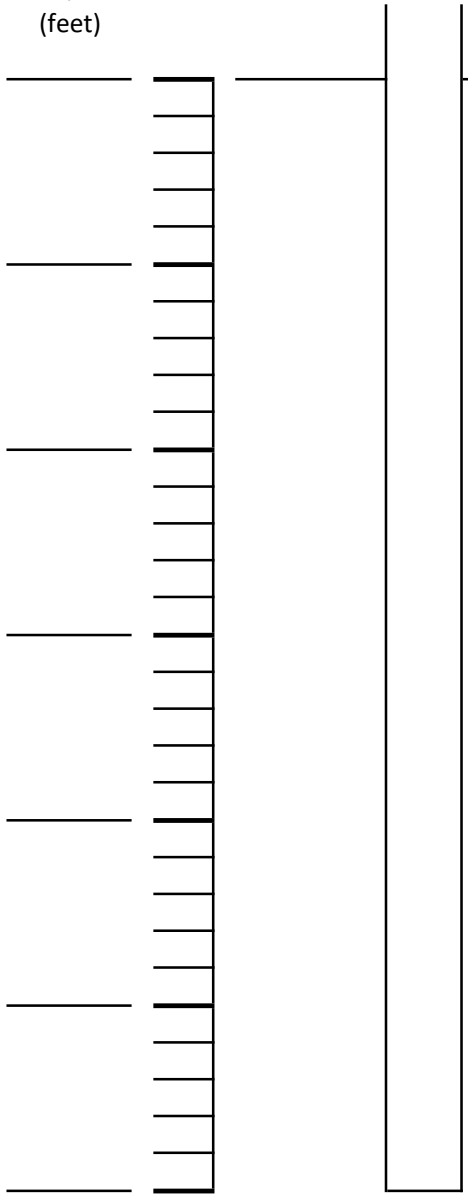
CASING PERFORATING

Equipment used	
Number of perforations/foot	
Size of perforations	
Interval perforated	

GROUTING

Interval grouted (FBLS)	
# of batches prepared	
For each batch record:	
Quantity of water used (gal.)	
Quantity of cement used (lbs.)	
Cement type	
Quantity of bentonite used (lbs.)	
Quantity of calcium chloride used (lbs.)	
Volume of grout prepared (gal.)	
Volume of grout used (gal.)	

WELL SCHEMATIC*



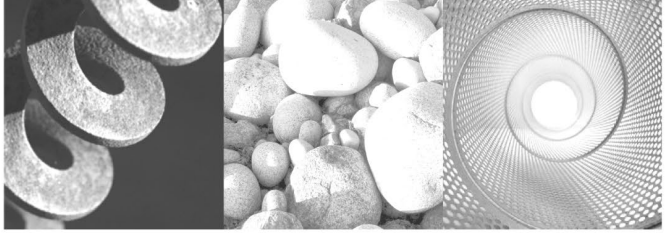
COMMENTS:

*Sketch in all relevant decommissioning data, including: interval overdrilled, interval grouted, casing left in hole, well stickup, etc.

Drilling Contractor

Department Representative

Appendix C Quality Assurance Project Plan



GEI Consultants

Appendix C. Quality Assurance Project Plan Remedial Investigation/Feasibility Study Work Plan - Lower Scajaquada Creek Operable Unit

Buffalo, New York

Submitted to:
National Fuel Gas Distribution Corporation
Williamsville, New York

Submitted by:
GEI Consultants, Inc.
100 Sylvan Parkway, Suite 400
Amherst, New York 14228
716.204.7154

June 2025
Project 2406810
This QAPP is applicable to the Remedial Investigation and Feasibility Study, which is projected to take place between 2025 and 2027.
Revision 1

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Appendix B Lab SOPs and Project Limits
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Appendix D GLNPO Requirements for Submission of Field Data

List of Acronyms

%D	percent difference
%R	percent recovery
%	percent
A	analytical
A.A.	Associate in Arts
ACOE	U.S. Army Corps of Engineers
AED	Automatic External Defibrillator
amsl	above mean sea level
ASTM	American Society for Testing and Materials
AVS	acid-volatile sulfide
AWQS	Ambient water quality standards
AWRBCL	Area-wide red-brown clay layer
BCL	below clay layer
BCP	Brownfield Cleanup Program
BFB	4-bromofluorobenzene
bgs	below ground surface
B.S.	Bachelor of Science
BTEX	benzene, toluene, ethylbenzene, and xylenes
BTU	calorific value
BUI	beneficial use impairments
CA	corrective action
CAS	Chemical Abstracts Service
CC	correlation coefficient
CCB	continuing calibration blank
CCV	continuing calibration verification
CCVC	tune-ending check standard
CFR	Code of Federal Regulations
CLP	Contract Laboratory Program
cm	centimeter
CMS	chip measuring system
COC	contaminants of concern
CPR	cardiopulmonary resuscitation
CSM	conceptual site model
CSO	combined sewer overflow
CVAA	cold vapor atomic absorption
CVOC	chlorinated volatile organic compound
DAP	diffuse anthropogenic pollution
DDT	1,1,1-trichloro-2,2-bis(4-chlorophenyl) ethane
DFTPP	decafluorotriphenylphosphine
DNAPL	dense non-aqueous phase liquid

DO	dissolved oxygen
DQI	data quality indicator
DQO	data quality objective
DRO	diesel range organics
EB	equipment blank
ECD	electron capture detector
EDD	electronic data deliverable
EIS	extracted internal standard
EPA	Environmental Protection Agency
FGDC	Federal Geographic Data Committee
FS	feasibility study
FSP	field sampling plan
ft	foot or feet
ft/ft	Feet per foot
GC	gas chromatography
GC/MS	gas chromatography/mass spectrometry
GEI	GEI Consultants, Inc
GIS	Geographic Information System
GLLA	Great Lakes Legacy Act
GLNPO	Great Lakes National Program Office
GPS	global positioning system
GRO	gasoline range organics
H&S	health and safety
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
IA	Indoor Air
IC	ion chromatograph
ICAL	initial calibration
ICB	initial calibration blank
ICP	inductively coupled plasma
ICV	initial calibration verification
ID	identification
IRM	interim remedial measure
IS	internal standard
L	liter
LCR	linear calibration range
LCS	laboratory control spike
LCSD	laboratory control spike duplicate
LD	laboratory duplicate
LDR	linear dynamic range
LEL	low effect level
LLC	limited Liability Company
LNAPL	light non-aqueous phase liquid

LOD	limit of detection
LOQ	limit of quantitation
MB	method blank
MDL	method detection limit
mg/kg	milligram per kilogram
mg/L	milligram per liter
MGP	manufactured gas plant
MPC	measurement performance criteria
MS	matrix spike
M.S.	Master of Science
MSD	matrix spike duplicate
MTBE	methyl-tert-butyl ether
MW	monitoring well
N/A	not applicable
NAPL	non-aqueous phase liquid
National Fuel	National Fuel Gas Distribution Corporation
ND	non-detect
NE	not established
NELAP	National Environmental Laboratory Accreditation Program
ng/L	nanograms per liter
NIST	National Institute of Standards and Technology
NIS	non-extracted internal standard
NY	New York
NYSDEC	New York State Department of Environmental Conservation
OSHA	Occupational Safety and Health Administration
OU	operable unit
PAH	polycyclic aromatic hydrocarbons
PAL	project action limit
PCB	polychlorinated biphenyls
PDF	portable document format
PE	Professional Engineer
PFAS	per- and polyfluoroalkyl substances
PFTBA	Perfluorotributyl-amine
PG	Professional Geologist
ppb	parts per billion
ppm	part per million
PQL	project quantitation limit
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
QL	quantitation limit
RAO	remedial action objective
RCRA	Resource Conservation and Recovery Act

RI	remedial investigation
RL	reporting limit
RPD	relative percent difference
RRT	relative retention times
RSD	relative standard deviation
RSL	regional screening level
S	sampling
SB	soil boring
SED	sediment
SEM	simultaneously extracted metals
SIM	selective ion monitoring
SOP	standard operating procedure
SSM	site safety manager
SVOC	semi-volatile organic compound
TBD	to be determined
TCL	target compound list
TCLP	Toxic Characteristic Leaching Procedure
TOC	total organic carbon
TPH	total petroleum hydrocarbons
TPH-DRO	total petroleum hydrocarbons – diesel range organics
TPH-GRO	total petroleum hydrocarbons – gasoline range organics
TSCA	Toxic Substance Control Act
µg/kg	micrograms per kilogram
µg/L	micrograms per liter
µmol/g	micromole per gram
VOA	volatile organic analysis
VOC	volatile organic compound

1. Introduction

This Quality Assurance Project Plan (QAPP) is being submitted on behalf of National Fuel Gas Distribution Corporation (National Fuel) in support of the remedial investigation (RI) to be performed in the Lower Scajaquada Creek area in Buffalo, New York. The work is being performed under the Great Lakes Legacy Act (GLLA) per a Project Agreement between the United States Environmental Protection Agency (USEPA), New York State Department of Environmental Conservation (NYSDEC), and National Fuel dated January 6, 2025. Under this agreement:

1. The USEPA will conduct a Feasibility Study (FS) and potentially initiate Remedial Design (RD) with the NYSDEC for the Black Rock Canal Operable Unit (OU).

2. National Fuel will conduct a Remedial Investigation (RI) and FS (RI/FS) for the Lower Scajaquada Creek OU focused primarily on historical releases from the former manufactured gas plant (MGP) located upstream of the West Avenue bridge crossing.

3. NYSDEC will provide oversight of the work in the Scajaquada Creek OU and complete source investigations of non-coal tar-related contaminant sources in adjacent upland properties, some of which may be commingled with coal tar-related sources.

The QAPP includes information regarding analytical methods, laboratories, and procedures that will be used during RI activities. GEI Consultants, Inc. (GEI) will be the primary environmental consultant for the Lower Scajaquada Creek OU. Pace Analytical Laboratories has been retained to provide environmental chemical analysis, GeoTesting Express has been retained to provide geotechnical analytical data.

This QAPP is Appendix C of the Remedial Investigation/Feasibility Study Work Plan – Lower Scajaquada Creek Operable Unit (April 2025) (RI/FS Work Plan) and is designed to be used in conjunction with the RI/FS Work Plan and other supporting documents, including the Field Sampling and Analytical Plan (April 2025, Appendix B of the RI/FS Work Plan). The RI/FS Work Plan contains site background information and investigation work scope details. Several of the QAPP Worksheets reference figures presented in the RI/FS Work Plan. Because the QAPP is an Appendix to the RI/FS Work Plan, the figures are not reproduced in the QAPP. Where appropriate, references are made to the figures included as part of the RI/FS Work Plan.

1.1 Development of the Quality Assurance Project Plan

As described on USEPA's Quality System Website (https://www.epa.gov/sites/default/files/2019-03/documents/g5-final.pdf), the objective of a QAPP is to document the planning, implementation, and assessment procedures for a particular project, as well as specific quality assurance and quality control activities. It integrates the technical and quality aspects of the project to provide a "blueprint" for obtaining the type and quality of environmental data and information needed for a specific decision or use.

In March 2005, the Intergovernmental Data Quality Task Force developed a policy and guidance document to fulfill the project-specific requirements of Part B of the American National Standards Institute/American Society of Quality Control E4 (ANSI/ASQC E4), to assist Federal departments and agencies with producing consistent QAPPs that reflect a systematic planning approach to collection and use of environmental data. That guidance document – the Uniform Federal Policy for Quality Assurance Project Plans (UFP-QAPP) Manual V1, March 2005 (USEPA, 2005a), and Optimized UFP-QAPP Worksheets, March 2012 (USEPA, 2012) – was followed in the development of this QAPP. Additionally, the provisions of this QAPP were reviewed against the minimum quality assurance/quality control (QA/QC) activities identified in the guidance document Uniform Federal Policy for Quality Assurance Project Plans: Part 2B, Quality Assurance/Quality Control Compendium: Minimum QA/QC Activities V1, March 2005 (USEPA, 2005b).

Investigation and characterization activities to be performed as part of the work at the Scajaquada Creek OU require development and submission of this QAPP to describe sampling, analyses, and data handling requirements for samples collected during work as part of OU-specific efforts.

1.2 Applicability of the QAPP

This QAPP is applicable to sampling and analysis work to be carried out at the Lower Scajaquada Creek OU and will be updated as needed to support modifications and additions to sampling activities. The period of applicability for this QAPP is the duration of the RI/FS. Any revisions made to this QAPP will be distributed as described in Worksheets #3 and #5, and all prior versions will be maintained by the GEI Quality Assurance Manager.

2. Document Overview

The Part 1 UFP-QAPP guidance and Optimized Worksheets (USEPA 2005a, 2012) include a series of 37 worksheets that are used to present the critical information required in a QAPP. The worksheets "ensure consistent content and presentation of information in a project-specific QAPP and streamline the review of QAPPs by regulators and others" (USEPA, 2005a). This QAPP was developed using the worksheet-based methodology; the worksheets follow this brief introductory text. Collectively, the QAPP worksheets provide information associated with four key topics:

- Project management and objectives
- Measurement and data acquisition
- Assessment and oversight
- Data review

A series of appendices is also included with this QAPP. These attachments include the following:

- Example chain of custody forms
- Laboratory verifications
- Laboratory quality assurance management plans and SOPs (included electronically only)
- Field SOPs and project operating procedures

When elements required by Part 1 UFP-QAPP are presented in other documents (e.g., SOPs), cross-referencing of these other documents can be used in lieu of repeating information (USEPA 2005a). Following this guidance, this QAPP provides certain relevant information by reference. This QAPP was prepared with information specific to the current scope of sampling and analysis activities associated with the RI/FS Work Plan to be conducted at the Scajaquada Creek OU by GEI and laboratories referenced herein.

Worksheets 1 through 37 were prepared to present universal information specific to investigation activities. Select worksheets that present similar information were combined.

Other pertinent site-specific information is presented in conjunction with other site-specific documents, which include but are not limited to the Lower Scajaquada Creek RI/FS Work Plan (April 2025). A list of such documents is provided in the References Section (Section 3).

QAPP Worksheets #1, 2: Title and Approval Page

Site Name/Project Name: Lower Scajaquada Creek Operable Unit Great Lakes Legacy Act (GLLA) Remedial Investigation and Feasibility Study (RI/FS Work Plan) – Appendix C

Site Location: The Lower Scajaquada Creek Operable Unit (OU) is comprised of the lands adjacent to Scajaquada Creek, extending south from the West Avenue Bridge to the creek's mouth where is discharges into the Black Rock Canal. The Scajaquada Creek Slip Area (Inlet Area), which was historically the creek's discharge location, is also part of the OU. Upland areas include properties owned by the former Penn Central Railroad, New York State, and City of Buffalo that are used for transportation and infrastructure corridors including a railroad passenger and freight train corridor, the NYS Thruway I-190, The Route 198 Scajaquada Expressway, and City of Buffalo roadways (West Avenue, Tonawanda Street, and Niagara Street).

Site Number/Code: Great Lakes Legacy Act (GLLA) NYSDEC Contract No. C011665

Regulatory Agency: United States Environmental Protection Agency (USEPA) Great Lakes National Program Office (GLNPO) and New York State Department of Environmental Conservation (NYSDEC)

Partnership Organization (USEPA)
GLNPO
c/o USEPA Region 5
77 West Jackson Boulevard
Chicago, IL 60604-3507
Lead Agency's Project Manager: Susan Virgilio
(virgilio.susan@epa.gov)

SUSAN VIRGILIO Digitally signed by SUSAN VIRGILIO
Date: 2025.06.10 15:16:37 -05'00'

Signature Date

QAPP Reviewer
GLNPO
c/o USEPA Region 5
77 West Jackson Boulevard
Chicago, IL 60604-3507
QAPP Reviewer: Christian Bako
(Bako.Christian@epa.gov)

CHRISTIAN BAKO 2025.06.10 16:23:12 -05'00'

Signature Date

Preparer
GEI Consultants, Inc.
100 Sylvan Parkway, Suite 400
Amherst, New York 14228
Preparer's Project Manager: Richard Frappa, P.G.
(rfrappa@geiconsultants.com)

Richard Frappa 6-26-2025

Signature Date

Quality Assurance Manager
GEI Consultants, Inc.
2000 CentreGreen Way, Suite 130
Cary, North Carolina 27513
Quality Assurance Manager: Jennifer Bash
(jbash@geiconsultants.com)

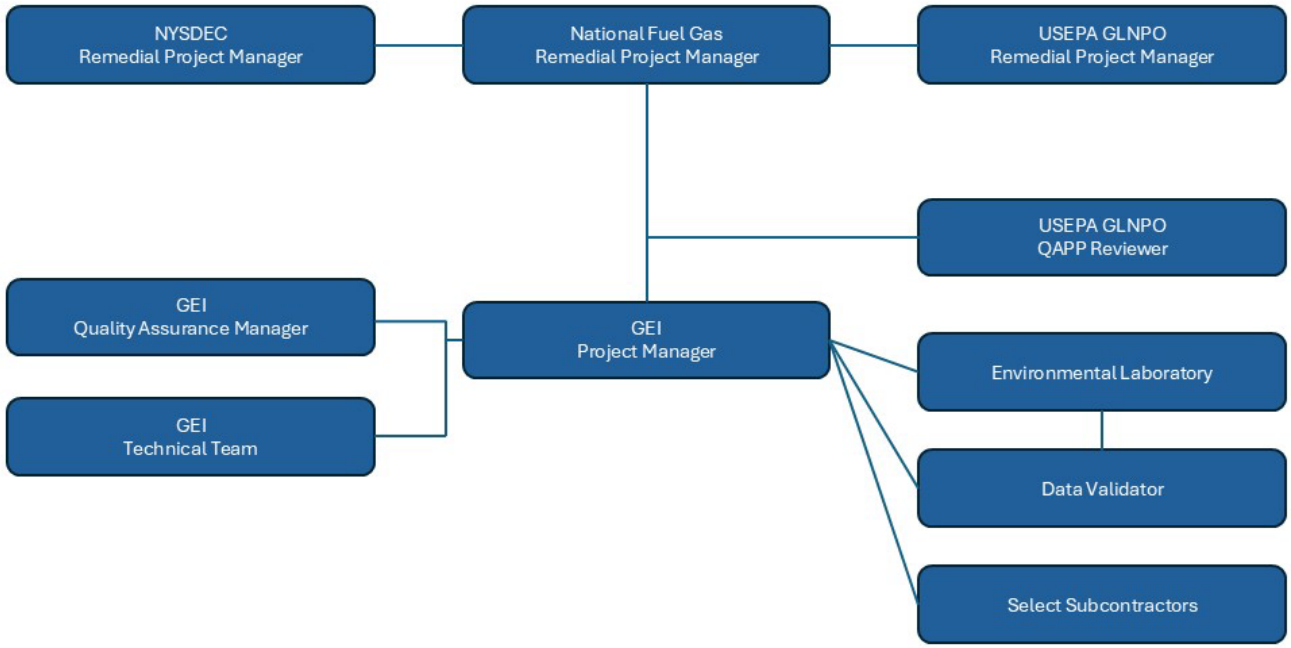
07-02-2025

Signature Date

Relevant Plans and Reports from Previous Investigations:

Site Characterization Report (GEI, October 2022)
Pre-Design Investigation and Interim Remedial Measures Report – Lower Scajaquada Creek City of Buffalo Pumphouse NYSDEC Spill #1406307 (GEI, March 2023)

QAPP Worksheets #3, 5: Distribution List and Project Organization Chart



QAPP recipients who are required to be notified of changes are indicated with an *.

The following stakeholders will be issued a copy of this QAPP according to the table below:

Stakeholder	Number of Copies	Address
USEPA-GLNPO*	Electronic	Virgilio.Susan@epa.gov
NYSDEC*	Electronic	mark.filipski@dec.ny.gov matthew.king@dec.ny.gov
National Fuel*	Electronic	alexandert@natfuel.com hoelscherk@natfuel.com
GEI Data Group*	Electronic	jwargo@geiconsultants.com
GEI Data Validators	Electronic	brussell@geiconsultants.com dmalone@geiconsultants.com
Pace Analytical*	Electronic	Melissa.Deyo@pacelabs.com
Alpha Analytical*	Electronic	Melissa.Deyo@pacelabs.com
Geo Express Testing*	Electronic	jcampbell@geotesting.com

QAPP Worksheets #4 & 7: Project Personnel Qualifications and Sign-Off Sheet

Copies of this form are to be signed by key project personnel from each organization to indicate they have read the applicable QAPP sections and will perform the tasks as described. Each organization will forward signed sheets to the central project file.

Organization: USEPA-GLNPO:

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature
Sue Virgilio	Remedial Project Manager	--	--	SUSAN VIRGILIO Digitally signed by SUSAN VIRGILIO Date: 2025.06.10 22:53:44 -05'00'
Christian Bako	QAPP Reviewer	--	--	CHRISTIAN BAKO 2025.06.26 12:25:53 -05'00'

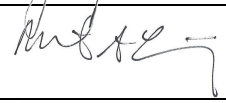
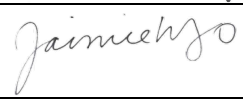
Organization: NYSDEC:

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature
Mark Filipski	Remedial Project Manager	--	--	Mark Filipski Digitally signed by Mark Filipski DN: cn=Mark Filipski, o=New York State Dept. of Environmental Conservation, ou=Great Lakes Basin Programs Unit, email=mark.filipski@dec.ny.gov, c=US Date: 2025.06.30 14:53:42 -04'00'
Matt King	RI Technical Lead	--	--	

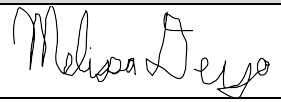
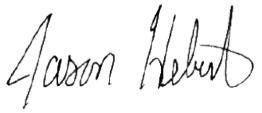
Organization: National Fuel

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature
Tanya Alexander	General Manager, Environmental Services; Project Manager	B.E Chemical Engineering 1981	CHMM, OSHA 40-Hr HAZWOPER and Refreshers; OSHA 8-Hr Supervisory Training, First Aid/CPR, IATA Training, 8-hr. RCRA Hazardous Waste Management – Generator Standards, DOT Training	Tanya B Alexander
Katie Hoelscher	Environmental Analyst II	Environmental Science 2003	CHMM, OSHA 40-Hr HAZWOPER and Refreshers; OSHA 8-Hr Supervisory Training, First Aid/CPR, IATA Training, 8-hr. RCRA Hazardous Waste Management – Generator Standards, DOT Training	Katie Hoelscher

Organization: GEI Consultants, Inc.

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature
Richard Frappa, P.G.	Project Manager	M.A. Geology; 30+ years' experience	Professional Geologist, NY; OSHA 40-Hr HAZWOPER and Refreshers; OSHA 8-Hr Supervisory Training	
Jennifer Bash	Quality Assurance Manager	B.S. Geology; 5 years' experience	OSHA 40-hour HAZWOPER and Refreshers; E-Railsafe	
Mike Cummings, P.G.	Field Team Lead	M.A. Geology; 20+ years' experience	Professional Geologist, NY; 40-Hr HAZWOPER and Refreshers	
Jaimie Wargo	Data Manager	A.A. General Studies; 20+ years' experience	N/A	
Steve Hawkins, CSP	Safety Director	B.S. Environmental Engineering; 20+ years' experience	Certified Safety Professional, OSHA Authorized Construction Trainer; 40-Hr HAZWOPER and Refreshers; OSHA Site Supervisor; OSHA Safety & Health Management; American Society of Safety Engineers Professional Member	

Organization: Alpha/Pace Analytical

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature
Melissa Deyo	Lab Project Manager	B.S. Geology; 20 years' experience	N/A	
Jason Hebert	Lab Quality Manager	B.S. Marine Safety and Environmental Protection; 20 years' experience	N/A	

Organization: GeoTesting Express

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature
Jonathan Campbell	Lab Project Manager	B.S. Earth Science; 12 years' experience	N/A	Mike Pratt signing for Jonathan Campbell
Michael Pratt	Lab Quality Manager	B.S. Environmental Science; MSc Environmental Engineering; 5 years' experience	N/A	

PROJECT FIELD PERSONNEL SIGN-OFF SHEET

I have read and understand the applicable information set forth in this QAPP for the tasks I am to perform. I agree to perform my work in accordance with this QAPP and applicable field standard operating procedures (SOPs). I also acknowledge that I have the necessary certifications and/or training required to perform the project tasks.

NAME (PRINTED)	SIGNATURE	DATE

QAPP Worksheet #6: Communication Pathways

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Regulatory agency interface	National Fuel	Tanya Alexander National Fuel Project Manager	alexandert@natfuel.com	Communicate as needed with regulatory agencies via phone or email with documentation saved to the project file.
Field progress reports	GEI	Mike Cummings GEI Field Team Leader/Site Safety Manager	mcummings@geiconsultants.com	Communicate daily or as needed with GEI Project Manager, via email or phone.
Field health and safety meetings and updates (i.e., tailgate meetings)	GEI	Mike Cummings GEI Field Team Leader/Site Safety Manager	mcummings@geiconsultants.com	Communicate daily, or as needed, with field personnel and subcontractors directly, or via e-mail or phone.
Significant health and safety or other project-related work issues, including stop work due to safety issues	GEI	Mike Cummings GEI Field Team Leader/Site Safety Manager	mcummings@geiconsultants.com	Each member of the field team has the authority to stop work if an unsafe condition is perceived. Health and safety incidents will be reported according to GEI's current procedure. The GEI Field Team Leader will be notified immediately by phone or directly and will be responsible for communicating with the GEI Project Manager, who will notify other entities (National Fuel Project Coordinator, GEI management, GEI Health and Safety Regional Manager), depending on the type/ magnitude of the Incident.
QAPP changes prior to fieldwork	GEI	Richard Frappa GEI Project Manager Jennifer Bash GEI QA Manager	rfrappa@geiconsultants.com jbash@geiconsultants.com	Notify the National Fuel Project Coordinator USEPA-GLNPO, and NYSDEC in writing or via email prior to implementation.

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Field activity execution, including variances from this QAPP and field corrective actions (Cas)	GEI	Mike Cummings GEI Field Team Leader/Site Safety Manager	mcummings@geiconsultants.com	Communicate daily or as needed, with GEI field personnel, subcontractors, and GEI Project Manager directly, or via email or phone. Significant variances and/or proposed revisions will be documented, approved by the GEI Project Manager and GEI QA Manager, and submitted for approval to NYSDEC and USEPA GLNPO.
Sample receipt variances	Laboratory	Melissa Deyo/ Pace Analytical Laboratory Project Manager Jonathan Campbell/GeoTesting Express Laboratory Project Manager	Melissa.deyo@pacelabs.com jcampbell@geotesting.com	Communicate significant issues to the GEI Field Team Leader via phone or email, within 24 hours of sample receipt.
Laboratory QC variances and analytical Cas	Laboratory	Laboratory staff (technicians, analysts, group leaders, Laboratory QA Manager)		Communicate/resolve per the Laboratory QA Manual. Communicate significant issues to the GEI Project Manager via phone or email, as variances are identified; narrate in the laboratory report. Minor variances require narration only.
Electronic data deliverable (EDD) issues, e.g., omissions, discrepancies, variances from standard	GEI	Jaimie Wargo GEI Data Manager	jwargo@geiconsultants.com	Communicate/resolve by phone or email with laboratory Project Manager.
Data verification issues, e.g., incomplete records	GEI	Mike Cummings GEI Field Team Leader Jaimie Wargo GEI Data Manager	mcummings@geiconsultants.com jwargo@geiconsultants.com	Communicate/resolve by phone or email with responsible party, i.e., field staff or laboratory Project Manager.

Communication Driver	Organization	Name	Contact Information	Procedure (timing, pathway, documentation, etc.)
Data validation issues, e.g., noncompliance with procedures	GEI	Jaimie Wargo	jwargo@geiconsultants.com	Communicate/resolve with Laboratory Project Manager by phone. Issues potentially impacting achievement of project objectives will be communicated to the GEI Project Manager, who will notify National Fuel's Project Coordinator.
Subcontractor issues, e.g., scope, schedule, budget	GEI	Richard Frappa GEI Project Manager	rfrappa@geiconsultants.com	Communicate/resolve by phone or email on an as-needed basis.

Notes:
TBD = To be determined; CA = corrective action; QA = quality assurance; QC = quality control; EDD = electronic data deliverable; GEI = GEI Consultants, Inc.

Worksheet #8: Special Personnel Training Requirements Table

Project Function	Specialized Training – Title or Description of Course	Training Providers	Training Date	Personnel/Groups Receiving Training	Personnel Titles/Organizational Affiliation	Location of Training Records/Certifications[1]
HAZWOPER	40-hour initial training as per 29 CFR 1910.120	Outsourced to certified trainers	Various	Field personnel, Project Manager, and other key staff	Various GEI staff and drilling subcontractor field staff	GEI's SharePoint site
HAZWOPER	8-hour annual refresher as per 29 CFR 1910.120	Certified in-house and out-sourced providers	Various	Field personnel, Project Manager, and other key staff	Various GEI staff and drilling subcontractor field staff	GEI's SharePoint site
HAZWOPER	CPR and first aid training as per 29 CFR 1910.120	Outsourced to certified trainers	Various	Site-Safety Officer on site during field work	Various GEI field staff	GEI's SharePoint site

[1] If training records and/or certificates are on file elsewhere, document their location in this column. If training records and/or certificates do not exist or are not available, then this should be noted.

Worksheet #9: Project Scoping Session Summaries

Project/Site Name: Lower Scajaquada Creek Operable Unit
Site Location: Buffalo, New York
Projected Date(s) of Sampling: May 2025
Program Manager: Rick Frappa

Date of Session: June 1, 2023
Scoping Session Purpose: Lower Scajaquada Creek OU RI/FS Meeting with Niagara River AOC Sediment Group

Name	Title	Affiliation	Phone No.	E-mail Address	Project Role
Rick Frappa	GEI Project Manager	GEI	(716) 204-7156	rfrappa@geiconsultants.com	Project Manager
Jeffrey Holden	GEI Senior Engineer	GEI	(607) 216-8956	jholden@geiconsultants.com	Senior Engineer
Mike Cummings	GEI Field Team Lead	GEI	(716) 204-7155	mcummings@geiconsultants.com	Field Team Lead
Katie Hoelscher	Environmental Analyst	National Fuel	(716) 857-7236	HoelscherK@natfuel.com	Project Manager
Stan Radon	Regional Geologist	NYSDEC	(716) 851-7220	stanley.radon@dec.ny.gov	NYSDEC Regional Lead
Matt King	Assistant Geologist	NYSDEC	(716) 851-7258	matthew.king@dec.ny.gov	RI Technical Lead
Josh Vaccaro	Assistant Engineer	NYSDEC	(716) 851-7220	Joshua.vacarro@dec.ny.gov	Field Oversight
Steve Moeller	Professional Geologist	NYSDEC	(716) 851-7220	Steven.moeller@dec.ny.gov	Field Oversight
Sue Virgilio	USEPA-GLNPO Remedial Project Manager	USEPA-GLNPO	(312) 886-4244	Virgilio.Susan@epa.gov	Remedial Project Manager
Andrew Lenox	US ACOE Technical Lead	ACOE-Buffalo District	Not Documented	Andrew.M.Lenox@usace.army.mil	Sediment Remediation Technical Lead

Consensus decisions made:

Action	Responsible Party	Due Date
Establishment of Recommended Cleanup Goals	NYSDEC	TBD
Fish Collection and Analysis Data	NYSDEC	TBD
Refining RI/FS Cost Estimate	National Fuel	July 2023

Project/Site Name: Lower Scajaquada Creek Operable Unit **Site Location: Buffalo, New York** **Projected Date(s) of Sampling: May 2025** **Program Manager: Rick Frappa**					
Date of Session: September 12, 2024 **Scoping Session Purpose: Black Rock/Lower Scajaquada Project Coordinating Team**					
Name	**Title**	**Affiliation**	**Phone No.**	**E-mail Address**	**Project Role**
Rick Frappa	GEI Project Manager	GEI	(716) 204-7156	rfrappa@geiconsultants.com	Project Manager
Jeffrey Holden	GEI Senior Engineer	GEI	(607) 216-8956	jholden@geiconsultants.com	Senior Engineer
Cam Davis	GEI Vice President	GEI	(312) 985-0368	cdavis@geiconsultants.com	Community Relations Coordinator
Sue Virgilio	USEPA-GLNPO Remedial Project Manager	USEPA-GLNPO	(312) 886-4244	Virgilio.Susan@epa.gov	Remedial Project Manager
Matt King	Assistant Geologist	NYSDEC	(716) 851-7258	matthew.king@dec.ny.gov	RI Technical Lead
Stan Radon	Regional Geologist	NYSDEC	(716) 851-7220	stanley.radon@dec.ny.gov	NYSDEC Regional Lead
Tanya Alexander	National Fuel Project Manager	National Fuel	716-857-7410	AlexanderT@natfuel.com	National Fuel Project Manager
Katie Hoelscher	Environmental Analyst	National Fuel	(716)-857-7236	HoelscherK@natfuel.com	Project Manager

Consensus decisions made:

Action	Responsible Party	Due Date
EPA to provide work scope typically assigned to its contractors for similar investigations.	USEPA	Included in Project Agreement executed January 6, 2025

EPA to invite GEI to September 30th Great Lakes Sediment Work Group call.	USEPA	Meeting on September 30, 2024
DEC to review planning document and provide any comments (e.g., additional objectives, scope, or work plan content including ecological evaluation, including QAPP data requirements for soil and groundwater data to be collected).	NYSDEC	RI/FS Framework – Agreed upon in May 2022 Scope of work in Project Agreement executed January 6, 2025
Scheduling scoping sessions to address the landside and creek side separately.	GEI	Meeting in early October 2024. Field meeting with NYSDEC in late December 2024
EPA to provide QAPP requirements, electronic data formats and EPA contractors who could do sediment data validation.	GEI/USEPA	Conference call with USEPA GLNPO, EPA QA lead, and EPA contractors on November 6, 2024

Project/Site Name: Lower Scajaquada Creek Operable Unit **Site Location: Buffalo, New York** **Projected Date(s) of Sampling: May 2025** **Program Manager: Rick Frappa**					
Date of Session: October 15, 2024 **Scoping Session Purpose: Black Rock/Lower Scajaquada Project Coordinating Team**					
Name	**Title**	**Affiliation**	**Phone No.**	**E-mail Address**	**Project Role**
Rick Frappa	GEI Project Manager	GEI	(716) 204-7156	rfrappa@geiconsultants.com	Project Manager
Jeffrey Holden	GEI Senior Engineer	GEI	(607) 216-8956	jholden@geiconsultants.com	Senior Engineer
Cam Davis	GEI Vice President	GEI	(312) 985-0368	cdavis@geiconsultants.com	Community Relations Coordinator
Tanya Alexander	National Fuel Project Manager	National Fuel	716-857-7410	AlexanderT@natfuel.com	National Fuel Project Manager
Katie Hoelscher	Environmental Analyst	National Fuel	(716) 857-7236	HoelscherK@natfuel.com	Project Manager
Sue Virgilio	USEPA-GLNPO Remedial Project Manager	USEPA-GLNPO	(312) 886-4244	Virgilio.Susan@epa.gov	Remedial Project Manager
Matt King	Assistant Geologist	NYSDEC	(716) 851-7258	matthew.king@dec.ny.gov	RI Technical Lead
Stan Radon	Regional Geologist	NYSDEC	(716) 851-7220	stanley.radon@dec.ny.gov	NYSDEC Regional Lead
Mark Filipski	NYSDEC Remedial Project Manager	NYSDEC	Not Documented	mark.filipski@dec.ny.gov	NYSDEC Remedial Project Manager
Andrew Lenox	US ACOE Technical Lead	ACOE-Buffalo District	Not Documented	Andrew.M.Lenox@usace.army.mil	Sediment Remediation Technical Lead

Consensus decisions made:

Action	Responsible Party	Due Date
GEI to contact USEPA for scheduling discussion for data validation options.	GEI	November 6, 2024
GEI to send invitations for next PCT meeting.	GEI	November 2024
DEC to provide any comments on GEI's proposed scope of Landside and Sediment Investigations.	GEI	December 13, 2024

Project/Site Name: Lower Scajaquada Creek Operable Unit
Site Location: Buffalo, New York
Projected Date(s) of Sampling: May 2025
Program Manager: Rick Frappa

Date of Session: November 6, 2024
Scoping Session Purpose: Black Rock/Lower Scajaquada Project QAPP & Data Validation/Management/EDD

Name	Title	Affiliation	Phone No.	E-mail Address	Project Role
Rick Frappa	GEI Project Manager	GEI	(716) 204-7156	rfrappa@geiconsultants.com	Project Manager
Jeffrey Holden	GEI Senior Engineer	GEI	(607) 216-8956	jholden@geiconsultants.com	Senior Engineer
Jaimie Wargo	Data Group Manager	GEI	(860) 790-0905	jwargo@geiconsultants.com	Data Manager
Jennifer Bash	GEI Staff Professional	GEI	(706) 945-4889	jbash@geiconsultants.com	GEI QA Manager
Katie Hoelscher	Environmental Analyst	National Fuel	(716) 857-7236	HoelscherK@natfuel.com	Project Manager
Tanya Alexander	National Fuel Project Manager	National Fuel	716-857-7410	AlexanderT@natfuel.com	National Fuel Project Manager
Sue Virgilio	USEPA-GLNPO Remedial Project Manager	USEPA-GLNPO	(312) 886-4244	Virgilio.Susan@epa.gov	Remedial Project Manager
Christian Bako	USEPA GLNPO Project Manager - QA	USEPA-GLNPO	(312) 886-6787	bako.christian@epa.gov	Quality Assurance Program
Matt King	Assistant Geologist	NYSDEC	(716) 851-7258	matthew.king@dec.ny.gov	RI Technical Lead
Shellee McGrath	Data Validator	GDIT, EPA contractor	Not Documented	shellee.mcgrath@gdit.com	TBD
Nazy Perkins	Sr. Program Manager	GDIT, EPA contractor	(571 546-6564	nazy.perkins@gdit.com	TBD
Timothy Vonnahme	Data Validator	APTIM, EPA Contractor	Not Documented	timothy.vonnahme@aptim.com	TBD

Consensus decisions made:

Action	Responsible Party	Due Date
EPA's data validation contractor can validate samples collected from all matrices during a GLLA project, including soil and groundwater.	EPA	Estimated beginning August 2025
EPA provided GLLA Data Reporting Standard (August 2023) and GLLA QA Considerations Final (August 2023)	EPA	November 6, 2024
GEI to provide data in Staged Electronic Data Deliverable (SEDD.	GEI	Established in January 2025

Project/Site Name: Lower Scajaquada Creek Operable Unit
Site Location: Buffalo, New York
Projected Date(s) of Sampling: May 2025
Program Manager: Rick Frappa

Date of Session: November 13, 2024
Scoping Session Purpose: Black Rock/Lower Scajaquada Project Coordinating Team

Name	Title	Affiliation	Phone No.	E-mail Address	Project Role
Rick Frappa	GEI Project Manager	GEI	(716) 204-7156	rfrappa@geiconsultants.com	Project Manager
Jeffrey Holden	GEI Senior Engineer	GEI	(607) 216-8956	jholden@geiconsultants.com	Senior Engineer
Tanya Alexander	National Fuel Project Manager	National Fuel	716-857-7410	AlexanderT@natfuel.com	National Fuel Project Manager
Katie Hoelscher	Environmental Analyst	National Fuel	(716) 857-7236	HoelscherK@natfuel.com	Project Manager
Sue Virgilio	USEPA-GLNPO Remedial Project Manager	USEPA-GLNPO	(312) 886-4244	Virgilio.Susan@epa.gov	Remedial Project Manager
Matt King	Assistant Geologist	NYSDEC	(716) 851-7258	matthew.king@dec.ny.gov	RI Technical Lead
Stan Radon	Regional Geologist	NYSDEC	(716) 851-7220	stanley.radon@dec.ny.gov	NYSDEC Regional Lead
Mark Filipski	NYSDEC Remedial Project Manager	NYSDEC	Not Documented	mark.filipski@dec.ny.gov	NYSDEC Remedial Project Manager
Andrew Lenox	US ACOE Technical Lead	ACOE-Buffalo District	Not Documented	Andrew.M.Lenox@usace.army.mil	Sediment Remediation Technical Lead

Consensus decisions made:

Action	Responsible Party	Due Date
GEI will provide refined mapping of sediment and landside investigation locations, along with analyte lists, to the PCT group.	GEI	November 22, 2024
The PCT group will provide feedback to GEI.	USEPA, NYSDEC, ACOE	December 13, 2024
GEI will revise the time for the next PCT meeting on December 18, 2024, to 11:00 AM Eastern and set up recurring meeting times in 2025 for the fourth Wednesday of each month at 3:00 PM Eastern.	GEI	Established in January 2025

Project/Site Name: Lower Scajaquada Creek Operable Unit **Site Location: Buffalo, New York** **Projected Date(s) of Sampling: May 2025** **Program Manager: Rick Frappa**					
Date of Session: December 18, 2024 **Scoping Session Purpose: Black Rock/Lower Scajaquada Project Coordinating Team**					
Name	**Title**	**Affiliation**	**Phone No.**	**E-mail Address**	**Project Role**
Rick Frappa	GEI Project Manager	GEI	(716) 204-7156	rfrappa@geiconsultants.com	Project Manager
Jeffrey Holden	GEI Senior Engineer	GEI	(607) 216-8956	jholden@geiconsultants.com	Senior Engineer
Tanya Alexander	National Fuel Project Manager	National Fuel	716-857-7410	AlexanderT@natfuel.com	National Fuel Project Manager
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Matt King	Assistant Geologist	NYSDEC	(716) 851-7258	matthew.king@dec.ny.gov	RI Technical Lead
Mark Filipski	NYSDEC Remedial Project Manager	NYSDEC	Not Documented	mark.filipski@dec.ny.gov	NYSDEC Remedial Project Manager
Andrew Lenox	US ACOE Technical Lead	ACOE-Buffalo District	Not Documented	Andrew.M.Lenox@usace.army.mil	Sediment Remediation Technical Lead

Consensus decisions made:

Action	Responsible Party	Due Date
GEI will meet with NYSDEC at the Scajaquada Creek OU site to discuss additional boring/well locations.	GEI/NYSDEC	December 30, 2024
GEI will provide a status report on work plan progress at the next PCT meeting.	GEI	January 22, 2025

Project/Site Name: Lower Scajaquada Creek Operable Unit **Site Location: Buffalo, New York** **Projected Date(s) of Sampling: May 2025** **Program Manager: Rick Frappa**					
Date of Session: December 30, 2024 **Scoping Session Purpose: Lower Scajaquada Creek OU RI/FS Meeting in Field with NYSDEC to Field Locate Additional Boring/Well Locations**					
Name	**Title**	**Affiliation**	**Phone No.**	**E-mail Address**	**Project Role**
Rick Frappa	GEI Project Manager	GEI	(716) 204-7156	rfrappa@geiconsultants.com	Project Manager
Mike Cummings	GEI Field Team Lead	GEI	(716) 204-7155	mcummings@geiconsultants.com	Field Team Lead
Katie Hoelscher	Environmental Analyst	National Fuel	(716) 857-7236	HoelscherK@natfuel.com	Project Manager
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Josh Vaccaro	Assistant Engineer	NYSDEC	(716) 851-7220	Joshua.vacarro@dec.ny.gov	Field Oversight
Steve Moeller	Professional Geologist	NYSDEC	(716) 851-7220	Steven.moeller@dec.ny.gov	Field Oversight

Consensus decisions made:

Action	Responsible Party	Due Date
GEI and NYSDEC walked entire OU and agreed upon additional boring/well location on the landside of the site.	GEI/NYSDEC	December 30,2024

Site Location: Buffalo, New York **Projected Date(s) of Sampling: May 2025** **Program Manager: Rick Frappa**					
Date of Session: January 22, 2025 **Scoping Session Purpose: Black Rock/Lower Scajaquada Project Coordinating Team**					
Name	**Title**	**Affiliation**	**Phone No.**	**E-mail Address**	**Project Role**
Rick Frappa	GEI Project Manager	GEI	(716) 204-7156	rfrappa@geiconsultants.com	Project Manager
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Mark Filipski	NYSDEC Remedial Project Manager	NYSDEC	Not Documented	mark.filipski@dec.ny.gov	NYSDEC Remedial Project Manager
Andrew Lenox	US ACOE Technical Lead	ACOE-Buffalo District	Not Documented	Andrew.M.Lenox@usace.army.mil	Sediment Remediation Technical Lead

Consensus decisions made:

Action	Responsible Party	Due Date
GEI to provide Draft Work Plans and QAPP with incorporation of additional boring/well locations requested by NYSDEC	GEI	February 21, 2025
Schedule next PCT meeting	GEI	February 26, 2025

Project/Site Name: Lower Scajaquada Creek Operable Unit
Site Location: Buffalo, New York
Projected Date(s) of Sampling: May 2025
Program Manager: Rick Frappa

Date of Session: May 28, 2025
Scoping Session Purpose: Black Rock/Lower Scajaquada Project Coordinating Team

Name	Title	Affiliation	Phone No.	E-mail Address	Project Role
Rick Frappa	GEI Project Manager	GEI	(716) 204-7156	rfrappa@geiconsultants.com	Project Manager
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Matt King	Assistant Geologist	NYSDEC	(716) 851-7258	matthew.king@dec.ny.gov	RI Technical Lead
Mark Filipski	NYSDEC Remedial Project Manager	NYSDEC	Not Documented	mark.filipski@dec.ny.gov	NYSDEC Remedial Project Manager
Andrew Lenox	US ACOE Technical Lead	ACOE-Buffalo District	Not Documented	Andrew.M.Lenox@usace.army.mil	Sediment Remediation Technical Lead

Consensus decisions made:

Action	Responsible Party	Due Date
NYSDEC will provide notice of acceptance of National Fuel's response to comments. Acceptance letter was forthcoming at the time of the meeting and was received on May 30, 2025.	NYSDEC	N/A
The QAPP was revised to address comments received from EPA on May 7, 2025. At the time of the meeting, the QAPP was under EPA review. Acceptance of the QAPP was received on June 9, 2025 via email.	GEI/USEPA	N/A

Worksheet #10: Conceptual Site Model

A detailed description of the Lower Scajaquada Creek OU is presented in the RI/FS Work Plan, which the QAPP is appended to. Appendix A of the Work Plan includes the Site Characterization Report (GEI, 2022) detailing known environmental information and the basis for Remedial Investigation. A summary is provided below.

Preliminary Conceptual Site Model

The 2021/2022 Site Characterization of the Lower Scajaquada Creek area identified contamination in soils, groundwater, creek sediments, and City of Buffalo infrastructure. This contamination is linked to historic dense non-aqueous phase liquid (DNAPL) transport from former manufactured gas plant (MGP) operations. Additionally, investigations by other parties and the NYSDEC at Brownfield Cleanup Program (BCP) sites revealed contamination of soil, groundwater, and sediments due to past industrial property use, described by the NYSDEC as commingled contamination.

Information from these investigations was used to develop a preliminary conceptual site model (CSM) for the Lower Scajaquada Creek OU. The CSM outlines potential sources of contamination, release mechanisms, exposure media, and exposure routes based on current and potential future land use. The CSM will be refined based on data collected in the RI.

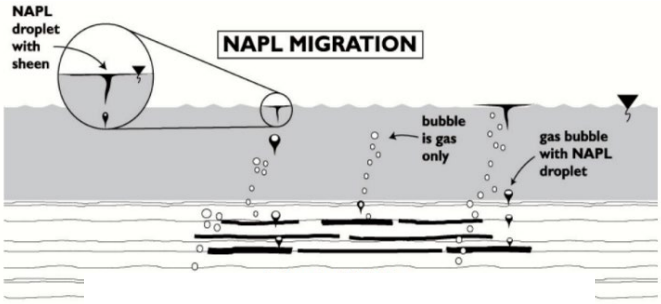
Previous investigations identified various chemical constituents in lower alluvial soil, groundwater, sediment, and City of Buffalo Pumphouse discharge in the Lower Scajaquada Creek OU. These environmental media could potentially serve as exposure media. The CSM provides a qualitative assessment of potential exposure via each medium for various types of receptors.

Data and findings from prior investigations indicate that sources of contamination affecting the Lower Scajaquada Creek originate from the former MGP operation, historic industrial use of nearby properties, and discharge to the creek from combined sewer outfall (CSOs) and the City of Buffalo Pumphouse. Contributions from upstream point and non-point sources are also likely given the highly urbanized status of the watershed.

Contamination release mechanisms refer to the processes by which contamination could be transferred from sources to environmental media. Contamination release mechanisms considered as part of the CSM are described below:

- Volatilization: volatile organic compounds (VOCs) from non-phase liquid (NAPL), contaminated soils, and/or groundwater that are released into air, including soil gas (i.e., the air entrained between soil particles in the vadose zone).

- DNAPL Migration: Gravity driven transport of DNAPL occurred as a result of product with specific gravity greater than 1.0 migrating vertically downward until encountering a low-permeability zone (i.e., clay) that precluded further vertical migration. When sufficient source existed, the DNAPL also spread laterally along the top of the clay confining layer, affecting a zone of coarser soil above the clay confining layer.
- Solubilization: Groundwater quality is affected through solubilization of contaminants from pooled or residual DNAPL. Once solubilized into groundwater, constituents are also transported with groundwater flow and may discharge into surface water.
- Discharges to the Creek: Surface water and groundwater collected in the City of Buffalo Pumphouse wet well was discharged to surface water as part of normal pumphouse operations (Pumphouse Interim Remedial Measure [IRMs] mitigated DNAPL entry into the system).
- Ebullition transport: NAPL migration from shallow DNAPL in creek sediments near the Pumphouse to the creek surface driven by gas bubbles caused by changes in hydraulic pressures (e.g., as created by Black Rock Canal lock operation). This contaminant release mechanism produces observed sheen on the surface water in the creek. Ebullition is conceptually shown graphically in the adjacent schematic.



Source: USEPA, 2015

Potential exposure describes the mechanism (exposure route) by which human receptors can be exposed to contamination during current or potentially foreseeable uses of the Lower Scajaquada Creek area. Such receptors include commercial workers, utility workers, construction workers, and passive recreational users of the creek and land adjacent to the waterway. Direct contact, ingestion, inhalation, and vapor intrusion/exposure are all exposure routes that could potentially impact receptors, depending on the nature of their specific activities and exposure areas.

Primary and secondary contact recreation and fishing exposure (i.e., consumption of fish caught from the creek) are pathways that could potentially impact human receptors. DNAPL-impacted soils are typically deeper than 15 feet below the ground surface, so human exposure to DNAPL in soil would be limited to construction workers involved in deep excavation work such as major utility infrastructure development or building construction with deep foundations. Under these scenarios, human exposure routes for chemicals include:

- inhalation of VOCs volatized from groundwater (particularly chlorinated volatile organic compounds [CVOCs] in the area of Niagara Street);
- dermal contact, inhalation, and incidental ingestion of soil/sediment particulates; and
- dermal contact and incidental ingestion of groundwater/surface water.

Groundwater in the area is not a source of drinking water. Scajaquada Creek surface water is also not a drinking water source.

Ecological exposure mechanisms in the Lower Scajaquada Creek are direct exposure to impacted sediments and pore water in the observed depositional areas and assimilation by a species (e.g., prey/food item) at one trophic level and transferred to a higher trophic level species through consumption. For certain types of chemicals, bioaccumulation can exacerbate this pathway at certain levels in the food chain. Based on the evaluation of exposure pathways, the key receptors of concern are benthic invertebrates, fish and semi-aquatic piscivorous mammals and avian species.
The key findings from the Site Characterization are summarized below and establish the preliminary CSM for the investigation area.

Soil Contamination

Soil Quality and DNAPL Presence: Pesticides, PCBs, herbicides, and cyanide were not detected in Site Characterization soil samples above NYSDEC's soil guidance values, regardless of the presence of DNAPL impacts. However, VOCs and semi-volatile organic compounds (SVOCs) (primarily polycyclic aromatic hydrocarbons [PAHs]) were found in soils affected by DNAPL. Certain metals, including arsenic, copper, lead, mercury, nickel, and zinc, were detected in some soil samples above soil guidance values. The presence of chemical impacts associated with DNAPL contamination did not correlate with elevated metal concentrations. Grossly contaminated soil and DNAPL were primarily found in deeper alluvial soils directly above the red-brown clay layer. DNAPL accumulation was observed in the sumps of several monitoring wells near Scajaquada Creek, with well screens monitoring the bottom of the alluvial soils above the clay layer.

The uniformity, thickness, and low permeability of the area-wide red-brown clay layer indicate that layer acts as a confining layer, preventing the downward vertical migration of DNAPL. DNAPL impacts extending into the red-brown clay layer were not identified during the Site Characterization. The extent to which the clay layer serves as a competent and contiguous vertical migration barrier will be further evaluated as part of the RI.

Source Analysis: Forensic analysis of DNAPL and grossly contaminated soil samples indicate a water gas tar source of pyrogenic origin. DNAPL impacts are confined to deeper alluvial soil due to the surface geometry of the red-brown clay and hydraulic gradients. DNAPL was not identified in shallow soil, suggesting lateral migration of DNAPL from an upgradient source on top of the red-brown clay layer. The DNAPL source is presumed to be historic migration from the Iroquois Gas/Westwood Pharmaceutical Terrestrial/Riparian Site, prior to the installation of engineering controls that contain and remove DNAPL from areas upstream of the West Avenue Bridge.

Chemical fingerprinting of the DNAPL found in borings and wells downstream from the Terrestrial/ Riparian Site engineering controls is similar to that recovered at the Iroquois Gas/Westwood Pharmaceutical Sites. DNAPL has not been detected in shallower soils or

sediments near the West Avenue Bridge, suggesting no ongoing releases are occurring. However, this will be further investigated as part of the RI. There is a possibility of CVOC DNAPL associated with historic industrial usage at BCP sites located at 31 Tonawanda St. and 1660 Niagara St. This is based on dissolved phase concentrations in groundwater of several chlorinated volatile organic compounds approaching 1% of their individual solubility limits.

Groundwater Contamination

MGP Constituent Detection: Site Characterization groundwater samples collected from the Lower Scajaquada Creek area contained benzene, toluene, ethylbenzene, and xylenes (BTEX) and PAH compounds of pyrogenic origin above New York State ambient water quality standards (AWQS). These constituents were detected in wells with wells screens intercepting the area-wide red-brown clay and where DNAPL was identified to accumulate in the bottom of the well.

Additional Contaminants: Petroleum-related (petrogenic) VOCs, CVOCs, 1,4-dioxane, and PFAS were also detected above AWQS in some wells located on and near BCP properties and near the Inlet. Where present with pyrogenic VOCs and PAHs, their presence is identified as comingled contamination. CVOCs and 1,4-dioxane were also present in the City of Buffalo Pumphouse discharge water at concentrations above AWQS. As described in Section 4 of the RI/FS Work Plan, an IRM was completed in 2022 to mitigate entry of DNAPL and comingled contamination into the manholes and conveyance piping.

Pesticides and herbicides were not detected in groundwater samples analyzed during the Site Characterization. Resource Conservation and Recovery Act (RCRA) metals were not detected above AWQS in most wells and none containing DNAPL.

Sediment Contamination

Shallow Sediments: DNAPL impacted recent fluvial deposits were observed in shallow sediments (0-6” depth) in the creek Inlet near the City of Buffalo Pumphouse discharge outlet. PAH concentrations in some shallow sediment samples were elevated, with the sample collected directly adjacent to the Pumphouse discharge outlet (SED-LSC-3; total PAH-16 concentration of 5,649 mg/kg). DNAPL was not identified in shallow sediments elsewhere in the creek during the Site Characterization.

Contaminant Levels: Shallow sediments exhibited elevated levels of volatile and extractable petroleum hydrocarbons, PCBs, and metals (copper, lead, nickel, zinc) when compared to sediment guidance values. Deeper sediments also showed significant contamination, particularly in the creek Inlet area, with high concentrations of BTEX, PAHs, polychlorinated biphenyls (PCBs), and metals (copper, lead nickel, mercury, zinc). DNAPL or sheens/ staining associated with suspected water gas tar residual was identified in deeper alluvial-derived soils below the recent fluvial deposits above the area-wide clay in five of nine sediment borings.

Data Gaps

Additional data are required to assess the nature and extent of impacts more fully in soil, groundwater, and sediments to evaluate remedial alternatives for the Lower Scajaquada Creek OU. Data gaps with RI objectives are summarized in Worksheet #11.

Worksheet #11: Data Quality Objectives/Systematic Planning Process Statements

Systematic Planning Process

Who will use the data?

National Fuel, GEI, and overseeing agencies will use the data to evaluate the extent of impacts and to identify and evaluate remedial alternatives through a feasibility study to support the design and completion of remedial actions for the Scajaquada Creek OU.

What will the data be used for?

The data will be used to 1) characterize and/or verify extent of vertical and horizontal contaminants; 2) develop remedial action objectives (RAOs); and 3) identify options for remedial actions that have the potential to fulfill the RAOs.

What types of data are required?

Data needs include the following:

- Contaminant concentration data from sediments, soils, and groundwater for purposes of defining and verifying extent of contamination to support the preparation of the feasibility study to evaluate remedial alternatives.
- Sediment and soil geophysical and geotechnical data to support geotechnical evaluations (e.g., stability of structures along the creek under certain remedial scenarios).
- Bathymetric, topographic, and hydrologic data to allow adequate characterization and design of remedial alternatives.
- Other physical and chemical data for purposes of compliance with disposal and reuse requirements.

How good does the data need to be?

Data needs to comply with the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) requirements, the quality assurance/quality control (QA/QC) requirements of this QAPP, and specific requirements of each associated work plan or field sampling and analytical plan (FSP). The data needs to be sufficient to make decisions based at the appropriate scale to achieve the goals for the areas and activities considered in this QAPP. See Worksheet #37 for information regarding the data usability assessment.

How much data is needed?

Sufficient data is needed to fill data gaps to assure that a comprehensive CSM can be developed to support the preparation of the feasibility study to evaluate remedial alternatives and for verification of remedy performance and for compliance with disposal or reuse, as needed. The number of borings, samples, and other data to be collected are described in Worksheets #17 and #18 and the RI/FS Work Plan (GEI, 2025). Actual numbers may vary based on field conditions, initial results, and whether additional sampling is necessary to achieve objectives.

Where, when, and how should the data be collected?

Sampling and analysis have been ongoing for many years and data will continue to be collected in the future. Locations and schedules are detailed in the RI/FS Work Plan and FSPs. Sampling will be conducted in accordance with the SOPs established in this QAPP and site-specific work plans.

Who will collect and generate the data?

GEI and its subcontractors will collect environmental samples in coordination with USEPA-GLNPO and NYSDEC. Pace Analytical Laboratory has been retained for environmental analytical requirements. GeoTesting Express will generate geotechnical analytical data. Other subcontractors may be retained for specialized needs.

How will the data be reported?

The data will be reported by laboratories in appropriate data packages and following validation by GEI Data will be provided in electronic data deliverables (EDDs) in EPA Region 5 EDD format and EquIS format which are compatible with USEPA and NYSDEC requirements.

How will the data be archived?

Electronic files will be maintained in their native forms on GEI's network servers physically located in Woburn, Massachusetts office and mirrored in multiple other offices throughout the Unites States.

Data Quality Objectives

Data quality objectives (DQOs) are required for environmental data collection activities. DQOs are statements that define the objectives of the project; the type, quality, quantity, purpose, and use of data to be collected; and are an important planning tool used to help support data credibility. The DQO process allows the site assessment team to determine the level of data quality needed for specific data collection activities, and to estimate the costs associated with these activities. The DQO process consists of seven steps, which are used during the planning of

the site assessment process to validate the field activities, data collection operations, and to demonstrate that resulting data meet the project objectives.

Step 1: State the Problem

The existing Project Agreement between the USEPA and NYSDEC (NYSDEC Contract No. C011665) was amended in 2024 to include work previously completed by National Fuel from 2021 to 2023 related to the Site Characterization (October, 2022), Interim Remedial Measures (March, 2023) completed to mitigate contamination discharge from a City of Buffalo Pumphouse, and the completion of the Scajaquada Creek RI and FS. National Fuel is the Lead Partner for the Scajaquada Creek OU, while the USEPA is the Lead Parter responsible for completing a FS for the Black Rock Canal OU. The NYSDEC provides project oversight and conducts certain source area investigations in the Scajaquada Creek OU.

The primary goal of the Scajaquada Creek OU investigation is to collect data to support conceptual design-level evaluations that will be presented as part of a FS. Generally, the objectives of the project are delineation of Site contaminants of concern (COCs), assessment of biological risks, and determination of disposal options.

Step 2: Identify the Goals of the Study

The goals for this program include, but are not limited to:

Adequacy of DNAPL Source Control

1. Assess the presence and mobility of DNAPL in the vicinity of the West Avenue bridge to evaluate the effectiveness of upstream physical barriers and DNAPL containment and/or collection and removal systems in preventing DNAPL transport to the south. While residual DNAPL is expected, it will be important to assess mobility and its potential to be a continuing source.

2. Inspect IRM work completed to seal the manholes and conveyance piping for DNAPL entry and groundwater infiltration, as feasible. Water quality will be evaluated by collecting samples from the stormwater conveyance manholes and water discharged at the Pumphouse outfall.

Identification of Utilities and Infrastructure as DNAPL Migration Pathways

1. Identify subsurface utilities that occur at depths where DNAPL has been identified and assess their potential as pathway for migration.

Extent of DNAPL in Shallow Creek Sediments

1. Use probing and cores to map the distribution DNAPL presence in shallow sediments near the Pumphouse discharge outfall. While DNAPL presence in shallow sediments was primarily observed in the creek Inlet, a comprehensive probing survey of the shallow creek sediments is needed for verification between the West Avenue bridge and the Black Rock Channel, including other areas of the Inlet.

2. Characterize sediment composition including total organic carbon content, DNAPL extents (lateral and vertical) and hydraulic influences from daily Black Rock canal water level fluctuations.

Sediment Quality and Ecological Risks

1. Assess sediment quality in the Lower Scajaquada Creek and Inlet with a detailed emphasis on known COCs including volatile and extractable petroleum hydrocarbons, PAHs, PCBs, and metals (mercury, copper, lead, nickel, and zinc) to evaluate the risks to benthos, fish, and wildlife. Identify the information needed to evaluate remedial alternatives aimed at removing beneficial use impairments (BUIs) in the creek.

Extent of DNAPL in Deeper Soil

1. Establish the horizontal and vertical extent of DNAPL in the OU in consideration of potential migration pathways in natural geologic materials and manmade pathways associated with buried urban infrastructure. This would include evaluating the northern extent of DNAPL in soils beyond the 1660 Niagara Street BCP site and the soil located near the former outlet of Scajaquada Creek to the Black Rock Canal below the I-190 Thruway. Fully characterizing the surface of the area-wide red-brown clay layer in the Lower Scajaquada Creek Area is important as it plays a major role in the fate and transport of DNAPL.

Shallow Groundwater Quality

1. Wells installed during the 2021/2022 SC were equipped with well screens extending through the deeper alluvial soils into the top of the clay layer. The presence of DNAPL impacts groundwater quality at this depth. Assess whether the presence of deeper DNAPL affects the groundwater quality near the water table, which is significantly shallower than the area-wide clay layer.

Information to Support the FS

1. Data should be collected in and near the Inlet to evaluate the eastern wall adjacent to 1660 Niagara Street, the sheet pile wall adjacent to the I-190 Thruway, and piers adjacent to the creek. Data will also be collected to characterize potential treatment and disposal options for media that may be considered for removal under FS alternatives.

Areas of the Site with concentrations above the respective screening level will be delineated. Screening levels used to evaluate chemical results from existing and proposed sampling are consistent with those used in the Site Characterization Report (GEI, 2022):

- Soil data will be compared to the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted use, Restricted-Residential, and Protection of Ecological Resources.

- Sediment data will be compared to the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) Chapter IV, Part 375-6 SCOs: Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B.

- Groundwater data will be compared to New York State Ambient Water Quality Standards and Guidance Values for GA groundwater (NYS AWQS) and NYSDEC Interim Groundwater Screening Levels based on NYSDEC guidance as follows:

    - March 2023 (1,4 Dioxane and per- and polyfluoroalkyl substances [PFAS])

For many individual PCB Aroclors and individual PAHs, screening levels are not available. Screening for total PCBs and PAHs will be performed by summing the individual analytes and comparing the total concentrations to the guidelines provided for total PAHs and total PCBs (Worksheet #15). The selected screening criteria are designed to be protective of the environment. Total PAH values will be calculated by summing the detected values and using a value equal to half the reporting limit (RL) for non-detects (non-detect = ½ RL). Total PCB values will be calculated by summing the detected values and using a value of zero for non-detects (non-detect = 0). The use of zero for non-detected PCBs assures that the total PCB concentration is not overestimated. Aroclor concentrations overlap each other, and many Aroclor mixtures are not common in the environment. The total PAH and PCB concentrations will be compared to the total PAH and total PCB sediment screening criteria.

Biological risks will be assessed via comparison of concentrations to screening levels, toxicity testing, and bioaccumulation testing, The results of the toxicity and bioaccumulation test will be statistically compared to the controls, reference, and pre-test (bioaccumulation testing only).

Step 3: Identify Information Inputs

Required data and information needed to meet the goals of the study:

Source Control Assessment

An assessment will be performed to verify the effectiveness of the Iroquois Gas/Westwood Pharmaceutical Terrestrial/Riparian Site Remedies in controlling DNAPL. This assessment will

include a review of the DNAPL collection records from the Southern DNAPL collection system at the Riparian Site, subsurface soil explorations, and assessments of DNAPL mobility, where identified. Subsurface soil investigations will be completed on land and in the creek and Inlet to assess the lateral and vertical extent of DNAPL impacted soils. Locations of the planned subsurface soil borings are included in Figures 5, 6, and 7 in the RI/FS Work Plan. Where DNAPL is encountered, wells will be installed to perform DNAPL mobility tests.

Subsurface Soil Investigation

Soil borings will be completed to evaluate the lateral and vertical extent of DNAPL presence in the subsurface soil. Locations of the planned subsurface soil borings are included in Figures 5, 6, and 7 in the RI/FS Work Plan. If DNAPL is encountered at the primary boring location, a contingency boring will be completed at a "Stepped out location" to satisfy the objective of mapping the extent of DNAPL. At select locations, soil borings will be completed as groundwater monitoring wells to evaluate the groundwater quality and for the assessment of DNAPL mobility.

At each proposed boring location, the boring will be advanced through fill and native soil and then a minimum of four feet into the red-brown clay unit. If DNAPL is observed within the top four feet of the native red-brown clay unit, the soil boring will be advanced until no visual evidence of impact is observed. Select deeper borings are proposed to evaluate the potential for DNAPL impacts below the red-brown clay unit into the underlying sand unit which directly overlies bedrock.

A minimum of two soil samples will be collected from each boring and submitted for laboratory analysis for the following:

- TCL VOCs – EPA Method 8260D/5035
- PAH-17 – EPA Method 8270E
- COC Metals (arsenic, copper, lead, mercury, nickel, and zinc) – EPA Method 6020B/7471B
- PCB – Aroclors – EPA Method 8082A
- Grain Size analysis – ASTM D422, Standard Penetration Testing (SPT) – ASTM D1586, Total Organic Carbon (TOC) –EPA Method 9060A/Lloyd Kahn

Groundwater Investigation

The groundwater investigation will be performed to assess groundwater quality in the unconfined water-bearing zone where DNAPL is known to exist. Locations of the planned monitoring wells are included in Figures 5, 6, and 7 in the RI/FS Work Plan. In areas where DNAPL presence is to

be investigated, well will be installed to screen the bottom of the alluvial soils directly above the area-wide clay layer which forms a barrier to vertical downward migration of DNAPL. These wells will also be used to support DNAPL mobility testing, if DNAPL is present.

In areas where DNAPL-impacted soils were identified and shallow wells are not present, monitoring wells will be installed with wells screens positioned to monitor the upper five feet of the saturated zone or five feet below the top of the water table as observed during drilling.

Following well development, new monitoring wells will be allowed to stabilize for at least two weeks and then sampled using low flow sampling methods.

Groundwater samples will be submitted for the following analyses:

- TCL VOCs – EPA Method 8260D
- TCL SVOCs – EPA Method 8270E including 1,4 dioxane by EPA Method 8270E – SIM with isotope dilution
- PFAS – EPA Method 1633
- PCB – Aroclors – EPA Method 8082A
- TAL Metals – EPA Method 6020B/7470A
- Total Cyanide – EPA Method 9012B

If NAPL is encountered in the new monitoring wells, and if observations based on olfactory and visual evidence, along with groundwater analytical testing results, indicate that the NAPL is inconsistent with previous findings (e.g., coal-tar like odor with elevated BTEX and naphthalene concentrations in the aqueous phase), a sample of the NAPL will be collected and submitted for forensic analysis using EPA Methods 8270E-SIM/8015D(M).

Inlet Creek Bank Surface Soil Investigation

Surface soil samples will be collected from the creek bank and analyzed for the following parameters:

- TCL VOCs – EPA Method 8260D/5035
- PAH-17 – EPA Method 8270E
- COC Metals (arsenic, copper, lead, mercury, nickel, and zinc) – EPA Method 6020B/7471B
- PCB – Aroclors – EPA Method 8082A

Locations of the planned surface soil samples are included in Figures 5, 6, and 7 in the RI/FS Work Plan.

Bathymetric Survey

A bathymetric survey of the investigation area will be performed using a single-beam bathymetric survey methodology with global positioning system (GPS) location. Sufficient points along the shore will be obtained so that the bathymetric survey can be tied to the upland topographic survey and a unified map of land and creek-bottom elevations.

Sediment Probing Transects

Systematic sediment probing will be performed to assess the thickness and quality of sediment within the investigation area and observe for sheen generation. Locations of the planned sediment probing transects are included in Figures 8, 9, and 10 in the RI/FS Work Plan.

Sediment Sampling

Sediment borings will be completed in the Scajaquada Creek main channel and Inlet to evaluate sediment quality. Locations of the planned sediment borings are included in Figures 8, 9, and 10 in the RI/FS Work Plan. At all sediment boring locations, the full length of recovered sediment/soil and underlying clay layer will be visually assessed. At certain locations, sediment will be collected from selected intervals for laboratory analysis. The target intervals for sampling are generally the upper two feet of sediment and the most impacted interval observed in the core. In the event the 0- to 2-ft depth interval is also most visibly impacted, or if no depth interval appears more impacted than others, the second sample will be collected from the 2-foot interval overlying the red-brown clay. Each sediment boring will advance a minimum of 4 feet into the area-wide red-brown clay layer.

Sediment samples will be analyzed for chemical, biological, waste profiling, and geotechnical purposes.

For COC delineation sediment samples, the associated analytical methods are:

- TCL VOCs – EPA Method 8260D/5035
- PAH-17 – EPA Method 8270E
- TCL SVOCs expanded National Oceanic and Atmospheric Administration (NOAA) PAH 34 – EPA Method 8270E-SIM
- PCB – Aroclors – EPA Method 8082A
- COC Metals (arsenic, copper, lead, mercury, nickel, and zinc) – EPA Method 6020B/7471B

- TAL Metals – EPA Method 6010D/7471B
- Total Cyanide – EPA Method 9012B
- TOC – EPA Method 9060A/Lloyd Kahn
- Extractable Petroleum Hydrocarbons (EPH) – MassDEP (MA-EPH)

For ecological risk assessment sediment samples, the associated analytical methods are:

- Ammonia – 4500NH3-BH
- TOC – EPA Method 9060A/Lloyd Kahn
- Sulfide – 9030B
- Acid Volatile Sulfide/Simultaneously Extracted Metals (AVS/SEM)
- Grain Size – ASTM D422

Standard benthic toxicity testing will include conducting chronic, 28-day whole-sediment toxicity test with the amphipod, *Hyalella azteca.* Endpoints measured in this chronic test will include effects on survival, growth, and reproduction from 28-day exposure to site sediments per "Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminants with Freshwater Invertebrates" (USEPA, 2000) and "A Guidance Manual to Support the Assessment of Contaminated Sediments in Freshwater Ecosystems" (USEPA, 2002).

The evaluation of waste management costs in the FS requires evaluation of chemistry and characteristics for sediment that may be dredged from creek and subject to offsite disposal. These analyses, combined with the suite of area-wide chemical tests, will allow for evaluation for both landfill and thermal treatment disposal options. Analyses for waste characterization will be performed on composite samples created from the uppermost 4 feet or full thickness of depositional material retrieved at each location, whichever is less. The analytical methods are summarized below:

- TCL and TCLP VOCs – EPA Method 8260D/5035
- TCL and TCLP SVOCs – EPA Method 8270E
- TAL and TCLP Metals – EPA Method 6010D/7471B/6020B/7470A
- PCB-Aroclors – EPA Method 8082A
- pH, ignitability, sulfide and cyanide reactivity

- Corrosivity
- Total Cyanide – EPA Method 9012B
- Total Petroleum Hydrocarbons (TPH) – EPA Method 8015D
- Percent Sulfur – ASTM D4239
- BTU – ASTM D240-87

The evaluation of certain remedial technologies for the creek as part of the planned FS may require an assessment of the effects of each technology on the stability of roads and structures along the creek. Knowledge of geotechnical properties may also be pertinent to designing support facilities, sediment dewatering, excavation support, and other ancillary activities that may be needed to implement certain remedial technologies. To support this assessment, limited geotechnical testing will be performed and focus on the following areas:

- Along the bulkhead supporting highway I-190 and closing the former creek outlet.
- Between the State Route 198 on-ramp and Niagara Street bridge
- Adjacent to the West Avenue bridge

At each of these locations, samples will be collected from the clay at the bottom of the sediment core and analyzed for the following:

- Grain Size – ASTM D422
- Atterberg Limits
- Permeability (if intact Shelby tube samples can be recovered)

DNAPL Mobility Assessments

DNAPL mobility assessments will be conducted at:

- Wells installed during the RI containing DNAPL
- Temporary wells installed at creek borings (if DNAPL is observed)
- Existing monitoring wells identified during the 2021/2022 SC that contained DNAPL.

Evaluation of DNAPL Migration Pathways Associated with Infrastructure

During the 2022 SC, numerous utilities and below-grade infrastructure was identified in the lower Scajaquada Creek area. An IRM was implemented to minimize contaminated groundwater from nearby BCP sites from infiltrating the storm sewer system and to mitigate the entry of DNAPL, including accumulated DNAPL. Although sampling was conducted shortly after the IRM's completion, the performance of the IRM has not been quantified through analytical testing.

The following will be conducted to assess DNAPL/contaminant groundwater entry into the Niagara St. storm sewer servicing the City of Buffalo Pumphouse:

- Manholes #1 through #4 in Niagara Street, Manholes 21 and 23, and the Pumphouse vault will be visually inspected for coal-tar DNAPL presence. These features are included in Section 4 of the RI/FS Work Plan.

- Groundwater samples will be collected from Manholes 21 and 23 and the Pumphouse discharge and analyzed for the full suite of compounds.

Step 4: Define the Boundaries of Data Collection

The study area includes:

- The lands adjacent to Scajaquada Creek, extending south of the West Avenue Bridge to the creek's mouth where it discharges into the Black Rock Canal.

- The Scajaquada Creek Slip Area (Inlet Area), which was historically the creek's discharge location before being blocked during the construction of the I-190 New York State Thruway.

- Upland areas include properties owned by the former Penn Central Railroad, New York State, and City of Buffalo that are used for transportation and infrastructure corridors including a railroad passenger and freight train corridor, the NYS Thruway I-190, the Route 198 Scajaquada Expressway, and City of Buffalo roadways (West Avenue, Tonawanda Street, and Niagara Street).

- The upland areas also include privately owned properties, many of which were former industrial properties with environmental concerns, many of which have been investigated, and some remediated and developed for reuse in the NYSDEC Brownfield Cleanup Program or former Voluntary Cleanup Program.

Step 5: Develop the Analytical Approach

- **Sample type:** Sample types are expected to include, but are not necessarily limited to, the following:

    - Subsurface soil. Soil sampling may be conducted by mechanical means or manual means.

    - Creek and inlet area sediment. Sediment sampling may be conducted by mechanical (e.g., macrocore) means or manual means, and may be collected from a boat, barge, platform, or in dewatered areas.

    - Groundwater. Groundwater sampling may be collected by mechanical (e.g., low-flow sampling) or manual means.

    - Geotechnical information, to be collected by various means, needed for remedial alternatives analysis.

    - Additional soil, sediment, and/or groundwater samples for disposal or reuse purposes.

- **Anticipated analytical methods:** Analytical methods proposed during the RI/FS implementation include:

    - Soil Samples
        - TCL VOCs by EPA Method 8260D/5035
        - PAH-17 by EPA Method 8270E
        - PCB – Aroclors by EPA Method 8082A
        - COC Metals (arsenic, copper, lead, mercury, nickel, and zinc) by EPA Method 6020B/7471B
        - Grain Size Analysis, Standard Penetration Testing (SPT), Total Organic Carbon (TOC) (ASTM D422, D5186, EPA 9060A/Lloyd Kahn)
        - If LNAPL or DNAPL is encountered in any soil boring exhibiting properties inconsistent with prior findings of NAPL (characteristic of coal tar-like gross contamination), a sample will be collected and submitted for laboratory forensic analysis of 1,4-dioxane and total petroleum hydrocarbons (TPH) by EPA Method 8270E – SIM/8015D(M).
    - Groundwater Samples (including samples from monitoring wells and stormwater conveyance piping and pumphouse discharge):
        - TCL VOCs by EPA Method 8260D
        - TCL SVOCs by EPA Method 8270E including 1,4-dioxane by EPA Method 8270E-SIM with isotope dilution
        - PFAS by EPA Method 1633
        - PCB – Aroclors by EPA Method 8082A
        - TAL Metals by EPA Method 6020B/7470A

  - Total Cyanide by EPA Method 9012B
  - If NAPL is encountered in new monitoring wells, and if observations based on olfactory and visual evidence, along with groundwater analytical testing results, indicate that the NAPL is inconsistent with previous findings (e.g., coal-tar like odor with elevated BTEX and naphthalene concentrations in the aqueous phase), a sample of the NAPL will be collected and submitted for forensic analysis of 1,4-dioxane and TPH using EPA Methods 8270E-SIM/8015D(M).
- Inlet Creek Bank Soil Samples:
  - TCL VOCs by EPA Method 8260D
  - PAH-17 by EPA Method 8270E
  - PCB – Aroclors by EPA Method 8082A
  - COC Metals (arsenic, copper, lead, mercury, nickel, and zinc) by EPA Method 6020B/7471B
- Sediment Samples:
  - TCL VOCs by EPA Method 8260D
  - PAH-17 by EPA Method 8270E
  - TCL SVOCs expanded NOAA PAH 34 by EPA Method 8270E-SIM
  - PCB – Aroclors by EPA Method 8082A
  - COC Metals (arsenic, copper, lead, mercury, nickel, and zinc) by EPA Method 6020B/7471B
  - TAL Metals by EPA Method 6010D/7471B
  - Total Cyanide by EPA Method 9012B
  - TOC by EPA Method 9060A/Lloyd Kahn
  - Ammonia by Method 4500NH3-BH
  - Sulfide by EPA Method 9030
  - Acid Volatile Sulfide/Simultaneously Extracted Metals (AVS/SEM)
  - Grain Size by ASTM D422
  - EPH by MassDEP Method (MA-EPH)
- Sediment Toxicity Samples:
  - Standard toxicity testing will include conducting chronic, 28-day whole-sediment toxicity test with the amphipod, *Hyalella azteca.*
- Waste Characterization Samples:
  - TCL and TCLP VOCs by EPA Method 8260D
  - TCL and TCLP SVOCs by EPA Method 8270E
  - TAL and TCLP Metals by EPA Method 6010D/6020B/7470A/7471B
  - Pesticides by EPA Method 8081B
  - Herbicides by EPA Method 8151A
  - PCB – Aroclors by EPA Method 8082A
  - Ignitability by EPA Method 1030
  - Reactivity (Sulfide and Cyanide) by EPA Method 7.3
  - pH by EPA Method 9045

  - Total Cyanide by EPA Method 9012B
  - Percent Sulfur by ASTM D4239
  - BTU by ASTM D240-87
  - TPH by EPA Method 8015D
- Geotechnical Samples:
  - Grain Size by ASTM D422
  - Atterberg Limits
  - Permeability

- **Project quantification limits:** Achievable laboratory limits are specified in Worksheet #15.

- **Quality assurance/quality control:** Field QA/QC samples will be sampled at frequencies indicated in Worksheet #20. Laboratory QA/QC will be evaluated at the frequencies indicated in Worksheet #28. Worksheet #12 presents performance criteria. Chemical data will be verified and validated (Worksheets #34, 35, and 36).

- **Anticipated data evaluations:** Data evaluation needs include but are not limited to the following:

  - Delineate the vertical/horizontal extent of NAPL/DNAPL and associated dissolved phase contamination.

  - Assess the effectiveness of containment remedies previously installed upstream of West Avenue.

  - Assess the fate and transport of dissolved and non-aqueous phase contamination.

  - Evaluate utility/infrastructure potential migration pathways to the extent feasible, without causing damage or risking worker safety during investigation.

  - Evaluate potential exposure pathways.

  - Evaluate existing sediment chemistry and sediment depth data to define the extent of contamination and the estimate volumes.

  - Identify data gaps necessary to successfully complete the FS.

  - Identify and evaluate potential structural and hydraulic limitations and any other physical factors affecting the remedial decision-making process. For the Inlet, the evaluation should include, at a minimum, the eastern wall adjacent to 1660 Niagara Street and the I-190 expressway piers.

- Identify options for remedial actions that have the potential to fulfill the remedial action objectives (RAOs).
- Identify the necessary technologies and general process options for implementing each of the general remedial actions.

Step 6: Specify Performance/Acceptance Criteria

Performance and acceptance criteria for the data collected under these DQOs include data collection and data evaluation. For lab analysis, performance and acceptance criteria are detailed in Worksheets #12 and #15. Data usability criteria are detailed in Worksheet #37.

Step 7: Develop the Detailed Plan for Obtaining Data

The detailed plan for data collection is provided in Worksheet #17.

Worksheet #12-1: Measurement Performance Criteria (6010D – Metals)

Matrix: Aqueous, Solid, Sludges, and Groundwater
Analytical Group: 6010D – Metals
Laboratory Facility: Alpha Analytical, Mansfield, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1]	RPD ≤ 20% Aqueous; RPD ≤ 35% Solids	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 20%	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[2]	Recovery range 80-120%	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates[3]	Recovery range 75-125%	S&A
Overall Accuracy/Bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ ½ LOQ	S&A
Sensitivity Precision	LOQ verification sample (spiked at LOQ)	Recovery within ±25% of LOQ	A
Completeness	Project-specific	Data completeness check	S&A

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[1]Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.

[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.

[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.

DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-2: Measurement Performance Criteria (6020B – Metals)

Matrix: Aqueous, Solid, Sludges and Groundwater
Analytical Group: 6020B – Metals
Laboratory Facility: Alpha Analytical, Mansfield, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1]	RPD ≤ 20% Aqueous; RPD ≤ 35% Solids	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 20%	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[2]	Recovery range 80-120%	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates[3]	Recovery range 75-125%	S&A
Analytical Accuracy/Bias	Internal Standards	Acceptance range >70%	A
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ LOQ	S&A
Sensitivity Precision	LOQ verification sample (spiked at LOQ)	See SOP	A
Completeness	Project-specific	Data completeness check	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; SOP = Standard Operating Procedure

Worksheet #12-3: Measurement Performance Criteria (ASTM D422 – Grain Size)

Matrix: Solids
Analytical Group: ASTM D422 – Grain Size
Laboratory Facility: Alpha Analytical, Mansfield, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1]	RPD ≤ 25%	S
Analytical Precision (laboratory)	Sample Duplicates[2]	RPD ≤ 25%	A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-4: Measurement Performance Criteria (1633 – PFAS)

Matrix: Water/Solid
Analytical Group: 1633 – PFAS
Laboratory Facility: Alpha Analytical, Mansfield, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1] (at client's request)	RPD ≤ 30%	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates (optional if no Field Duplicate or MSD)[2]	RPD ≤ 30% for mid-level LCSD RPD ≤ 50% for low-level LCSD	A
Analytical Precision (laboratory)	Matrix Spike Duplicates[3] (at client's request)	Per SOP	S&A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	Per SOP	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/Matrix Spike Duplicates[3] (at client's request)	50-150%R	S&A
Accuracy/Extraction Efficiency	Extracted Internal Standards	50-150%R	A
Overall Accuracy/Bias (contamination)	Method Blank	No target compounds ≥ RL	A
Sensitivity	Method Detection Limit	MDL < RL	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
DQI = data quality indicators; QC = quality control; QA = quality assurance; MDL = method detection limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; SOP = standard operating procedure

Worksheet #12-5: Measurement Performance Criteria (9060A – Total Organic Carbon)

Matrix: Soils/Sediments
Analytical Group: 9060A/Lloyd Kahn – Total Organic Carbon
Laboratory Facility: Alpha Analytical, Mansfield, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1]	RPD ≤ 25% when TOC is detected in both samples ≥ sample-specific LOQ	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates[2]	RPD ≤ 25%	A
Analytical Accuracy/Bias (laboratory)	Standard Reference Material/Laboratory Control Sample	75-125% Recovery	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates[3]	75-125% Recovery	S&A
Overall Accuracy/Bias (contamination)	Method Blanks	No analyte at or above RL; "B" qualify if detected < 5x the blank concentration	A
Sensitivity	LOQ verification sample (spiked at LOQ)	Recovery within 90-110% of LOQ	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.

[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-6: Measurement Performance Criteria (7470A – Mercury)

Matrix: Aqueous/Groundwater
Analytical Group: 7470A – Mercury
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1]	RPD ≤ 20%	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates[2]	RPD ≤ 20%	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	Recovery range 80-120%	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates[3]	Recovery range 70-130%	S&A
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ LOQ	S&A
Sensitivity Precision	% Relative Error	Low point 50%, mid-level 30%	A
Completeness	Project-specific	Data completeness check	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-7: Measurement Performance Criteria (7471B – Mercury)

Matrix : Solid, Semi-Solid Waste
Analytical Group: 7471B – Mercury
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1]	RPD ≤ 35%	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 20%	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[2]	Recovery range 80-120%	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike Duplicates[3]	Recovery range 75-125%	S&A
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ LOQ	S&A
Sensitivity Precision	% Relative Error	Low point 50%, Mid-point 30%	A
Completeness	Project-specific	Data completeness check	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-8: Measurement Performance Criteria (4500NH3-BH – Ammonia, Nitrogen)

Matrix: Water
Analytical Group: 4500NH3-BH – Ammonia, Nitrogen
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1]	RPD ≤ 20% when analyte is detected in both samples	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	NA	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[2]	LCS 80%-120%	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/Matrix Spike Duplicates[3]	MS 80%-120%	S&A
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ RL	S&A
Sensitivity	LOD verification[4] (spiked at 2-3xDL)	Detected	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
[4] Surrogate spike amount may vary by lab. Surrogate spike should be at the upper end of curve.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-9: Measurement Performance Criteria (9012B – Cyanide, Total)

Matrix: Water/Soils
Analytical Group: 9012B – Cyanide, Total
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1]	RPD ≤ 30% when analyte is detected in both samples	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	NA	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[2]	LCS 85-115 % for method 9010 waters; 90-110 % for method 4500; 80-120% for method 9010 soils	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike[3]	MS 90-110% for waters for method 4500; 80-120 for waters for method 9010; 75-125% for soils for method 9010; 65-135% for soils for method 4500	S&A
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ RL	S&A
Sensitivity	LOD verification[4] (spiked at 2-3xDL)	Detected	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
[4] Surrogate spike amount may vary by lab. Surrogate spike should be at the upper end of curve.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-10: Measurement Performance Criteria (9045D – pH)

Matrix: Water/Soils
Analytical Group: 9045D – pH
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1]	RPD ≤ 5%	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	NA	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[2]	LCS 99-101 % (6.95-7.05)	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike[3]	NA	S&A
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ RL	S&A
Sensitivity	LOD verification[4] (spiked at 2-3xDL)	NA	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
[4] Surrogate spike amount may vary by lab. Surrogate spike should be at the upper end of curve.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-11: Measurement Performance Criteria (8260D/5035 – VOCs)

Matrix: Water/Soils
Analytical Group: 8260D/5035 – VOCs
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates[1]	RPD ≤ 20%	A
Analytical Precision (laboratory)	Matrix Spike Duplicates[2] (at client's request)	RPD ≤ 30%	S&A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[1]	Generally, 70-130%R – analyte specific	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/ Matrix Spike Duplicates[2] (at client's request)	Generally, 40-140%R – analyte specific	S&A
Accuracy/Extraction efficiency	Surrogates[3]	70-130%R	A
Analytical Accuracy	Internal Standards[1]	50-200% of the IS area count in the CCV	A
Overall accuracy/bias (contamination)	Method blank	No target compounds ≥ RL	A
Sensitivity	Method Detection Limit	MDL < RL	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[2]MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
[3] Surrogate spike amount may vary by lab. Surrogate spike should be at the upper end of curve.
DQI = data quality indicators; QC = quality control; QA = quality assurance; MDL = method detection limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-12: Measurement Performance Criteria (8270E – SVOCs)

Matrix: Solids
Analytical Group: 8270E – SVOCs
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates (at client's request)[1]	RPD ≤ 30% for target compounds > 5x LOQ	A
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates[2]	RPD ≤ 30%	A
Analytical Precision (laboratory)	Matrix Spike Duplicates (at client's request)[3]	RPD ≤ 30%	S&A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[2]	40-140%R (Base/Neutral), 30-130%R (Acid Compounds)	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/Matrix Spike Duplicates[3] (at client's request)	40-140%R (Base/Neutral), 30-130%R (Acid Compounds)	S&A
Accuracy/Extraction Efficiency	Surrogates[4]	30-130%R	A
Overall Accuracy/Bias (contamination)	Method Blank	No target compounds ≥ LOQ	A
Sensitivity	LOD verification[4] (spiked at 1-4x LOD)	Detected	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
[4] Surrogate spike amounts may vary by lab. Surrogate spike should be at the upper end of curve.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = level of detection

Worksheet #12-13: Measurement Performance Criteria (8270E – SVOCs)

Matrix: Aqueous
Analytical Group: 8270E – SVOCs
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates (at client's request)[1]	RPD ≤ 30% for target compounds > 5x LOQ	A
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates[2]	RPD ≤ 30%	A
Analytical Precision (laboratory)	Matrix Spike Duplicates (at client's request)[3]	RPD ≤ 30%	S&A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[2]	40-140%R (Base/Neutral), 30-130%R (Acid Compounds)	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/Matrix Spike Duplicates[3] (at client's request)	40-140%R (Base/Neutral), 30-130%R (Acid Compounds)	S&A
Accuracy/Extraction Efficiency	Surrogates[4]	30-130%R (Base/Neutral), 15-115%R (Acid Compounds)	A
Overall Accuracy/Bias (contamination)	Method Blank	No target compounds ≥ LOQ	A
Sensitivity	LOD verification[4] (spiked at 1-4x LOD)	Detected	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2] Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
[4] Surrogate spike amount may vary by lab. Surrogate spike should be at the upper end of curve.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #12-14: Measurement Performance Criteria (8270E SIM – 1,4 Dioxane)

Matrix: Water/Soils
Analytical Group: 8270E SIM – 1,4 Dioxane
Laboratory Facility: Alpha Analytical, Mansfield, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1] (at client's request)	RPD ≤ 30% for target compounds > 5x QL	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates[2]	RPD ≤ 30%	A
Analytical Precision (laboratory)	Matrix Spike Duplicates[3] (at client's request)	RPD ≤ 30%	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	40-140%R	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/Matrix Spike Duplicates[3] (at client's request)	40-140%R	S&A
Accuracy/Extraction Efficiency	Surrogates[4]	15-110%R	A
Overall Accuracy/Bias (contamination)	Method Blank	No target compounds ≥ QL	A
Sensitivity	LOD verification (spiked at 1-4x DL)	Detected	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
[4] Surrogate spike amount may vary by lab. Surrogate spike should be at the upper end of curve.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #12-15: Measurement Performance Criteria (8151A – Herbicides)

Matrix: Soils and Waters
Analytical Group: 8151A – Herbicides
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates[1]	RPD ≤ 25%	A
Analytical Precision (laboratory)	Matrix Spike Duplicates[2] (at client's request)	RPD ≤ 30%	S&A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[1]	30-150%R	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/ Matrix Spike Duplicates[2] (at client's request)	30-150%R	S&A
Accuracy/Extraction efficiency	Surrogates[3]	30-150%R	A
Analytical Accuracy	Internal Standards	50-200% of the IS area count in the CCV	A
Overall accuracy/bias (contamination)	Method blank	No target compounds ≥ RL	A
Sensitivity	Method Detection Limit	MDL < RL	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[2] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
[3] Surrogate spike amounts may vary by lab. Surrogate spike should be at the upper end of curve.
DQI = data quality indicators; QC = quality control; QA = quality assurance; MDL = method detection limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-16: Measurement Performance Criteria (8081B – Pesticides)

Matrix: Soils and Waters
Analytical Group: 8081B – Pesticides
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1] (at client's request)	RPD ≤ 30%	A
Analytical Precision (laboratory)	Matrix Spike Duplicates[2] (at client's request)	RPD ≤ 30%	S&A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[3]	30-150%R	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/ Matrix Spike Duplicates[2] (at client's request)	30-150%R	S&A
Accuracy/Extraction efficiency	Surrogates[4]	30-150%R	A
Analytical Accuracy	Internal Standards[3]	50-200% of the IS area count in the CCV	A
Overall accuracy/bias (contamination)	Method blank	No target compounds ≥ RL	A
Sensitivity	Method Detection Limit	MDL < RL	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
[3] Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[4] Surrogate spike amount may vary by lab. Surrogate spike should be at the upper end of curve.
DQI = data quality indicators; QC = quality control; QA = quality assurance; MDL = method detection limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-17: Measurement Performance Criteria (8082A – PCB Aroclors)

Matrix: Soils and Waters
Analytical Group: 8082A – PCB Aroclors
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates[1]	RPD ≤ 30%	A
Analytical Precision (laboratory)	Matrix Spike Duplicates[2] (at client's request)	RPD ≤ 30%	S&A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[1]	40-140%R	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/ Matrix Spike Duplicates[2] (at client's request)	40-140%R	S&A
Accuracy/Extraction efficiency	Surrogates[3]	30-150%R	A
Analytical Accuracy	Internal Standards[1]	50-200% of the IS area count in the CCV	A
Overall accuracy/bias (contamination)	Method blank	No target compounds ≥ RL	A
Sensitivity	Method Detection Limit	MDL < RL	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[2] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
[2] Surrogate spike amount may vary by lab. Surrogate spike should be at the upper end of curve.
DQI = data quality indicators; QC = quality control; QA = quality assurance; MDL = method detection limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; CCV = continuing calibration verification

Worksheet #12-18: Measurement Performance Criteria (8015D(M) – TPH)

Matrix: Solids and Waters
Analytical Group: 8015D(M) – TPH
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates[1]	RPD ≤ 30%	A
Analytical Precision (laboratory)	Matrix Spike Duplicates[2] (at client's request)	RPD ≤ 30%	S&A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[1]	40-140%R	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/ Matrix Spike Duplicates[2] (at client's request)	40-140%R	S&A
Accuracy/Extraction efficiency	Surrogates[3]	30-130%R	A
Overall accuracy/bias (contamination)	Method blank	No target compounds ≥ RL	A
Sensitivity	Method Detection Limit	MDL < RL	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[2] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
[3] Surrogate spike amount may vary by lab. Surrogate spike should be at the upper end of curve.
DQI = data quality indicators; QC = quality control; QA = quality assurance; MDL = method detection limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-19: Measurement Performance Criteria (7.3 – Sulfide, Reactive)

Matrix: Water/Solids
Analytical Group: 7.3 – Sulfide, Reactive
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1]	RPD ≤ 20% when analyte is detected in both samples	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates[2]	Aqueous: % RPD ≤ 25% Solid: % RPD ≤ 40%	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[2]	LCS 60-125%	A
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ RL	S&A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-20: Measurement Performance Criteria (7.3 – Cyanide, Reactive)

Matrix: Water/Solids
Analytical Group: 7.3 – Cyanide, Reactive
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1]	RPD ≤ 20% when analyte is detected in both samples	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates[2]	Aqueous: % RPD ≤ 25% Solid: % RPD ≤ 40%	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[2]	LCS 30-125%	A
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ RL	S&A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-21: Measurement Performance Criteria (ASTM D420-87 – BTU)

Matrix: Solids
Analytical Group: ASTM D240-87 – Calorific Value (BTU/LB)
Laboratory Facility: Alpha Analytical, Asheville, NC

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1]	RPD ≤ 10%	S
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-22: Measurement Performance Criteria (ASTM D4239 – Percent Sulfur)

Matrix: Solids
Analytical Group: ASTM D4239 – Percent Sulfur
Laboratory Facility: Alpha Analytical, Asheville, NC

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Completeness	See Worksheet #34	See Worksheet #34	S&A

S = sampling; A = analytical

Worksheet #12-23: Measurement Performance Criteria (AVS/SEM)

Matrix: Sediment
Analytical Group: Acid Volatile Sulfide and Simultaneously Extracted Metals (AVS/SEM)
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Duplicates[1]	RPD ≤ 20%	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates[2]	RPD ≤ 20%	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[2]	LCS 85-115%	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike[3]	MS 75-125%	S&A
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ RL	S&A
Sensitivity	Detection Limits	Detection Limits ≤ to PALs	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-24: Measurement Performance Criteria (9030B – Sulfide)

Matrix: Sediment
Analytical Group: 9030B – Sulfide
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1]	RPD ≤ 20% when analyte is detected in both samples	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates[2]	RPD ≤ 25%	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples[2]	LCS 75-125%	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike[3]	%R within in-house limits	S&A
Overall accuracy/bias (contamination)	Equipment Blanks	No target analyte concentrations ≥ RL	S&A
Sensitivity	Detection Limits	Detection Limits ≤ to PALs	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-25: Measurement Performance Criteria (1030 – Ignitability)

Matrix: Solids
Analytical Group: 1030 – Ignitability
Laboratory Facility: Alpha Analytical, Asheville, NC

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Completeness	See Worksheet #34	See Worksheet #34	S&A

S = sampling; A = analytical

Worksheet #12-26: Measurement Performance Criteria (8270E – SIM PAHs)

Matrix: Sediment
Analytical Group: 8270E – SIM PAHs
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria[2]	QC Sample Assesses Error for Sampling, Analytical, or Both
Overall Precision	Field Duplicates[1] (at client's request)	RPD ≤ 30% for target compounds > 5x QL	S
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 30%	
Analytical Precision (laboratory)	Matrix Spike Duplicates (at client's request)	RPD ≤ 30%	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	40-140%R	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/Matrix Spike Duplicates (at client's request)	40-140%R	S&A
Analytical Accuracy/Bias (matrix interference)	Standard Reference Material (at client's request)	40-140%R (no SRM for Water matrix)	S&A
Accuracy/Extraction efficiency	Surrogates	30-130%R	A
Overall Accuracy/Bias (contamination)	Method Blank	No target compounds ≥ QL	
Sensitivity	LOD verification (spiked at 1-4x DL)	Detected	
Completeness	See Worksheet #37	See Worksheet #37	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #12-27: Measurement Performance Criteria (MassDEP Method – EPH)

Matrix: Sediment
Analytical Group: EPH
Laboratory Facility: Alpha Analytical, Westborough, MA

Data Quality Indicators (DQIs)	QA Sample and/or Activity Used to Assess Measurement Performance	Measurement Performance Criteria	QC Sample Assesses Error for Sampling, Analytical, or Both
Analytical Precision (laboratory)	Laboratory Control Sample Duplicates	RPD ≤ 30%	A
Analytical Precision (laboratory)	Matrix Spike Duplicates (at client's request)	RPD ≤ 30%	A
Analytical Accuracy/Bias (laboratory)	Laboratory Control Samples	40-140%R	A
Analytical Accuracy/Bias (matrix interference)	Matrix Spike/ Matrix Spike Duplicates (at client's request)	40-140%R	S&A
Accuracy/Extraction efficiency	Surrogates	30-130%R	A
Overall accuracy/bias (contamination)	Method blank	No target compounds ≥ RL	A
Sensitivity	Method Detection Limit	MDL < RL	A
Completeness	See Worksheet #34	See Worksheet #34	S&A

[1] Field duplicate data review guidance as referenced in Region 1 Environmental Data Review Supplement for Region 1 Data Review Elements and Superfund Specific Guidance/Procedures, June 2018 https://www.epa.gov/sites/production/files/2018-06/documents/r1-dr-supplement-june-2018.pdf.
[2]Internally derived limits are updated periodically; current limits will be used at the time of analyses.
[3] MS and MSD must be client-provided (where applicable). Laboratory may select project samples to be spiked for batch accuracy and precision. LCS/LCSD performed when no MS/MSD are supplied.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation

Worksheet #13: Secondary Data Criteria and Limitations Table

Secondary Data	Data Source (Originating Organization, Report Title, and Date)	Data Generator(s) (Originating Organization, Data Types, Data Generation/Collection Dates)	How Data Will Be Used	Limitations on Data Use
Site Characterization	Site Characterization Report Former Buffalo Gaslight Iroquois Gas Corp. Site and Lower Scajaquada Creek (GEI Consultants, Inc. P.C. June 2022, Revised October 2022)	GEI Consultants, Inc.	Soil, sediment, and groundwater characterization	None
Site Characterization	Pre-Design Investigation and Interim Remedial Measures Report – Lower Scajaquada Creek City of Buffalo Pumphouse (GEI Consultants, Inc. P.C. March 2023)	GEI Consultants, Inc.	Soil and groundwater characterization	None
Sediment sampling in inlet	Sediment Sampling – Scajaquada Creek Slip behind 1654 Niagara Street (NYSDEC/Empire GEO Services, 2015)	NYSDEC/Empire Geo Services, Inc.	Sediment characterization	None
DNAPL in Wells at 1660 Niagara St.	Phase II Environmental Site Assessment, 1660 Niagara Street (Labella Associates, 2016)	Labella Associates, P.C./NYSDEC	DNAPL extent	None
Sediment sampling in main channel and inlet	Summary Report for Sediment Screening Assessment Within the Lower Scajaquada Creek Corridor and Black Rock Canal (NYSDEC/Empire GEO Services, 2017)	NYSDEC/Empire Geo Services, Inc.	Sediment characterization	None

Worksheet #14: Summary of Project Tasks

Sampling Tasks:

- Sample soil using hollow stem augers or using a Geoprobe traditional or dual-tube sampling system for collection of samples for chemical analysis.
    - Soil borings will be completed to evaluate the lateral and vertical extent of DNAPL in the subsurface soil.
    - A total of 28 borings are identified as either Primary or Contingency borings.
        - Primary borings will be completed to refine the extent of known DNAPL presence.
        - If DNAPL is identified at the Primary boring location, a Contingency boring will be completed at a "stepped out location" to satisfy the objective of mapping the extent of DNAPL. If the Contingency boring contains DNAPL, a "stepped out location" will be completed with consultation with the NYSDEC.
        - At select locations, soil borings will be completed as groundwater monitoring wells to evaluate groundwater quality, flow direction, and for the assessment of DNAPL mobility.
- Sample sediment using drilling methods described in the FSP for collection of samples for chemical analysis (Appendix B of the RI/FS Work Plan). If recovery is poor (e.g., less than 50%), alternate sample collection methods may be employed.
- Sample bank soil samples from the creek using drilling methods and sampling systems described in the FSP for collection of samples for chemical analysis (Appendix B of the RI/FS Work Plan).
- Sample groundwater using low flow sampling methods for chemical analysis.
- Sample soil, sediment, and groundwater for waste characterization analysis.
- Sample sediment at locations adjacent to structures that may be most susceptible to being destabilized by certain remedial activities, with a focus on the following areas:
    - Along the bulkhead supporting highway I-190 and closing the former creek outlet (T2A, T3A, and T5A);

  - Between the State Route 198 on ramp and Niagara Street bridge (T16B); and
  - Adjacent to the West Avenue bridge (T26A and T26B).

- Digitally photograph sampling locations, recovered sample cores, and sampling activities.
- Install monitoring wells in areas where DNAPL-impacted soils are to be investigated, and shallow wells are not present.
  - Well screens will be positioned to monitor the upper 5 feet of the saturated zone.
- Install monitoring wells to determine DNAPL presences and assess groundwater quality in the unconfined water-bearing zone where DNAPL is known to exist.
  - Well screens will be positioned to monitor the bottom of the alluvial soils directly above the area-wide clay layer which forms a barrier to vertical downward migration of DNAPL.
- Develop newly installed monitoring wells and check for NAPL presence (LNAPL and DNAPL).
  - If NAPL is encountered, the field observations will be documented and an assessment of NAPL presence and mobility will be conducted.
- Perform slug testing using rising head methods from RI monitoring wells and monitoring wells installed during the 2021/2022 SC.
- Perform mapping and surveying work by a combination of standard Geographical Information Services (GIS) mapping combined with survey-grade GPS methods and bathymetric surveying.
- Inspecting Manholes #1 through #4 in Niagara Street, Manholes #21 and #23, and the Pumphouse vault.
- Sample water from Manholes #21 and #23 and the Pumphouse discharge.
- Collect groundwater elevation measurements (synoptic rounds) monthly for a duration of six months and quarterly for one year afterward.
- Perform community air monitoring to provide real-time measurements of total VOCs and particulate (airborne dust) concentrations in air at the downwind perimeter of each designated work area when intrusive activities are in progress.

Sample Processing and Analysis Tasks:

- Process, prepare, and analyze soil samples for:
    - TCL VOCs by EPA Method 8260D/5035
    - PAH-17 by EPA Method 8270E
    - PCB – Aroclors by EPA Method 8082A
    - COC Metals (arsenic, copper, lead, mercury, nickel, and zinc) by EPA Method 6020B/7471B
    - Grain Size Analysis, Standard Penetration Testing (SPT), Total Organic Carbon (TOC) (ASTM D422, D5186, EPA 9060A)
    - If LNAPL or DNAPL is encountered in any soil boring exhibiting properties inconsistent with prior findings of NAPL (characteristic of coal tar-like gross contamination), a sample will be collected and submitted for laboratory forensic analysis by method 8270E – SIM/8015D(M).
- Prepare and analyze groundwater samples for:
    - TCL VOCs by EPA Method 8260D
    - TCL SVOCs by EPA Method 8270E-SIM including 1,4 Dioxane by EPA Method 8270E-SIM with isotope dilution
    - PFAS by EPA Method 1633
    - PCB – Aroclors by EPA Method 8082A
    - TAL Metals by EPA Method 6010D/7470A
    - Total Cyanide by EPA Method 9012B
    - If NAPL is encountered in new monitoring wells, and if observations based on olfactory and visual evidence, along with groundwater analytical testing results, indicate that the NAPL is inconsistent with previous findings (e.g., coal-tar like odor with elevated BTEX and naphthalene concentrations in the aqueous phase), a sample of the NAPL will be collected and submitted for forensic analysis using EPA Methods 8270E-SIM/8015D(M).
- Process, prepare, and analyze soil samples collected in the Inlet Creek Bank for:
    - TCL VOCs by EPA Method 80260D
    - PAH-17 by EPA Method 8270E

  - PCB – Aroclors by EPA Method 8082A
  - COC Metals (arsenic, copper, lead, mercury, nickel, and zinc) by EPA Method 6020B/7471B

- Process, prepare, and analyze sediment samples collected for COC delineation for:
  - TCL VOCs by EPA Method 80260D
  - TCL SVOCs expanded NOAA PAH 34 – EPA Method 8270E-SIM
  - PAH-17 by EPA Method 8270E
  - PCB – Aroclors by EPA Method 8082A
  - COC Metals (arsenic, copper, lead, mercury, nickel, and zinc) by EPA Method 6020B/7471B
  - TAL Metals by EPA Method 6010D/7471B
  - Total Cyanide by EPA Method 9012B
  - TOC by EPA Method 9060A/Lloyd Kahn
  - EPH by MassDEP Method (MA-EPH)
- Process, prepare, and analyze select sediment samples for:
  - Ammonia by Method 4500NH3-BH
  - TOC by EPA Method 9060A
  - Sulfide by EPA Method 9030B
  - Acid Volatile Sulfide/Simultaneously Extracted Metals (AVS/SEM)
  - Grain Size by ASTM D422
- Process, prepare, and analyze select sediment samples for laboratory toxicity testing.
  - Standard toxicity testing will include conducting chronic, 28-day whole-sediment toxicity test with the amphipod, *Hyalella azteca.*
- Process, prepare, and analyze soil, sediment, and groundwater waste characterization samples for:

  - TCL and TCLP VOCs by EPA Method 8260/5035D
  - TCL and TCLP SVOCs by EPA Method 8270E
  - TAL and TCLP Metals by EPA Method 6010D/6020B/7470A/7471B
  - Pesticides by EPA Method 8081B
  - Herbicides by EPA Method 8151A
  - PCB – Aroclors by EPA Method 8082A
  - Ignitability by EPA Method 1030
  - Reactivity (Sulfide and Cyanide) by EPA Method 7.3
  - pH by EPA Method 9045D
  - Total Cyanide by EPA Method 9012B
  - Percent Sulfur by ASTM D4239
  - BTU by ASTM D240-87
  - TPH by EPA Method 8015D
- Process, prepare, and analyze sediment geotechnical sample for:
  - Grain Size by ASTM D422
  - Atterberg Limits
  - Permeability

Quality Control Tasks:

- Samples collected during investigation activities will be collected, handled, and disposed of in accordance with field SOPs presented as an appendix to this QAPP (Appendix A).

Secondary Data:

- See Worksheet #13.

Other Data:

- Other data may be acquired or consulted as needed to meet project objectives.

Data Management Tasks:

- GEI will manage the analytical data in data spreadsheets and/or relational databases.
- A comprehensive electronic database will be maintained for all laboratory analytical data and survey data generated throughout the lifespan of the project. This electronic database shall be shared with the RPM quarterly.

Documentation and Records:

Project data and information will be tracked and managed from its generation in the field to storage.

Document Control

- Official and original documents relating to the investigation must be properly stored in a centralized project file location.
- Relevant hard and electronic copies, records, reports, correspondence, logs, field logbooks, photographs, subcontractor reports, data, etc. will be maintained by the GEI Project Manager (and GEI Project QAM) in a secured, limited access area, in accordance with contract specifications and GEI's records retention policy.
- As part of the document control system, the QAPP will be reviewed and updated by the GEI Project Quality Assurance Manager (QAM), as necessary, if project activities substantially change or if new activities are authorized.
- Documents, records, photographs, and information relating to field activities will be maintained in the project file via electronic files and/or hard copy. Field data will be captured in compliance with the GLLA Data Report Standard Guidance Document, the Locational Data Checklist, Metadata Reporting Form, and the Great Lakes National Program Office (GLNPO) Requirements for Submission of Field Data for Sediment Projects (Appendix D). Field logbooks, field forms, and photographs will be scanned/uploaded daily and stored electronically.
- Other records of sample collection activities will include data collection logs, chain-of-custody records, custody seals, sample labels, phone conversation records, airbills, and corrective action reports. Chain-of-custody records will be used to document the progression of field samples and QC samples. These records will be uploaded and stored electronically by a GEI field team member.

Computer and Hardcopy Storage

- Electronic files and project deliverables will be maintained in a centralized location and retained for a minimum of 10 years. In addition, electronic data will be backed up on a nightly basis.

- Hard copy project files and data, such as original field forms and calculations, will be stored in a centralized project file location in the GEI office in Buffalo, New York. Hardcopy files will be maintained for a minimum of 10 years.

- Should questions arise concerning the data at any point in the project, Rick Frappa (GEI Project Manager) can be contacted at rfrappa@geiconsultants.com for support.

Field Documentation and Records

- **Field Sample Identification –** The following nomenclature will be used for field sample identification will be described in Work Plans or Field Sampling Plans for the Site.

    - Sample Locations: Landside Investigation

        | Sample Type | - | Core/Unique Identification (ID) and/or Number |

            - The sample type will be referenced using an identifier as outlined below:

                - MW – monitoring well (groundwater)

                - SB – soil boring (soil)

                - SS – surface soil (soil)

        Example:

        Monitoring Well: MW-RI-1

        Soil Boring: SB-RI-1

    - Analytical Samples: Landside Investigation

        | Sample Media | - | Core/Unique ID | (For Soil Samples Only: Depth or Depth Interval) |

    - Sample Locations: Waterside Investigation

        | Sample Type | Transect Number | Unique ID |

- The sample type will be referenced using an identifier as outlined below:
    - T – creek transect location (soil/sediment)

Example:

Sediment Transect – T1A

- Analytical Samples: Waterside Investigation

| Sample Type | Transect Number | Unique ID | (Depth or Depth Interval)

- QA/QC Samples: Landside and Waterside Investigations

Field Duplicate

| DUP | - | Unique Number |

Example :

Parent Sample : SB-RI-1(0-5)

Field Duplicate: DUP-01

Field duplicates will be submitted as blind samples and identified using "DUP" + unique number. Numbering is consecutive for each type of sample media. Numbers will be assigned to each field team when multiple teams are working simultaneously. The linkage to the source analytical sample is recorded in the field notes.

Equipment Blanks

| EB | - | Collection Date - | Unique Number |

Example:

EB-03152025-01

Equipment blank numbering is consecutive within each type of sample media. Numbers will be assigned to each field team when multiple teams are working simultaneously.

Matrix Spike/Matrix Spike Duplicate

| Analytical Sample ID | | MS or MSD |

Example:

Parent Sample: SB-RI-1(0-5)

MS: SB-RI-1(0-5)MS

MSD: SB-RI-1(0-5)MSD

For MS and MSD samples, the suffix "MS" or "MSD" will be added to the extra sample containers based on the parent sample ID.

- **Field Documentation** – During field investigation activities, documentation and records will be prepared and maintained to cover the various tasks required to support core advancement, core processing, sampling, and submittal. The documentation generated will allow for tracking of field activities and core/sample handling by the field team. Originally generated documents and records will be retained in the designated centralized project location (i.e., electronic, or physical location).

- **Field Reporting –** Collected field data, including observations, measurements, calibrations, etc., will be recorded in project-specific field notebooks. Hard copies of boring/core logs will be reviewed by the appropriate field or data manager for consistency and adherence to the task work plan and QA/QC procedures. Corrections or improvements will be discussed with the project manager and field team members, corrected where necessary, then processed.

- **Field Records –** Field records such as logbooks and instrument logs are considered part of the project's permanent records. Logbook entry errors will be crossed out in a single line stroke, initialed, and dated. Errors will not be corrected using white-out or overwriting. Unused portions of pages will be completely lined out, initialed, and dated.

    - The field logbooks will contain the following as a minimum:

        - Date/time/weather.
        - GEI personnel.
        - Purpose of visit/daily objectives.
        - People (client, contractor, landowners, etc.) present upon GEI arrival.
        - Site number and name.
        - Locations and descriptions of sampling points.
        - Sample identification number and date and time of sample collection.
        - Type of sample.
        - Field equipment calibration information.
        - Any field measurements taken.
        - Use of field data sheets or electronic logging equipment (e.g., boring logs, monitoring well sampling logs, etc.).
        - References for maps and photographs of the sample site.

- Recommendations made to the client representative and GEI Project Manager.
- Summary of work completed/work remaining.
- Any procedural steps taken that deviate from those presented in this QAPP.

Laboratory Documentation and Records

The analytical laboratory will be responsible for maintaining analytical logbooks and laboratory data. Raw laboratory data files and electronic and hard copy data will be inventoried and maintained by the laboratory for a minimum of 10 years. Laboratory analytical data packages will contain the following information at a minimum: case narrative, completed chain-of-custody forms, calibration summary and raw data, mass spectrometer tuning data (as applicable), gas chromatogram (as applicable), QC summary forms and raw data, blank results, and method and instrument DLs. The laboratory will provide an electronic copy of the data, which will follow EPA Region 5 EDD format.

- **Laboratory Project Files –** Each laboratory conducting project work will establish a centralized filing system for storage of digital and hard copy documentation. The laboratory will retain project files for a minimum of 10 years.
    - Documents and records include data such as chain-of-custody forms, correspondence, data packages, lab technician workbooks, bench sheets, and instrument logbooks that include calibration logs, instrumentation printouts, and sample tracking. Data records such as logbooks and instrument logs are considered part of the laboratory permanent record, and each page or entry should be initialed at the time of entry by the analyst. Entry errors will be crossed out in a single stroke, initialed, and dated. Each analyst will provide dated notes regarding the sample, performed analytical procedures, and testing results on laboratory forms or personal notebook pages. These logs and notebooks will be periodically reviewed by the appropriate laboratory leader for accuracy, completeness, and conformity to this QAPP. Entries, calculations, and results will be verified by the appropriate laboratory group leader. Once verified, the laboratory group leader will initial and date the pages or corrective action that will be taken to rectify the errors.
- **Laboratory Data Reporting –** Data reports for project parameters will at a minimum include the following:
    - Narrative – sample information included during analysis are as follows:
        - Laboratory name and address.
        - Sample delivery group.

- Sample receipt date.
- Cross reference of laboratory identification number to contractor sample identification.
- Analytical methods.
- Deviations from specific protocol.
- Corrective actions taken.
- Sample handling documents (field and/or internal chain-of-custodies and sample condition receipt forms).

- Analytical results – soil and sediment data will be reported on a dry-weight basis. Results will be reported uncorrected for blank contamination. Analytical results will be reported based on analysis type/method and include the following:

  - Sample ID.
  - Laboratory ID.
  - Collection date.
  - Sample receipt date.
  - Sample extraction date.
  - Sample analysis date.
  - Detection limits (reporting and method).
  - Associated methods and sample QC results.
  - Dilution factor and associated analyte-specific results.
    - The laboratory is not required to run samples undiluted if there is a risk of instrument damage. As site contaminant concentrations are often unknown, laboratories should screen samples and submit results of the screening in the laboratory data package. Laboratories should document the decision to not run the samples undiluted in the laboratory case narrative, and provide supporting information (e.g., screening chromatograms, pictures, description of the samples).
  - Re-extractions and re-analyses with a single reportable result clearly identified.
  - Data report levels.
    - Level 1 – minimum reporting: "results only" reporting for analyses performed. To be used either by their nature (field monitoring) or intended data use (i.e., preliminary screening), and therefore, extensive supporting documentation is not required. Geotechnical analytical data used for remedy design also is reported at Level 1.
    - Level 4 – Full reporting: full Contract Laboratory Program (CLP) reporting for analyses performed requiring full documentation. Chemical analytical data is reported at Level 4.

- Data associated with CLP-equivalent reporting will also include necessary documentation to meet a CLP-equivalent package. The additional documentation will include the following:

    - Raw data to recalculate results.
    - Instrument printouts.
    - Quantitation reports.
    - Calibration standards.
    - Analytical results calculations.
    - Sample extraction.
    - Digestion.
    - Other preparation logs.
    - Standard preparation logs.
    - Instrument run logs.
    - Moisture content calculations.
    - Analytical runs (i.e., dilutions) included in the raw data of the data package.
    - When manual peak integrations are being performed, include blown up versions of before and after chromatograms to demonstrate adequate peak resolution.

- The laboratory must report the results of all analytical runs, including all dilutions, re-extractions, and re-analyses. Laboratories should use best professional judgement to identify the Reportable Results for each specific analyte and must designate which results is reportable in the electronic data deliverable (EDD). This is typically achieved by including a "Reportable" field with a Y/N valid value.

- Laboratory data deliverables should be provided in accordance with this QAPP, including a portable document format (PDF) of the complete supplemental laboratory data package (i.e., raw data).

Data Submittals

- Spatial data will be submitted in the ESRI File Geodatabase format with a defined coordinate system. If applicable, submissions should include the collection method(s). Each geodatabase should be accompanied by Federal Geographic Data Committee (FGDC) compliant metadata.

- The analytical laboratories will submit electronic data deliverables (EDDs) for evaluation and compliance. The EDDs will be self-checked by the laboratories using their self-inspection tool prior to submitting to GEI. GEI will then convert the lab EDDs into an EPA Region 5 EDD format and will conduct data validation.

- A Data Usability Memorandum will be submitted by GEI to the EPA in PDF format following the completion of data validation. This submittal will include an overall summary report on the usability of the data, individual validation narrative/checklists for each individual data package, and marked-up data forms showing the data validation flags and the GLNPO checklists. In addition, analytical report narratives and data validation reports will be included in the RI Data Summary Report. A Final Data Package will be submitted following the completion of the Final Data Usability Memorandum. The Final Data Package includes the Region 5 EDD (Chemical analysis), supplemental attachments, data validation reports and narratives, data qualifier definitions, location data checklist, core logs, and additional reports that will be uploaded to GLSED. GEI will verify that the Final Data Package deliverables, including the EDDs containing toxicity and bioaccumulation data, are compatible with the most recent version of the GLLA Data Reporting Standard.

    - Additionally, the data review should include validator qualifiers, final interpreted data qualifiers, qualifier definitions, and information on summation schemes or correction factors, including how non-detects were accounted for.

- GEI will prepare an RI Data Summary Report that provides the findings of the investigation, including but not limited to, data tables and maps summarizing results and analysis. Additionally, GEI will also identify deviations and step-out locations not sampled.

    - This deliverable will be submitted in PDF describing the number, location, results, data analysis, and recommendation from the assessment. It will also document sampling changes made in the field along with any issues, concerns, or problem encountered during data collection and analysis.

Project Data Assessment Records

- Records of project assessments will be maintained by the GEI Project Manager and GEI Project QAM, including field technical systems audit checklists, field performance audit checklists, laboratory performance audit checklists, data validation reports, internal reviewer sign-off, phone conversation records, and corrective action reports.

Assessment/Audit Tasks

Field and laboratory audits of the performance and systems will be completed during investigation activities as described in the following paragraphs and Worksheets #31 and #32.

- **Field Audits –** The following performance and system audits will be performed throughout the project:

- The appropriate field team lead will be responsible for monitoring field performance. Field audits will be performed by the GEI project director, GEI project manager, or their designee. Field audits will be documented in field performance audit summaries that provide field activity evaluations based on their established work plans and procedures. The frequency of the audits will be determined by the project manager and field team lead and may fluctuate throughout the duration of the project. Observations noted during the performance audit that either deviate from established project protocols or processes identified as improvements to established project protocols will be recorded and documented.

- The data manager or their designee will perform period review of equipment and trip blank results to identify if field decontamination and sampling procedures are being executed effectively. In addition, review of the QA/QC sampling activities will be reviewed for accuracy and consistency with this QAPP. Deficiencies will be discussed with the project manager and addressed for compliance with this QAPP and associated work plans.

- **Laboratory Audits –** The laboratory QA manager will be responsible for performing periodic internal laboratory audits to assess the overall performance of the laboratory staff to the performance criteria outlined in the laboratory plans and SOPs. Audit results will be summarized and provided to each laboratory department manager, the laboratory manager, and laboratory director. In addition, the data QA manager will also conduct periodic system audits of each laboratory as needed to ensure that the laboratory complies with the procedures detailed in the QA management plans and SOPs.

    - Audits will be performed in accordance with state and federal certification programs by the regulatory agency issuing the laboratory certifications. These audits are typically performed annually and focus on performance to the specific program procedures and methodology for which the laboratory is seeking certification. During the audit, documentation related to sample handling and tracking, analytical methodologies, analytical supportive summaries, and final reports are reviewed. Audit findings are formally documented and outline necessary corrective action to the laboratory.

- **Corrective Action –** If during field of laboratory audits, corrective actions are implemented for activities not in compliance with objectives and procedures outline in this QAPP, the corrective actions will include immediate investigation/evaluation, documentation, and corrections to the appropriate procedures as detailed below.

    - **Field Procedures –** If the field crew or audit findings identify a condition which has the potential to cause adverse effects on data quality, this condition will be immediately brought to the attention of the program director or designee. A

corrective action form will be prepared by the program director or designee who will document the condition, the root cause, appropriate information points, and the corrective action that should be implemented. The field team lead will immediately report the identified condition, review status, and corrective action to the program director, project manager, and QA data manager. Examples of conditions which may require corrective actions include:

- Procedures and methodologies are not being executed by the field team in accordance with this QAPP and appropriate work plans.
- Field conditions change or were different than anticipated, requiring modification of field procedures.
- Equipment is dysfunctional or inadequate for the task or is not properly calibrated.

- **Laboratory Procedures –** If a condition in the laboratory is identified by laboratory staff or during audits that is or has the potential to cause an adverse effect on data quality, the laboratory project manager will be notified immediately. The condition will be reviewed and documented by the laboratory project manager or designee. The documentation will include the identified condition, cause, corrective action taken, and additional pertinent information. Conditions and corrective action will be reported to GEI project manager and data quality manager. Examples of conditions that may require corrective actions include:

  - Procedures and methodologies are not being executed in accordance with this QAPP, SOPs, and appropriate work plans.
  - Pre-determined data acceptance and usability standards are not obtained.
  - Equipment is dysfunctional or not calibrated properly.
  - Analytical results are not completely traceable, or QC requirements are not being met.

Data Review Tasks

See Worksheets #36 and #37.

Worksheet #15-1: Reference Limits and Evaluation Table – VOCs in Soil

Matrix: Soil
Analytical Method: 8260D/5035 – VOCs

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Methylene chloride	75-09-2	50	Unrestricted SCO	5	2.29	5
1,1-Dichloroethane	75-34-3	270	Unrestricted SCO	1	0.145	1
Chloroform	67-66-3	370	Unrestricted SCO	1.5	0.14	1.5
Carbon tetrachloride	56-23-5	760	Unrestricted SCO	1	0.23	1
1,2-Dichloropropane	78-87-5	700000	NY CP-51 Restricted Use Eco SSCO	1	0.125	1
Dibromochloromethane	124-48-1	10000	NY CP-51 Restricted Use Eco SSCO	1	0.14	1
1,1,2-Trichloroethane	79-00-5	NE	N/A	1	0.267	1
Tetrachloroethene	127-18-4	1300	Unrestricted SCO	0.5	0.196	0.5
Chlorobenzene	108-90-7	1100	Unrestricted SCO	0.5	0.127	0.5
Trichlorofluoromethane	75-69-4	NE	N/A	4	0.695	4
1,2-Dichloroethane	107-06-2	20	Unrestricted SCO	1	0.257	1
1,1,1-Trichloroethane	71-55-6	680	Unrestricted SCO	0.5	0.167	0.5

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Bromodichloromethane	75-27-4	NE	N/A	0.5	0.109	0.5
trans-1,3-Dichloropropene	10061-02-6	NE	N/A	1	0.273	1
cis-1,3-Dichloropropene	10061-01-5	NE	N/A	0.5	0.158	0.5
1,3-Dichloropropene, Total	542-75-6	NE	N/A	0.5	0.158	0.5
Bromoform	75-25-2	NE	N/A	4	0.246	4
1,1,2,2-Tetrachloroethane	79-34-5	NE	N/A	0.5	0.166	0.5
Benzene	71-43-2	60	Unrestricted SCO	0.5	0.166	0.5
Toluene	108-88-3	700	Unrestricted SCO	1	0.543	1
Ethylbenzene	100-41-4	1000	Unrestricted SCO	1	0.141	1
Chloromethane	74-87-3	NE	N/A	4	0.932	4
Bromomethane	74-83-9	NE	N/A	2	0.581	2
Vinyl chloride	75-01-4	20	Unrestricted SCO	1	0.335	1
Chloroethane	75-00-3	NE	N/A	2	0.452	2
1,1-Dichloroethene	75-35-4	330	Unrestricted SCO	1	0.238	1
trans-1,2-Dichloroethene	156-60-5	190	Unrestricted SCO	1.5	0.137	1.5
Trichloroethene	79-01-6	470	Unrestricted SCO	0.5	0.137	0.5

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
1,2-Dichlorobenzene	95-50-1	1100	Unrestricted SCO	2	0.144	2
1,3-Dichlorobenzene	541-73-1	2400	Unrestricted SCO	2	0.148	2
1,4-Dichlorobenzene	106-46-7	1800	Unrestricted SCO	2	0.171	2
Methyl tert butyl ether	1634-04-4	930	Unrestricted SCO	2	0.201	2
p/m-Xylene	179601-23-1	260	Unrestricted SCO	2	0.56	2
o-Xylene	95-47-6	260	Unrestricted SCO	1	0.291	1
Xylene (Total)	1330-20-7	NE	N/A	1	0.291	1
cis-1,2-Dichloroethene	156-59-2	250	Unrestricted SCO	1	0.175	1
1,2-Dichloroethene (total)	540-59-0	NE	N/A	1	0.137	1
Styrene	100-42-5	300000	NY CP-15 Restricted Use Eco SSCO	1	0.196	1
Dichlorodifluoromethane	75-71-8	NE	N/A	10	0.915	10
Acetone	67-64-1	50	Unrestricted SCO	10	4.811	10
Carbon disulfide	75-15-0	NE	N/A	10	4.55	10
2-Butanone	78-93-3	120	Unrestricted SCO	10	2.22	10
4-Methyl-2-pentanone	108-10-1	NE	N/A	10	1.28	10
2-Hexanone	591-78-6	NE	N/A	10	1.18	10

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Bromochloromethane	74-97-5	NE	N/A	2	0.205	2
1,2-Dibromoethane	106-93-4	NE	N/A	1	0.279	1
n-Butylbenzene	104-51-8	NE	N/A	1	0.167	1
sec-Butylbenzene	135-98-8	NE	N/A	1	0.146	1
tert-Butylbenzene	98-06-6	NE	N/A	2	0.118	2
1,2-Dibromo-3-chloropropane	96-12-8	NE	N/A	3	0.998	3
Isopropylbenzene	98-82-8	NE	N/A	1	0.109	1
p-Isopropyltoluene	99-87-6	NE	N/A	1	0.109	1
n-Propylbenzene	103-65-1	3900	Unrestricted SCO	1	0.171	1
1,2,3-Trichlorobenzene	87-61-6	20000	NY CP-15 Restricted Use Eco SSCO	2	0.322	2
1,2,4-Trichlorobenzene	120-82-1	20000	NY CP-15 Restricted Use Eco SSCO	2	0.272	2
1,3,5-Trimethylbenzene	108-67-8	8400	Unrestricted SCO	2	0.193	2
1,2,4-Trimethylbenzene	95-63-6	NE	N/A	2	0.334	2

Notes:

[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).

[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources. PAL is based on the lower regulatory criteria.

The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.

The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.

MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-2: Reference Limits and Evaluation Table – VOCs in Sediment

Matrix: Sediment
Analytical Method: 8260D/5035 – VOCs

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Methylene chloride	75-09-2	50	Unrestricted SCO	5	2.29	5
1,1-Dichloroethane	75-34-3	270	Unrestricted SCO	1	0.145	1
Chloroform	67-66-3	370	Unrestricted SCO	1.5	0.14	1.5
Carbon tetrachloride	56-23-5	760	Unrestricted SCO	1	0.23	1
1,2-Dichloropropane	78-87-5	700000	NY CP-51 Restricted Use Eco SSCO	1	0.125	1
Dibromochloromethane	124-48-1	10000	NY CP-51 Restricted Use Eco SSCO	1	0.14	1
1,1,2-Trichloroethane	79-00-5	3500	Freshwater Sediment Guidance Values Class B	1	0.267	1
Tetrachloroethene	127-18-4	1300	Unrestricted SCO	0.5	0.196	0.5
Chlorobenzene	108-90-7	1100	Unrestricted SCO	0.5	0.127	0.5
Trichlorofluoromethane	75-69-4	NE	N/A	4	0.695	4
1,2-Dichloroethane	107-06-2	20	Unrestricted SCO	1	0.257	1

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
1,1,1-Trichloroethane	71-55-6	680	Unrestricted SCO	0.5	0.167	0.5
Bromodichloromethane	75-27-4	NE	N/A	0.5	0.109	0.5
trans-1,3-Dichloropropene	10061-02-6	NE	N/A	1	0.273	1
cis-1,3-Dichloropropene	10061-01-5	NE	N/A	0.5	0.158	0.5
1,3-Dichloropropene, Total	542-75-6	NE	N/A	0.5	0.158	0.5
Bromoform	75-25-2	NE	N/A	4	0.246	4
1,1,2,2-Tetrachloroethane	79-34-5	5400	Freshwater Sediment Guidance Values Class B	0.5	0.166	0.5
Benzene	71-43-2	60	Unrestricted SCO	0.5	0.166	0.5
Toluene	108-88-3	700	Unrestricted SCO	1	0.543	1
Ethylbenzene	100-41-4	1000	Unrestricted SCO	1	0.141	1
Chloromethane	74-87-3	NE	N/A	4	0.932	4
Bromomethane	74-83-9	NE	N/A	2	0.581	2
Vinyl chloride	75-01-4	20	Unrestricted SCO	1	0.335	1
Chloroethane	75-00-3	NE	N/A	2	0.452	2
1,1-Dichloroethene	75-35-4	330	Unrestricted SCO	1	0.238	1
trans-1,2-Dichloroethene	156-60-5	190	Unrestricted SCO	1.5	0.137	1.5

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Trichloroethene	79-01-6	470	Unrestricted SCO	0.5	0.137	0.5
1,2-Dichlorobenzene	95-50-1	1100	Unrestricted SCO	2	0.144	2
1,3-Dichlorobenzene	541-73-1	2400	Unrestricted SCO	2	0.148	2
1,4-Dichlorobenzene	106-46-7	1800	Unrestricted SCO	2	0.171	2
Methyl tert butyl ether	1634-04-4	930	Unrestricted SCO	2	0.201	2
p/m-Xylene	179601-23-1	260	Unrestricted SCO	2	0.56	2
o-Xylene	95-47-6	260	Unrestricted SCO	1	0.291	1
Xylene (Total)	1330-20-7	NE	N/A	1	0.291	1
cis-1,2-Dichloroethene	156-59-2	250	Unrestricted SCO	1	0.175	1
1,2-Dichloroethene (total)	540-59-0	NE	N/A	1	0.137	1
Styrene	100-42-5	300000	NY CP-15 Restricted Use Eco SSCO	1	0.196	1
Dichlorodifluoromethane	75-71-8	NE	N/A	10	0.915	10
Acetone	67-64-1	50	Unrestricted SCO	10	4.811	10
Carbon disulfide	75-15-0	NE	N/A	10	4.55	10
2-Butanone	78-93-3	120	Unrestricted SCO	10	2.22	10
4-Methyl-2-pentanone	108-10-1	NE	N/A	10	1.28	10

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
2-Hexanone	591-78-6	NE	N/A	10	1.18	10
Bromochloromethane	74-97-5	NE	N/A	2	0.205	2
1,2-Dibromoethane	106-93-4	NE	N/A	1	0.279	1
n-Butylbenzene	104-51-8	12000	Unrestricted SCO	1	0.167	1
sec-Butylbenzene	135-98-8	11000	Unrestricted SCO	1	0.146	1
tert-Butylbenzene	98-06-6	5900	Unrestricted SCO	2	0.118	2
1,2-Dibromo-3-chloropropane	96-12-8	NE	N/A	3	0.998	3
Isopropylbenzene	98-82-8	1800	Freshwater Sediment Guidance Values Class B	1	0.109	1
p-Isopropyltoluene	99-87-6	NE	N/A	1	0.109	1
n-Propylbenzene	103-65-1	3900	Unrestricted Use	1	0.171	1
1,2,3-Trichlorobenzene	87-61-6	2800	Freshwater Sediment Guidance Values Class B	2	0.322	2
1,2,4-Trichlorobenzene	120-82-1	20000	Freshwater Sediment Guidance Values Class B	2	0.272	2
1,3,5-Trimethylbenzene	108-67-8	8400	Unrestricted SCO	2	0.193	2
1,2,4-Trimethylbenzene	95-63-6	30000	Freshwater Sediment Guidance Values Class B	2	0.334	2

Notes:

[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).

[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.

The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.

The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.

MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-3: Reference Limits and Evaluation Table – SVOCs in Soil

Matrix: Soil
Analytical Method: 8270E – SVOCs

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Acenaphthene	83-32-9	20000	Unrestricted SCO	133.6	17.3012	133.6
1,2,4-Trichlorobenzene	120-82-1	20000	Unrestricted SCO	167	19.1048	167
Hexachlorobenzene	118-74-1	330	Unrestricted SCO	100.2	18.704	100.2
Bis(2-chloroethyl)ether	111-44-4	NE	N/A	150.3	22.6452	150.3
2-Chloronaphthalene	91-58-7	NE	N/A	167	16.5664	167
1,2-Dichlorobenzene	95-50-1	1100	Unrestricted SCO	167	29.9932	167
1,3-Dichlorobenzene	541-73-1	2400	Unrestricted SCO	167	28.724	167
1,4-Dichlorobenzene	106-46-7	1800	Unrestricted SCO	167	29.1582	167
3,3'-Dichlorobenzidine	91-94-1	NE	N/A	167	44.422	167
2,4-Dinitrotoluene	121-14-2	NE	N/A	167	33.4	167
2,6-Dinitrotoluene	606-20-2	NE	N/A	167	28.6572	167
Fluoranthene	206-44-0	100000	Unrestricted SCO	100.2	19.1716	100.2
4-Chlorophenyl phenyl ether	7005-72-3	NE	N/A	167	17.869	167

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
4-Bromophenyl phenyl ether	101-55-3	NE	N/A	167	25.4842	167
Bis(2-chloroisopropyl)ether	108-60-1	NE	N/A	200.4	28.5236	200.4
Bis(2-chloroethoxy)methane	111-91-1	NE	N/A	180.36	16.7334	180.36
Hexachlorobutadiene	87-68-3	NE	N/A	167	24.4488	167
Hexachlorocyclopentadiene	77-47-4	10000	NY CP-51 Restricted Use Eco SSCO	477.62	151.302	477.62
Hexachloroethane	67-72-1	NE	N/A	133.6	27.0206	133.6
Isophorone	78-59-1	NE	N/A	150.3	21.6766	150.3
Naphthalene	91-20-3	12000	Unrestricted SCO	167	20.3406	167
Nitrobenzene	98-95-3	40000	NY CP-51 Restricted Use Eco SSCO	150.3	24.716	150.3
NitrosoDiPhenylAmine(NDPA)/DPA	86-30-6	20000	NY CP-51 Restricted Use Eco SSCO	133.6	19.0046	133.6
n-Nitrosodi-n-propylamine	621-64-7	NE	N/A	167	25.7848	167
Bis(2-Ethylhexyl)phthalate	117-81-7	239000	NY CP-51 Restricted Use Eco SSCO	167	57.782	167
Butyl benzyl phthalate	85-68-7	NE	N/A	167	42.084	167
Di-n-butylphthalate	84-74-2	14	NY CP-51 Restricted Use Eco SSCO	167	31.6632	167

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Di-n-octylphthalate	117-84-0	NE	N/A	167	56.78	167
Diethyl phthalate	84-66-2	100000	NY CP-51 Restricted Use Eco SSCO	167	15.4642	167
Dimethyl phthalate	131-11-3	200000	NY CP-51 Restricted Use Eco SSCO	167	35.07	167
Benzo(a)anthracene	56-55-3	1000	Unrestricted SCO	100.2	18.8042	100.2
Benzo(a)pyrene	50-32-8	1000	Unrestricted SCO	133.6	40.748	133.6
Benzo(b)fluoranthene	205-99-2	1000	Unrestricted SCO	100.2	28.1228	100.2
Benzo(k)fluoranthene	207-08-9	800	Unrestricted SCO	100.2	26.72	100.2
Chrysene	218-01-9	1000	Unrestricted SCO	100.2	17.368	100.2
Acenaphthylene	208-96-8	100000	Unrestricted SCO	133.6	25.7848	133.6
Anthracene	120-12-7	100000	Unrestricted SCO	100.2	32.565	100.2
Benzo(ghi)perylene	191-24-2	100000	Unrestricted SCO	133.6	19.6392	133.6
Fluorene	86-73-7	30000	Unrestricted SCO	167	16.2324	167
Phenanthrene	85-01-8	100000	Unrestricted SCO	100.2	20.3072	100.2
Dibenzo(a,h)anthracene	53-70-3	330	Unrestricted SCO	100.2	19.3052	100.2
Indeno(1,2,3-cd)Pyrene	193-39-5	500	Unrestricted SCO	133.6	23.2798	133.6
Pyrene	129-00-0	100000	Unrestricted SCO	100.2	16.5998	100.2

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Biphenyl	92-52-4	60000	Unrestricted SCO	380.76	21.71	380.76
4-Chloroaniline	106-47-8	NE	N/A	167	30.394	167
2-Nitroaniline	88-74-4	NE	N/A	167	32.1976	167
3-Nitroaniline	99-09-2	NE	N/A	167	31.4962	167
4-Nitroaniline	100-01-6	NE	N/A	167	69.138	167
Dibenzofuran	132-64-9	7000	Unrestricted SCO	167	15.7982	167
2-Methylnaphthalene	91-57-6	NE	N/A	200.4	20.1736	200.4
Acetophenone	98-86-2	NE	N/A	167	20.6746	167
2,4,6-Trichlorophenol	88-06-2	NE	N/A	100.2	31.6632	100.2
P-Chloro-M-Cresol	59-50-7	NE	N/A	167	24.883	167
2-Chlorophenol	95-57-8	800	NY CP-51 Restricted Use Eco SSCO	167	19.7394	167
2,4-Dichlorophenol	120-83-2	20000	NY CP-51 Restricted Use Eco SSCO	150.3	26.8536	150.3
2,4-Dimethylphenol	105-67-9	NE	N/A	167	55.11	167
2-Nitrophenol	88-75-5	7000	NY CP-51 Restricted Use Eco SSCO	360.72	62.792	360.72
4-Nitrophenol	100-02-7	7000	NY CP-51 Restricted Use Eco SSCO	233.8	68.136	233.8

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
2,4-Dinitrophenol	51-28-5	20000	NY CP-51 Restricted Use Eco SSCO	801.6	77.822	801.6
4,6-Dinitro-o-cresol	534-52-1	NE	N/A	434.2	80.16	434.2
Pentachlorophenol	87-86-5	800	Unrestricted SCO	133.6	36.74	133.6
Phenol	108-95-2	330	Unrestricted SCO	167	25.217	167
2-Methylphenol	95-48-7	330	Unrestricted SCO	167	25.885	167
3-Methylphenol/4-Methylphenol	106-44-5	NE	N/A	240.48	26.1522	240.48
2,4,5-Trichlorophenol	95-95-4	4000	NY CP-51 Restricted Use Eco SSCO	167	31.9972	167
Benzoic Acid	65-85-0	NE	N/A	541.08	169.004	541.08
Benzyl Alcohol	100-51-6	NE	N/A	167	51.102	167
Carbazole	86-74-8	NE	N/A	167	16.2324	167
1,4-Dioxane	123-91-1	100	Unrestricted SCO	25.05	7.682	25.05

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-4: Reference Limits and Evaluation Table – SVOCs in Sediment

Matrix: Sediment
Analytical Method: 8270E – SVOCs

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Acenaphthene	83-32-9	20000	Unrestricted SCO	133.6	17.3012	133.6
1,2,4-Trichlorobenzene	120-82-1	20000	Unrestricted SCO	167	19.1048	167
Hexachlorobenzene	118-74-1	330	Unrestricted SCO	100.2	18.704	100.2
Bis(2-chloroethyl)ether	111-44-4	NE	N/A	150.3	22.6452	150.3
2-Chloronaphthalene	91-58-7	NE	N/A	167	16.5664	167
1,2-Dichlorobenzene	95-50-1	1100	Unrestricted SCO	167	29.9932	167
1,3-Dichlorobenzene	541-73-1	2400	Unrestricted SCO	167	28.724	167
1,4-Dichlorobenzene	106-46-7	1800	Unrestricted SCO	167	29.1582	167
3,3'-Dichlorobenzidine	91-94-1	NE	N/A	167	44.422	167
2,4-Dinitrotoluene	121-14-2	NE	N/A	167	33.4	167
2,6-Dinitrotoluene	606-20-2	NE	N/A	167	28.6572	167
Fluoranthene	206-44-0	100000	Unrestricted SCO	100.2	19.1716	100.2

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
4-Chlorophenyl phenyl ether	7005-72-3	NE	N/A	167	17.869	167
4-Bromophenyl phenyl ether	101-55-3	NE	N/A	167	25.4842	167
Bis(2-chloroisopropyl)ether	108-60-1	NE	N/A	200.4	28.5236	200.4
Bis(2-chloroethoxy)methane	111-91-1	NE	N/A	180.36	16.7334	180.36
Hexachlorobutadiene	87-68-3	12000	Freshwater Sediment Guidance Values Class B	167	24.4488	167
Hexachlorocyclopentadiene	77-47-4	8100	Freshwater Sediment Guidance Values Class B	477.62	151.302	477.62
Hexachloroethane	67-72-1	NE	N/A	133.6	27.0206	133.6
Isophorone	78-59-1	NE	N/A	150.3	21.6766	150.3
Naphthalene	91-20-3	12000	Unrestricted SCO	167	20.3406	167
Nitrobenzene	98-95-3	40000	NY CP-51 Restricted Use Eco SSCO	150.3	24.716	150.3
NitrosoDiPhenylAmine(NDPA)/DPA	86-30-6	20000	NY CP-51 Restricted Use Eco SSCO	133.6	19.0046	133.6
n-Nitrosodi-n-propylamine	621-64-7	NE	N/A	167	25.7848	167
Bis(2-Ethylhexyl)phthalate	117-81-7	14	Freshwater Sediment Guidance Values Class B	167	57.782	167
Butyl benzyl phthalate	85-68-7	NE	N/A	167	42.084	167

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Di-n-butylphthalate	84-74-2	14	NY CP-51 Restricted Use Eco SSCO	167	31.6632	167
Di-n-octylphthalate	117-84-0	NE	N/A	167	56.78	167
Diethyl phthalate	84-66-2	100000	NY CP-51 Restricted Use Eco SSCO	167	15.4642	167
Dimethyl phthalate	131-11-3	200000	NY CP-51 Restricted Use Eco SSCO	167	35.07	167
Benzo(a)anthracene	56-55-3	1000	Unrestricted SCO	100.2	18.8042	100.2
Benzo(a)pyrene	50-32-8	1000	Unrestricted SCO	133.6	40.748	133.6
Benzo(b)fluoranthene	205-99-2	1000	Unrestricted SCO	100.2	28.1228	100.2
Benzo(k)fluoranthene	207-08-9	800	Unrestricted SCO	100.2	26.72	100.2
Chrysene	218-01-9	1000	Unrestricted SCO	100.2	17.368	100.2
Acenaphthylene	208-96-8	100000	Unrestricted SCO	133.6	25.7848	133.6
Anthracene	120-12-7	100000	Unrestricted SCO	100.2	32.565	100.2
Benzo(ghi)perylene	191-24-2	100000	Unrestricted SCO	133.6	19.6392	133.6
Fluorene	86-73-7	30000	Unrestricted SCO	167	16.2324	167
Phenanthrene	85-01-8	100000	Unrestricted SCO	100.2	20.3072	100.2
Dibenzo(a,h)anthracene	53-70-3	330	Unrestricted SCO	100.2	19.3052	100.2

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Indeno(1,2,3-cd)Pyrene	193-39-5	500	Unrestricted SCO	133.6	23.2798	133.6
Pyrene	129-00-0	100000	Unrestricted SCO	100.2	16.5998	100.2
Biphenyl	92-52-4	60000	Unrestricted SCO	380.76	21.71	380.76
4-Chloroaniline	106-47-8	NE	N/A	167	30.394	167
2-Nitroaniline	88-74-4	NE	N/A	167	32.1976	167
3-Nitroaniline	99-09-2	NE	N/A	167	31.4962	167
4-Nitroaniline	100-01-6	NE	N/A	167	69.138	167
Dibenzofuran	132-64-9	7000	Unrestricted SCO	167	15.7982	167
2-Methylnaphthalene	91-57-6	NE	N/A	200.4	20.1736	200.4
Acetophenone	98-86-2	NE	N/A	167	20.6746	167
2,4,6-Trichlorophenol	88-06-2	NE	N/A	100.2	31.6632	100.2
P-Chloro-M-Cresol	59-50-7	NE	N/A	167	24.883	167
2-Chlorophenol	95-57-8	800	NY CP-51 Restricted Use Eco SSCO	167	19.7394	167
2,4-Dichlorophenol	120-83-2	20000	NY CP-51 Restricted Use Eco SSCO	150.3	26.8536	150.3
2,4-Dimethylphenol	105-67-9	NE	N/A	167	55.11	167

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
2-Nitrophenol	88-75-5	7000	NY CP-51 Restricted Use Eco SSCO	360.72	62.792	360.72
4-Nitrophenol	100-02-7	7000	NY CP-51 Restricted Use Eco SSCO	233.8	68.136	233.8
2,4-Dinitrophenol	51-28-5	20000	NY CP-51 Restricted Use Eco SSCO	801.6	77.822	801.6
4,6-Dinitro-o-cresol	534-52-1	NE	N/A	434.2	80.16	434.2
Pentachlorophenol	87-86-5	800	Unrestricted SCO	133.6	36.74	133.6
Phenol	108-95-2	330	Unrestricted SCO	167	25.217	167
2-Methylphenol	95-48-7	330	Unrestricted SCO	167	25.885	167
3-Methylphenol/4-Methylphenol	106-44-5	NE	N/A	240.48	26.1522	240.48
2,4,5-Trichlorophenol	95-95-4	4000	NY CP-51 Restricted Use Eco SSCO	167	31.9972	167
Benzoic Acid	65-85-0	NE	N/A	541.08	169.004	541.08
Benzyl Alcohol	100-51-6	NE	N/A	167	51.102	167
Carbazole	86-74-8	NE	N/A	167	16.2324	167
1,4-Dioxane	123-91-1	100	Unrestricted SCO	25.05	7.682	25.05

Notes:

[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).

[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.

The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.

The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.

MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-5: Reference Limits and Evaluation Table – PCBs in Soil

Matrix: Soil
Analytical Method: 8082A – PCB – Aroclors

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Aroclor 1016	12674-11-2	NE	Unrestricted SCO	0.05	0.00444	0.05
Aroclor 1221	11104-28-2	NE	Unrestricted SCO	0.05	0.00501	0.05
Aroclor 1232	11141-16-5	NE	Unrestricted SCO	0.05	0.0106	0.05
Aroclor 1242	53469-21-9	NE	Unrestricted SCO	0.05	0.00674	0.05
Aroclor 1248	12672-29-6	NE	Unrestricted SCO	0.05	0.0075	0.05
Aroclor 1254	11097-69-1	NE	Unrestricted SCO	0.05	0.00547	0.05
Aroclor 1260	11096-82-5	NE	Unrestricted SCO	0.05	0.00924	0.05
Aroclor 1262	37324-23-5	NE	Unrestricted SCO	0.05	0.00635	0.05
Aroclor 1268	11100-14-4	NE	Unrestricted SCO	0.05	0.00518	0.05
PCBs, Total	1336-36-3	0.1	Unrestricted SCO	0.05	0.00444	0.05

Notes:

[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).

[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources. PAL is based on the lower regulatory criteria.

The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.

The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.

MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-6: Reference Limits and Evaluation Table – PCBs in Sediment

Matrix: Sediment
Analytical Method: 8082A – PCB – Aroclors

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Aroclor 1016	12674-11-2	NE	Unrestricted SCO	0.05	0.00444	0.05
Aroclor 1221	11104-28-2	NE	Unrestricted SCO	0.05	0.00501	0.05
Aroclor 1232	11141-16-5	NE	Unrestricted SCO	0.05	0.0106	0.05
Aroclor 1242	53469-21-9	NE	Unrestricted SCO	0.05	0.00674	0.05
Aroclor 1248	12672-29-6	NE	Unrestricted SCO	0.05	0.0075	0.05
Aroclor 1254	11097-69-1	NE	Unrestricted SCO	0.05	0.00547	0.05
Aroclor 1260	11096-82-5	NE	Unrestricted SCO	0.05	0.00924	0.05
Aroclor 1262	37324-23-5	NE	Unrestricted SCO	0.05	0.00635	0.05
Aroclor 1268	11100-14-4	NE	Unrestricted SCO	0.05	0.00518	0.05
PCBs, Total	1336-36-3	0.1	Unrestricted SCO	0.05	0.00444	0.05

Notes:

[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).

[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.

The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.

The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.

MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-7: Reference Limits and Evaluation Table – PAHs in Soil

Matrix: Soil
Analytical Method: 8270E – PAH-17

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Acenaphthene	83-32-9	20	Unrestricted SCO	0.1332	0.0172494	0.1332
Fluoranthene	206-44-0	100	Unrestricted SCO	0.0999	0.0191142	0.0999
Naphthalene	91-20-3	12	Unrestricted SCO	0.1665	0.0202797	0.1665
Benzo(a)anthracene	56-55-3	1.0	Unrestricted SCO	0.0999	0.0187479	0.0999
Benzo(a)pyrene	50-32-8	1.0	Unrestricted SCO	0.1332	0.040626	0.1332
Benzo(b)fluoranthene	205-99-2	1.0	Unrestricted SCO	0.0999	0.0280386	0.0999
Benzo(k)fluoranthene	207-08-9	0.8	Unrestricted SCO	0.0999	0.02664	0.0999
Chrysene	218-01-9	1.0	Unrestricted SCO	0.0999	0.017316	0.0999
Acenaphthylene	208-96-8	100	Unrestricted SCO	0.1332	0.0257076	0.1332
Anthracene	120-12-7	100	Unrestricted SCO	0.0999	0.0324675	0.0999
Benzo(ghi)perylene	191-24-2	100	Unrestricted SCO	0.1332	0.0195804	0.1332
Fluorene	86-73-7	30	Unrestricted SCO	0.1665	0.0161838	0.1665
Phenanthrene	85-01-8	100	Unrestricted SCO	0.0999	0.0202464	0.0999

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Dibenzo(a,h)anthracene	53-70-3	0.33	Unrestricted SCO	0.0999	0.0192474	0.0999
Indeno(1,2,3-cd)Pyrene	193-39-5	0.5	Unrestricted SCO	0.1332	0.0232101	0.1332
Pyrene	129-00-0	100	Unrestricted SCO	0.0999	0.0165501	0.0999
2-Methylnaphthalene	91-57-6	NE	Unrestricted SCO	0.1998	0.0201132	0.1998

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-8: Reference Limits and Evaluation Table – PAHs in Sediment

Matrix: Sediment
Analytical Method: 8270E – PAH-17

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Acenaphthene	83-32-9	20	Unrestricted SCO	0.1332	0.0172494	0.1332
Fluoranthene	206-44-0	100	Unrestricted SCO	0.0999	0.0191142	0.0999
Naphthalene	91-20-3	12	Unrestricted SCO	0.1665	0.0202797	0.1665
Benzo(a)anthracene	56-55-3	1.0	Unrestricted SCO	0.0999	0.0187479	0.0999
Benzo(a)pyrene	50-32-8	1.0	Unrestricted SCO	0.1332	0.040626	0.1332
Benzo(b)fluoranthene	205-99-2	1.0	Unrestricted SCO	0.0999	0.0280386	0.0999
Benzo(k)fluoranthene	207-08-9	0.8	Unrestricted SCO	0.0999	0.02664	0.0999
Chrysene	218-01-9	1.0	Unrestricted SCO	0.0999	0.017316	0.0999
Acenaphthylene	208-96-8	100	Unrestricted SCO	0.1332	0.0257076	0.1332
Anthracene	120-12-7	100	Unrestricted SCO	0.0999	0.0324675	0.0999
Benzo(ghi)perylene	191-24-2	100	Unrestricted SCO	0.1332	0.0195804	0.1332
Fluorene	86-73-7	30	Unrestricted SCO	0.1665	0.0161838	0.1665

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Phenanthrene	85-01-8	100	Unrestricted SCO	0.0999	0.0202464	0.0999
Dibenzo(a,h)anthracene	53-70-3	0.33	Unrestricted SCO	0.0999	0.0192474	0.0999
Indeno(1,2,3-cd)Pyrene	193-39-5	0.5	Unrestricted SCO	0.1332	0.0232101	0.1332
Pyrene	129-00-0	100	Unrestricted SCO	0.0999	0.0165501	0.0999
2-Methylnaphthalene	91-57-6	NE	Unrestricted SCO	0.1998	0.0201132	0.1998

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-9: Reference Limits and Evaluation Table – TCLP VOCs in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: 8260D – TCLP VOCs

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/L)	PAL Reference[2]	Project Quantification Limit (µg/L)	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
Methylene chloride	75-09-2	NE	N/A	30	6.78	30
1,1-Dichloroethane	75-34-3	NE	N/A	7.5	2.1	7.5
Chloroform	67-66-3	6.0	TCLP	7.5	2.22	7.5
Carbon tetrachloride	56-23-5	0.5	TCLP	5	1.34	5
1,2-Dichloropropane	78-87-5	NE	N/A	17.5	1.37	17.5
Dibromochloromethane	124-48-1	NE	N/A	5	1.49	5
1,1,2-Trichloroethane	79-00-5	NE	N/A	7.5	1.44	7.5
Tetrachloroethene	127-18-4	0.5	TCLP	5	1.81	5
Chlorobenzene	108-90-7	100	TCLP	5	1.78	5
Trichlorofluoromethane	75-69-4	NE	N/A	25	1.61	25
1,2-Dichloroethane	107-06-2	0.5	TCLP	5	1.32	5
1,1,1-Trichloroethane	71-55-6	NE	N/A	5	1.58	5

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/L)	PAL Reference[2]	Project Quantification Limit (µg/L)	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
Bromodichloromethane	75-27-4	NE	N/A	5	1.92	5
trans-1,3-Dichloropropene	10061-02-6	NE	N/A	5	1.64	5
cis-1,3-Dichloropropene	10061-01-5	NE	N/A	5	1.44	5
1,1-Dichloropropene	563-58-6	NE	N/A	25	2.4	25
Bromoform	75-25-2	NE	N/A	20	2.48	20
1,1,2,2-Tetrachloroethane	79-34-5	NE	N/A	5	1.67	5
Benzene	71-43-2	0.5	TCLP	5	1.59	5
Toluene	108-88-3	NE	N/A	7.5	2.03	7.5
Ethylbenzene	100-41-4	NE	N/A	5	1.67	5
Chloromethane	74-87-3	NE	N/A	25	2	25
Bromomethane	74-83-9	NE	N/A	10	2.56	10
Vinyl chloride	75-01-4	0.2	TCLP	10	0.714	10
Chloroethane	75-00-3	NE	N/A	10	1.34	10
1,1-Dichloroethene	75-35-4	0.7	TCLP	5	1.69	5
trans-1,2-Dichloroethene	156-60-5	NE	N/A	7.5	1.63	7.5
Trichloroethene	79-01-6	0.5	TCLP	5	1.75	5

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/L)	PAL Reference[2]	Project Quantification Limit (µg/L)	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
1,2-Dichlorobenzene	95-50-1	NE	N/A	25	1.84	25
1,3-Dichlorobenzene	541-73-1	NE	N/A	25	1.86	25
1,4-Dichlorobenzene	106-46-7	NE	N/A	25	1.87	25
Methyl tert butyl ether	1634-04-4	NE	N/A	10	1.66	10
p/m-Xylene	179601-23-1	NE	N/A	10	3.32	10
o-Xylene	95-47-6	NE	N/A	10	3.92	10
cis-1,2-Dichloroethene	156-59-2	NE	N/A	5	1.87	5
Dibromomethane	74-95-3	NE	N/A	50	3.63	50
1,4-Dichlorobutane	110-56-5	NE	N/A	50	4.64	50
1,2,3-Trichloropropane	96-18-4	NE	N/A	50	1.76	50
Styrene	100-42-5	NE	N/A	10	3.59	10
Dichlorodifluoromethane	75-71-8	NE	N/A	50	2.44	50
Acetone	67-64-1	NE	N/A	50	14.6	50
Carbon disulfide	75-15-0	NE	N/A	50	2.99	50
2-Butanone	78-93-3	NE	N/A	50	19.4	50
Vinyl acetate	108-05-4	NE	N/A	50	3.11	50

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/L)	PAL Reference[2]	Project Quantification Limit (µg/L)	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
4-Methyl-2-pentanone	108-10-1	NE	N/A	50	4.16	50
2-Hexanone	591-78-6	NE	N/A	50	5.15	50
Ethyl methacrylate	97-63-2	NE	N/A	50	6.06	50
Acrylonitrile	107-13-1	NE	N/A	50	4.3	50
Bromochloromethane	74-97-5	NE	N/A	25	1.52	25
Tetrahydrofuran	109-99-9	NE	N/A	50	5.25	50
2,2-Dichloropropane	594-20-7	NE	N/A	25	2.04	25
1,2-Dibromoethane	106-93-4	NE	N/A	20	1.93	20
1,3-Dichloropropane	142-28-9	NE	N/A	25	2.12	25
1,1,1,2-Tetrachloroethane	630-20-6	NE	N/A	5	1.64	5
Bromobenzene	108-86-1	NE	N/A	25	1.52	25
n-Butylbenzene	104-51-8	NE	N/A	5	1.92	5
sec-Butylbenzene	135-98-8	NE	N/A	5	1.81	5
tert-Butylbenzene	98-06-6	NE	N/A	25	1.96	25
o-Chlorotoluene	95-49-8	NE	N/A	25	2.15	25
p-Chlorotoluene	106-43-4	NE	N/A	25	1.85	25

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/L)	PAL Reference[2]	Project Quantification Limit (µg/L)	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
1,2-Dibromo-3-chloropropane	96-12-8	NE	N/A	25	3.53	25
Hexachlorobutadiene	87-68-3	NE	N/A	5	2.17	5
Isopropylbenzene	98-82-8	NE	N/A	5	1.87	5
p-Isopropyltoluene	99-87-6	NE	N/A	5	1.88	5
Naphthalene	91-20-3	NE	N/A	25	2.16	25
n-Propylbenzene	103-65-1	NE	N/A	5	1.73	5
1,2,3-Trichlorobenzene	87-61-6	NE	N/A	25	2.34	25
1,2,4-Trichlorobenzene	120-82-1	NE	N/A	25	2.2	25
1,3,5-Trimethylbenzene	108-67-8	NE	N/A	25	2.17	25
1,2,4-Trimethylbenzene	95-63-6	NE	N/A	25	1.91	25
trans-1,4-Dichloro-2-butene	110-57-6	NE	N/A	25	2.13	25
Ethyl ether	60-29-7	NE	N/A	25	1.63	25

Notes:
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; NE = not established; N/A = not applicable; TCLP = Toxicity Characteristic Leaching Procedure.

Worksheet #15-10: Reference Limits and Evaluation Table – TPH in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: 8015D(M) – TPH-GRO/TPH-GRO

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1]	PAL Reference	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Gasoline Range Organics	None	N/A	N/A	2500	1450	2500
DRO (C10-C28)	None	N/A	N/A	33350	1840.92	33350

Notes:
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-11: Reference Limits and Evaluation Table – TCLP ABN in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: 8270E/1311 – TCLP ABN Compounds

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL) (mg/L)	PAL Reference	Project Quantification Limit (µg/L)	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
Acenaphthene	83-32-9	NE	N/A	2	0.403	2
Benzidine	92-87-5	NE	N/A	20	6.01	20
1,2,4-Trichlorobenzene	120-82-1	NE	N/A	5	0.977	5
Hexachlorobenzene	118-74-1	0.13[1]	TCLP	2	0.452	2
Bis(2-chloroethyl)ether	111-44-4	NE	N/A	2	0.392	2
2-Chloronaphthalene	91-58-7	NE	N/A	2	0.35	2
1,2-Dichlorobenzene	95-50-1	NE	N/A	2	0.329	2
1,3-Dichlorobenzene	541-73-1	NE	N/A	2	0.315	2
1,4-Dichlorobenzene	106-46-7	7.5	TCLP	2	0.391	2
3,3'-Dichlorobenzidine	91-94-1	NE	N/A	5	1.85	5
2,4-Dinitrotoluene	121-14-2	0.13[1]	TCLP	5	0.541	5
2,6-Dinitrotoluene	606-20-2	NE	N/A	5	0.845	5

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL) (mg/L)	PAL Reference	Project Quantification Limit (µg/L)	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
Azobenzene	122-66-7	NE	N/A	2	0.418	2
Fluoranthene	206-44-0	NE	N/A	2	0.411	2
4-Chlorophenyl phenyl ether	7005-72-3	NE	N/A	2	0.386	2
4-Bromophenyl phenyl ether	101-55-3	NE	N/A	2	0.244	2
Bis(2-chloroisopropyl)ether	108-60-1	NE	N/A	2	0.403	2
Bis(2-chloroethoxy)methane	111-91-1	NE	N/A	5	0.842	5
Hexachlorobutadiene	87-68-3	0.5	TCLP	2	0.355	2
Hexachlorocyclopentadiene	77-47-4	NE	N/A	20	1.23	20
Hexachloroethane	67-72-1	3.0	TCLP	2	0.203	2
Isophorone	78-59-1	NE	N/A	5	0.862	5
Naphthalene	91-20-3	NE	N/A	2	0.542	2
Nitrobenzene	98-95-3	2.0	TCLP	2	0.205	2
NitrosoDiPhenylAmine(NDPA)/DPA	86-30-6	NE	N/A	2	0.924	2
n-Nitrosodi-n-propylamine	621-64-7	NE	N/A	5	0.906	5
Bis(2-Ethylhexyl)phthalate	117-81-7	NE	N/A	3	1.42	3
Butyl benzyl phthalate	85-68-7	NE	N/A	5	2.61	5

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL) (mg/L)	PAL Reference	Project Quantification Limit (µg/L)	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
Di-n-butylphthalate	84-74-2	NE	N/A	5	0.957	5
Di-n-octylphthalate	117-84-0	NE	N/A	5	2.26	5
Diethyl phthalate	84-66-2	NE	N/A	5	0.765	5
Dimethyl phthalate	131-11-3	NE	N/A	5	0.916	5
Benzo(a)anthracene	56-55-3	NE	N/A	2	0.323	2
Benzo(a)pyrene	50-32-8	NE	N/A	2	0.368	2
Benzo(b)fluoranthene	205-99-2	NE	N/A	2	0.533	2
Benzo(k)fluoranthene	207-08-9	NE	N/A	2	0.621	2
Chrysene	218-01-9	NE	N/A	2	0.222	2
Acenaphthylene	208-96-8	NE	N/A	2	0.315	2
Anthracene	120-12-7	NE	N/A	2	0.467	2
Benzo(ghi)perylene	191-24-2	NE	N/A	2	0.369	2
Fluorene	86-73-7	NE	N/A	2	0.439	2
Phenanthrene	85-01-8	NE	N/A	2	0.419	2
Dibenzo(a,h)anthracene	53-70-3	NE	N/A	2	0.286	2
Indeno(1,2,3-cd)Pyrene	193-39-5	NE	N/A	2	0.484	2

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL) (mg/L)	PAL Reference	Project Quantification Limit (µg/L)	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
Pyrene	129-00-0	NE	N/A	2	0.407	2
Biphenyl	92-52-4	NE	N/A	2	0.196	2
Aniline	62-53-3	NE	N/A	2	0.67	2
4-Chloroaniline	106-47-8	NE	N/A	5	0.468	5
1-Methylnaphthalene	90-12-0	NE	N/A	2	0.415	2
2-Nitroaniline	88-74-4	NE	N/A	5	1.03	5
3-Nitroaniline	99-09-2	NE	N/A	5	1.16	5
4-Nitroaniline	100-01-6	NE	N/A	5	1.45	5
Dibenzofuran	132-64-9	NE	N/A	2	0.401	2
2-Methylnaphthalene	91-57-6	NE	N/A	2	0.372	2
Acetophenone	98-86-2	NE	N/A	5	0.917	5
n-Nitrosodimethylamine	62-75-9	NE	N/A	2	0.44	2
2,4,6-Trichlorophenol	88-06-2	NE	N/A	5	2.09	5
P-Chloro-M-Cresol	59-50-7	NE	N/A	2	0.606	2
2-Chlorophenol	95-57-8	NE	N/A	2	0.653	2
2,4-Dichlorophenol	120-83-2	NE	N/A	5	1.7	5

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL) (mg/L)	PAL Reference	Project Quantification Limit (µg/L)	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
2,4-Dimethylphenol	105-67-9	NE	N/A	5	2.04	5
2-Nitrophenol	88-75-5	NE	N/A	10	1.95	10
4-Nitrophenol	100-02-7	NE	N/A	10	1.42	10
2,4-Dinitrophenol	51-28-5	NE	N/A	20	5.42	20
4,6-Dinitro-o-cresol	534-52-1	NE	N/A	10	2.31	10
Pentachlorophenol	87-86-5	100	TCLP	10	2.51	10
Phenol	108-95-2	NE	N/A	5	0.35	5
2-Methylphenol	95-48-7	NE	N/A	5	2.31	5
3-Methylphenol/4-Methylphenol	106-44-5	NE	N/A	5	1.39	5
2,4,5-Trichlorophenol	95-95-4	NE	N/A	5	2.07	5
Benzoic Acid	65-85-0	NE	N/A	50	2.62	50
Benzyl Alcohol	100-51-6	NE	N/A	2	0.381	2
Carbazole	86-74-8	NE	N/A	2	0.309	2
Pyridine	110-86-1	NE	N/A	3.5	0.308	3.5
2,6-Dichlorophenol	87-65-0	NE	N/A	10	2.57	10

Notes:

[1] Quantitation limit is greater than the calculated regulatory level. The quantitation limit therefore becomes the regulatory level (40 CFR §261.24).

The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.

The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.

MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; NE = not established; N/A = not applicable; TCLP = Toxicity Characteristic Leaching Procedure.

Worksheet #15-12: Reference Limits and Evaluation Table – Metals in Soil

Matrix: Soil
Analytical Method: 6020B – Metals

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (µmoles/gm)	Achievable Laboratory Limits	
					MDL (µmoles/gm)	RL (µmoles/gm)
Cadmium, Total	7440-43-9	2.5	Unrestricted SCO	0.00089	0.00089	0.00089
Copper, Total	7440-50-8	50	Unrestricted SCO	0.0157	0.0157	0.0157
Lead, Total	7439-92-1	63	Unrestricted SCO	0.00966	0.00966	0.00966
Nickel, Total	7440-02-0	30	Unrestricted SCO	0.0342	0.0342	0.0342
Zinc, Total	7440-66-6	109	Unrestricted SCO	0.0612	0.0612	0.0612

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-13: Reference Limits and Evaluation Table – COC Metals in Sediment

Matrix: Sediment
Analytical Method: 6020B – COC Metals

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (µmoles/gm)	Achievable Laboratory Limits	
					MDL (µmoles/gm)	RL (µmoles/gm)
Cadmium, Total	7440-43-9	2.5	Unrestricted SCO	0.00089	0.00089	0.00089
Copper, Total	7440-50-8	50	Unrestricted SCO	0.0157	0.0157	0.0157
Lead, Total	7439-92-1	63	Unrestricted SCO	0.00966	0.00966	0.00966
Nickel, Total	7440-02-0	30	Unrestricted SCO	0.0342	0.0342	0.0342
Zinc, Total	7440-66-6	109	Unrestricted SCO	0.0612	0.0612	0.0612

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-14: Reference Limits and Evaluation Table – TAL Metals in Soil

Matrix: Soil
Analytical Method: 6010D – TAL Metals

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Aluminum, Total	7429-90-5	10000	NY CP-51 Restricted Use Eco SSCO	4	1.08	4
Antimony, Total	7440-36-0	12	NY CP-51 Restricted Use Eco SSCO	2	0.152	2
Arsenic, Total	7440-38-2	13	Unrestricted SCO	0.4	0.0832	0.4
Arsenic, TCLP	7440-38-2	5.0 (mg/L)	TCLP	1	0.019	1
Barium, TCLP	7440-39-3	100 (mg/L)	TCLP	0.5	0.021	0.5
Barium, Total	7440-39-3	350	Unrestricted SCO	0.4	0.0696	0.4
Beryllium, Total	7440-41-7	7.2	Unrestricted SCO	0.2	0.0132	0.2
Cadmium, TCLP	7440-43-9	1.0 (mg/L)	TCLP	0.1	0.01	0.1
Cadmium, Total	7440-43-9	2.5	Unrestricted SCO	0.4	0.0392	0.4
Calcium, Total	7440-70-2	10000	NY CP-51 Restricted Use Eco SSCO	4	1.4	4
Chromium, Total	7440-47-3	30	Unrestricted SCO	0.4	0.0384	0.4
Chromium, TCLP	7440-47-3	5.0 (mg/L)	TCLP	0.2	0.021	0.2

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Cobalt, Total	7440-48-4	20	NY CP-51 Restricted Use Eco SSCO	0.8	0.0664	0.8
Copper, Total	7440-50-8	50	Unrestricted SCO	0.4	0.1032	0.4
Iron, Total	7439-89-6	NE	N/A	2	0.3612	2
Lead, Total	7439-92-1	63	Unrestricted SCO	2	0.1072	2
Lead, TCLP	7439-92-1	5.0 (mg/L)	TCLP	0.5	0.027	0.5
Magnesium, Total	7439-95-4	NE	N/A	4	0.616	4
Manganese, Total	7439-96-5	1600	Unrestricted SCO	0.4	0.0636	0.4
Nickel, Total	7440-02-0	30	Unrestricted SCO	1	0.0968	1
Potassium, Total	7440-09-7	NE	N/A	100	5.76	100
Selenium, TCLP	7782-49-2	1.0 (mg/L)	TCLP	0.5	0.035	0.5
Selenium, Total	7782-49-2	3.9	Unrestricted SCO	0.8	0.1032	0.8
Silver, TCLP	7440-22-4	5.0 (mg/L)	TCLP	0.1	0.028	0.1
Silver, Total	7440-22-4	2.0	Unrestricted SCO	0.2	0.1132	0.2
Sodium, Total	7440-23-5	NE	N/A	80	1.26	80
Thallium, Total	7440-28-0	5	NY CP-51 Restricted Use Eco SSCO	0.8	0.126	0.8
Vanadium, Total	7440-62-2	39	NY CP-51 Restricted Use Eco SSCO	0.4	0.0812	0.4

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Zinc, Total	7440-66-6	109	Unrestricted SCO	2	0.1172	2

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-15: Reference Limits and Evaluation Table – TAL Metals in Sediment

Matrix: Sediment
Analytical Method: 6010D – TAL Metals

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1]	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Aluminum, Total	7429-90-5	10000	NY CP-51 Restricted Use Eco SSCO	4	1.08	4
Antimony, Total	7440-36-0	12	NY CP-51 Restricted Use Eco SSCO	2	0.152	2
Arsenic, Total	7440-38-2	13	Unrestricted SCO	0.4	0.0832	0.4
Arsenic, TCLP	7440-38-2	5.0 (mg/L)	TCLP	1	0.019	1
Barium, TCLP	7440-39-3	100 (mg/L)	TCLP	0.5	0.021	0.5
Barium, Total	7440-39-3	350	Unrestricted SCO	0.4	0.0696	0.4
Beryllium, Total	7440-41-7	7.2	Unrestricted SCO	0.2	0.0132	0.2
Cadmium, TCLP	7440-43-9	1.0 (mg/L)	TCLP	0.1	0.01	0.1
Cadmium, Total	7440-43-9	2.5	Unrestricted SCO	0.4	0.0392	0.4
Calcium, Total	7440-70-2	10000	NY CP-51 Restricted Use Eco SSCO	4	1.4	4
Chromium, Total	7440-47-3	30	Unrestricted SCO	0.4	0.0384	0.4
Chromium, TCLP	7440-47-3	5.0 (mg/L)	TCLP	0.2	0.021	0.2

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1]	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Cobalt, Total	7440-48-4	20	NY CP-51 Restricted Use Eco SSCO	0.8	0.0664	0.8
Copper, Total	7440-50-8	50	Unrestricted SCO	0.4	0.1032	0.4
Iron, Total	7439-89-6	NE	N/A	2	0.3612	2
Lead, Total	7439-92-1	63	Unrestricted SCO	2	0.1072	2
Lead, TCLP	7439-92-1	5.0 (mg/L)	TCLP	0.5	0.027	0.5
Magnesium, Total	7439-95-4	NE	N/A	4	0.616	4
Manganese, Total	7439-96-5	1600	Unrestricted SCO	0.4	0.0636	0.4
Nickel, Total	7440-02-0	30	Unrestricted SCO	1	0.0968	1
Potassium, Total	7440-09-7	NE	N/A	100	5.76	100
Selenium, TCLP	7782-49-2	1.0 (mg/L)	TCLP	0.5	0.035	0.5
Selenium, Total	7782-49-2	3.9	Unrestricted SCO	0.8	0.1032	0.8
Silver, TCLP	7440-22-4	5.0 (mg/L)	TCLP	0.1	0.028	0.1
Silver, Total	7440-22-4	2.0	Unrestricted SCO	0.2	0.1132	0.2
Sodium, Total	7440-23-5	NE	N/A	80	1.26	80
Thallium, Total	7440-28-0	5	NY CP-51 Restricted Use Eco SSCO	0.8	0.126	0.8
Vanadium, Total	7440-62-2	39	NY CP-51 Restricted Use Eco SSCO	0.4	0.0812	0.4

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1]	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Zinc, Total	7440-66-6	109	Unrestricted SCO	2	0.1172	2

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable; TCLP = Toxicity Characteristics Leaching Procedure.

Worksheet #15-16: Reference Limits and Evaluation Table – TCLP Mercury in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: 7470A – Mercury, TCLP

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/L)	PAL Reference[2]	Project Quantification Limit (mg/L)	Achievable Laboratory Limits	
					MDL (mg/L)	RL (mg/L)
Mercury, TCLP	7439-97-6	0.2	TCLP	0.000001	0.0000004575	0.000001

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable; TCLP = Toxicity Characteristics Leaching Procedure.

Worksheet #15-17: Reference Limits and Evaluation Table – Mercury in Soil

Matrix: Soil
Analytical Method: 7471B – Mercury, Total

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1]	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Mercury, Total	7439-97-6	0.18	Unrestricted SCO	0.08	0.05216	0.08

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-18: Reference Limits and Evaluation Table – Mercury in Sediment

Matrix: Sediment
Analytical Method: 7471B – Mercury, Total

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1]	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Mercury, Total	7439-97-6	0.18	Unrestricted SCO	0.08	0.05216	0.08

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-19: Reference Limits and Evaluation Table – Pesticides in Soil

Matrix: Soil
Analytical Method: 8081B – Pesticides

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1]	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Delta-BHC	319-86-8	40	Unrestricted SCO	1.6008	0.31349	1.6008
Lindane	58-89-9	100	Unrestricted SCO	0.667	0.298149	0.667
Alpha-BHC	319-84-6	20	Unrestricted SCO	0.667	0.189428	0.667
Beta-BHC	319-85-7	36	Unrestricted SCO	1.6008	0.60697	1.6008
Heptachlor	76-44-8	42	Unrestricted SCO	0.8004	0.358846	0.8004
Aldrin	309-00-2	5	Unrestricted SCO	1.6008	0.563615	1.6008
Heptachlor epoxide	1024-57-3	NE	N/A	3.0015	0.90045	3.0015
Endrin	72-20-8	14	Unrestricted SCO	0.667	0.27347	0.667
Endrin aldehyde	7421-93-4	NE	N/A	2.001	0.70035	2.001
Endrin ketone	53494-70-5	NE	N/A	1.6008	0.412206	1.6008
Dieldrin	60-57-1	5	Unrestricted SCO	1.0005	0.50025	1.0005
4,4’-DDE	72-55-9	3.3	Unrestricted SCO	1.6008	0.370185	1.6008

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1]	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
4,4'-DDD	72-54-8	3.3	Unrestricted SCO	1.6008	0.570952	1.6008
4,4'-DDT	50-29-3	3.3	Unrestricted SCO	1.6008	1.28731	1.6008
Endosulfan I	959-98-8	2400	Unrestricted SCO	1.6008	0.378189	1.6008
Endosulfan II	33213-65-9	2400	Unrestricted SCO	1.6008	0.534934	1.6008
Endosulfan sulfate	1031-07-8	2400	Unrestricted SCO	0.667	0.317492	0.667
Methoxychlor	72-43-5	1200	NY CP-51 Restricted Use Eco SSCO	3.0015	0.9338	3.0015
Toxaphene	8001-35-2	NE	N/A	30.015	8.4042	30.015
cis-Chlordane	5103-71-9	94	Unrestricted SCO	2.001	0.557612	2.001
trans-Chlordane	5103-74-2	NE	N/A	2.001	0.528264	2.001
Chlordane	57-74-9	NE	N/A	13.34	5.30265	13.34

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-20: Reference Limits and Evaluation Table – Pesticides in Sediment

Matrix: Sediment
Analytical Method: 8081B – Pesticides

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1]	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
Delta-BHC	319-86-8	40	Unrestricted SCO	1.6008	0.31349	1.6008
Lindane	58-89-9	78	Freshwater Sediment Guidance Values B	0.667	0.298149	0.667
Alpha-BHC	319-84-6	20	Unrestricted SCO	0.667	0.189428	0.667
Beta-BHC	319-85-7	36	Unrestricted SCO	1.6008	0.60697	1.6008
Heptachlor	76-44-8	42	Unrestricted SCO	0.8004	0.358846	0.8004
Aldrin	309-00-2	5	Unrestricted SCO	1.6008	0.563615	1.6008
Heptachlor epoxide	1024-57-3	2100	Freshwater Sediment Guidance Values B	3.0015	0.90045	3.0015
Endrin	72-20-8	14	Unrestricted SCO	0.667	0.27347	0.667
Endrin aldehyde	7421-93-4	NE	N/A	2.001	0.70035	2.001
Endrin ketone	53494-70-5	NE	N/A	1.6008	0.412206	1.6008
Dieldrin	60-57-1	5	Unrestricted SCO	1.0005	0.50025	1.0005

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1]	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL (µg/kg)	RL (µg/kg)
4,4'-DDE	72-55-9	3.3	Unrestricted SCO	1.6008	0.370185	1.6008
4,4'-DDD	72-54-8	3.3	Unrestricted SCO	1.6008	0.570952	1.6008
4,4'-DDT	50-29-3	3.3	Unrestricted SCO	1.6008	1.28731	1.6008
Endosulfan I	959-98-8	2400	Unrestricted SCO	1.6008	0.378189	1.6008
Endosulfan II	33213-65-9	2400	Unrestricted SCO	1.6008	0.534934	1.6008
Endosulfan sulfate	1031-07-8	2400	Unrestricted SCO	0.667	0.317492	0.667
Methoxychlor	72-43-5	1200	NY CP-51 Restricted Use Eco SSCO	3.0015	0.9338	3.0015
Toxaphene	8001-35-2	250	Freshwater Sediment Guidance Values B	30.015	8.4042	30.015
cis-Chlordane	5103-71-9	94	Unrestricted SCO	2.001	0.557612	2.001
trans-Chlordane	5103-74-2	NE	N/A	2.001	0.528264	2.001
Chlordane	57-74-9	NE	N/A	13.34	5.30265	13.34

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-21: Reference Limits and Evaluation Table – Reactive Cyanide in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: 7.3 – Cyanide, Reactive

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)	PAL Reference	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Cyanide, Reactive	57-12-5	N/A	N/A	10	10	10

Notes:
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-22: Reference Limits and Evaluation Table – Reactive Sulfide in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: 7.3 – Sulfide, Reactive

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)	PAL Reference	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Sulfide, Reactive	None	N/A	N/A	10	10	10

Notes:
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-23: Reference Limits and Evaluation Table – pH in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: 9045D – pH

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)	PAL Reference	Project Quantification Limit (SU)	Achievable Laboratory Limits	
					MDL (SU)	RL (SU)
pH	12408-02-5	N/A	N/A	0	N/A	0

Notes:
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-24: Reference Limits and Evaluation Table – Total Cyanide in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: 9012B – Cyanide, Total

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)	PAL Reference	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Cyanide, Total	57-12-5	27	Unrestricted SCO	1	0.212	1

Notes:
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-25: Reference Limits and Evaluation Table – Nitrogen/Ammonia in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: 4500NH3-BH – Nitrogen, Ammonia

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1]	PAL Reference[2]	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
Nitrogen, Ammonia	7664-41-7	N/A	N/A	7.5	2.79	7.5

Notes:
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-26: Reference Limits and Evaluation Table – Total Organic Carbon in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: 9060A/Lloyd Kahn – Total Organic Carbon

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)	PAL Reference	Project Quantification Limit (%)	Achievable Laboratory Limits	
					MDL (%)	RL (%)
Total Organic Carbon	7440-44-0	NE	N/A	0.01	0.01	0.01
Total Organic Carbon	7440-44-0	NE	N/A	0.05	0.05	0.05

Notes:
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-27: Reference Limits and Evaluation Table – Grain Size in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: ASTM D422 – Grain Size

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)	PAL Reference	Project Quantification Limit (%)	Achievable Laboratory Limits	
					MDL (%)	RL (%)
Cobbles	None	N/A	N/A	0.1	N/A	0.1
% Coarse Gravel	None	N/A	N/A	0.1	N/A	0.1
% Fine Gravel	None	N/A	N/A	0.1	N/A	0.1
% Total Gravel	None	N/A	N/A	0.1	N/A	0.1
% Coarse Sand	None	N/A	N/A	0.1	N/A	0.1
% Medium Sand	None	N/A	N/A	0.1	N/A	0.1
% Fine Sand	None	N/A	N/A	0.1	N/A	0.1
% Total Sand	None	N/A	N/A	0.1	N/A	0.1
% Silt Fine	None	N/A	N/A	0.1	N/A	0.1
% Clay Fine	None	N/A	N/A	0.1	N/A	0.1
% Total Fines	None	N/A	N/A	0.1	N/A	0.1

Notes:
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; NE = not established; N/A = not applicable.

Worksheet #15-28: Reference Limits and Evaluation Table – Herbicides in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: 8151A – Herbicides

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL) (mg/kg)	PAL Reference	Project Quantification Limit (mg/kg)	Achievable Laboratory Limits	
					MDL (mg/kg)	RL (mg/kg)
2,4-D	94-75-7	NE	N/A	0.0104895	0.1665	0.0104895
2,4,5-T	93-76-5	NE	N/A	0.0051615	0.1665	0.0051615
2,4,5-TP (Silvex)	93-72-1	NE	N/A	0.0044289	0.1665	0.0044289

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-29: Reference Limits and Evaluation Table – Ignitability in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: 1030 – Ignitability

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)	PAL Reference	Project Quantification Limit	Achievable Laboratory Limits	
					MDL	RL
Ignitability	N/A	NE	N/A	N/A	N/A	NA/

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte. Results are reported as either positive or negative.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-30: Reference Limits and Evaluation Table – BTU in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: ASTM D240-87 – BTU

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)	PAL Reference	Project Quantification Limit (BTU/lb)	Achievable Laboratory Limits	
					MDL (BTU/lb)	RL (BTU/lb)
BTU	N/A	NE	N/A	50	50	50

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte. Results are reported as either positive or negative.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-31: Reference Limits and Evaluation Table – Percent Sulfur in Soil/Sediment

Matrix: Soil/Sediment
Analytical Method: ASTM D4239 – Percent Sulfur

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)	PAL Reference	Project Quantification Limit (%)	Achievable Laboratory Limits	
					MDL (%)	RL (%)
Percent Sulfur	N/A	NE	N/A	0.02	0.02	0.02

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte. Results are reported as either positive or negative.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-32: Reference Limits and Evaluation Table – TCL SVOCs Expanded NOAA PAH 34 in Sediment

Matrix: Sediment
Analytical Method: 8270E-SIM – PAH 34

Analyte	CAS Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL	RL
Cis/Trans-Decalin	493-01-6/493-02-7	NE	N/A	0.5025	0.099897	0.5025
C1-Decalins	DECALINSC1	NE	N/A	0.5025	0.099897	0.5025
C2-Decalins	DECALINSC2	NE	N/A	0.5025	0.099897	0.5025
C3-Decalins	DECALINSC3	NE	N/A	0.5025	0.099897	0.5025
C4-Decalins	DECALINSC4	NE	N/A	0.5025	0.099897	0.5025
Naphthalene	91-20-3	12000	Unrestricted SCO	1.005	0.288837	1.005
C1-Naphthalenes	91-20-3C1	NE	N/A	1.005	0.288837	1.005
C2-Naphthalenes	91-20-3C2	NE	N/A	1.005	0.288837	1.005
C3-Naphthalenes	91-20-3C3	NE	N/A	1.005	0.288837	1.005
C4-Naphthalenes	91-20-3C4	NE	N/A	1.005	0.288837	1.005
2-Methylnaphthalene	91-57-6	NE	N/A	1.005	0.259156	1.005
1-Methylnaphthalene	90-12-0	NE	N/A	1.005	0.316642	1.005
Benzothiophene	95-15-8	NE	N/A	1.005	0.314766	1.005

Analyte	CAS Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL	RL
C1-Benzo(b)thiophenes	95-15-8C1	NE	N/A	1.005	0.314766	1.005
C2-Benzo(b)thiophenes	95-15-8C2	NE	N/A	1.005	0.314766	1.005
C3-Benzo(b)thiophenes	95-15-8C3	NE	N/A	1.005	0.314766	1.005
C4-Benzo(b)thiophenes	95-15-8C4	NE	N/A	1.005	0.314766	1.005
Biphenyl	92-52-4	60000	NY CP-51 Restricted Use ECO SSCO	1.005	0.310545	1.005
2,6-Dimethylnaphthalene	581-42-0	NE	N/A	1.005	0.238855	1.005
Dibenzofuran	132-64-9	7000	Unrestricted SCO	1.005	0.316508	1.005
Acenaphthylene	208-96-8	100000	Unrestricted SCO	1.005	0.191754	1.005
Acenaphthene	83-32-9	20000	Unrestricted SCO	1.005	0.177148	1.005
2,3,5-Trimethylnaphthalene	2245-38-7	NE	N/A	1.005	0.164351	1.005
Fluorene	86-73-7	30000	Unrestricted SCO	1.005	0.268067	1.005
C1-Fluorenes	86-73-7C1	NE	N/A	1.005	0.268067	1.005
C2-Fluorenes	86-73-7C2	NE	N/A	1.005	0.268067	1.005
C3-Fluorenes	86-73-7C3	NE	N/A	1.005	0.268067	1.005
Dibenzothiophene	132-65-0	NE	N/A	1.005	0.277112	1.005
4-Methyldibenzothiophene(4MDT)	7372-88-5	NE	N/A	1.005	0.277112	1.005
2/3-Methyldibenzothiophene(2MDT)	20928-02-3/16587-52-3	NE	N/A	1.005	0.277112	1.005

Analyte	CAS Number	Project Action Limit (PAL)[1] (mg/kg)	PAL Reference[2]	Project Quantification Limit (µg/kg)	Achievable Laboratory Limits	
					MDL	RL
1-Methyldibenzothiophene(1MDT)	31317-07-4	NE	N/A	1.005	0.277112	1.005
C1-Dibenzothiophenes	132-65-0C1	NE	N/A	1.005	0.277112	1.005
C2-Dibenzothiophenes	132-65-0C2	NE	N/A	1.005	0.277112	1.005
C3-Dibenzothiophenes	132-65-0C3	NE	N/A	1.005	0.277112	1.005
C4-Dibenzothiophenes	132-65-0C4	NE	N/A	1.005	0.277112	1.005
Phenanthrene	85-01-8	100000	Unrestricted SCO	1.005	0.33299	1.005
3-Methylphenanthrene (3MP)	832-71-3	NE	N/A	1.005	0.33299	1.005
2-Methylphenanthrene (2MP)	2531-84-2	NE	N/A	1.005	0.33299	1.005
2-Methylanthracene (2MA)	613-12-7	NE	N/A	1.005	0.33299	1.005
9/4-Methylphenanthrene (9MP)	883-20-5/832-64-4	NE	N/A	1.005	0.33299	1.005
1-Methylphenanthrene (1MP)	832-69-9	NE	N/A	1.005	0.33299	1.005
C1-Phenanthrenes/Anthracenes	PHENANTHC1	NE	N/A	1.005	0.33299	1.005

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-33: Reference Limits and Evaluation Table – EPH in Sediment

Matrix: Solid
Analytical Method: MassDEP EPH

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (mg/L)	RL (mg/L)
C9-C18 Aliphatics	C9-C18-ALPHA-UJ	NE	N/A	6.65	6.65	6.65
C19-C36 Aliphatics	C19-C36-ALPHA-UJ	NE	N/A	6.65	6.65	6.65
C11-C12 Aromatics	C11-C22-ALPHA-UJ	NE	N/A	6.65	6.65	6.65
C11-C22 Aromatics	C11-C22-ALPHA-J	NE	N/A	6.65	6.65	6.65

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with the New York State Register and Official Compilation of Codes, Rules and Regulations of the State of New York (6 NYCRR) and NYCRR, Chapter IV, Part 375-6 Soil Cleanup Objectives (SCOs): Unrestricted Use, Restricted-Residential, Protection of Ecological Resources, and Freshwater Sediment Guidance Values Class B. PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; SCO = Soil Cleanup Objectives; NE = not established; N/A = not applicable.

Worksheet #15-34: Reference Limits and Evaluation Table – PFAS in Water

Matrix: Aqueous
Analytical Method: 1633 – PFAS

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (ng/L)	PAL Reference[2]	Project Quantification Limit (ng/L)	Achievable Laboratory Limits	
					MDL (ng/L)	RL (ng/L)
Perfluorobutanoic Acid (PFBA)	375-22-4	NE	N/A	6.4	0.528	6.4
Perfluoropentanoic Acid (PFPeA)	2706-90-3	NE	N/A	3.2	0.36	3.2
Perfluorobutanesulfonic Acid (PFBS)	375-73-5	NE	N/A	1.6	0.4	1.6
1H,1H,2H,2H-Perfluorohexanesulfonic Acid (4:2FTS)	757124-72-4	NE	N/A	6.4	0.912	6.4
Perfluorohexanoic Acid (PFHxA)	307-24-4	NE	N/A	1.6	0.248	1.6
Perfluoropentanesulfonic Acid (PFPeS)	2706-91-4	NE	N/A	1.6	0.208	1.6
Perfluoroheptanoic Acid (PFHpA)	375-85-9	NE	N/A	1.6	0.24	1.6
Perfluorohexanesulfonic Acid (PFHxS)	355-46-4	NE	N/A	1.6	0.136	1.6
Perfluorooctanoic Acid (PFOA)	335-67-1	6.7	NYSDEC Ambient Water Quality Guidance	1.6	0.264	1.6
1H,1H,2H,2H-Perfluorooctanesulfonic Acid (6:2FTS)	27619-97-2	NE	N/A	6.4	4.816	6.4
Perfluoroheptanesulfonic Acid (PFHpS)	375-92-8	NE	N/A	1.6	0.2	1.6

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (ng/L)	PAL Reference[2]	Project Quantification Limit (ng/L)	Achievable Laboratory Limits	
					MDL (ng/L)	RL (ng/L)
Perfluorononanoic Acid (PFNA)	375-95-1	NE	N/A	1.6	0.264	1.6
Perfluorooctanesulfonic Acid (PFOS)	1763-23-1	2.7	NYSDEC Ambient Water Quality Guidance	1.6	0.264	1.6
Perfluorodecanoic Acid (PFDA)	335-76-2	NE	N/A	1.6	0.208	1.6
1H,1H,2H,2H-Perfluorodecanesulfonic Acid (8:2FTS)	39108-34-4	NE	N/A	6.4	1.224	6.4
Perfluorononanesulfonic Acid (PFNS)	68259-12-1	NE	N/A	1.6	0.2	1.6
N-Methyl Perfluorooctanesulfonamidoacetic Acid (NMeFOSAA)	2355-31-9	NE	N/A	1.6	0.48	1.6
Perfluoroundecanoic Acid (PFUnA)	2058-94-8	NE	N/A	1.6	0.176	1.6
Perfluorodecanesulfonic Acid (PFDS)	335-77-3	NE	N/A	1.6	0.136	1.6
Perfluorooctanesulfonamide (FOSA)	754-91-6	NE	N/A	1.6	0.096	1.6
N-Ethyl Perfluorooctanesulfonamidoacetic Acid (NEtFOSAA)	2991-50-6	NE	N/A	1.6	0.48	1.6
Perfluorododecanoic Acid (PFDoA)	307-55-1	NE	N/A	1.6	0.216	1.6
Perfluorotridecanoic Acid (PFTrDA)	72629-94-8	NE	N/A	1.6	0.184	1.6
Perfluorotetradecanoic Acid (PFTA)	376-06-7	NE	N/A	1.6	0.16	1.6

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (ng/L)	PAL Reference[2]	Project Quantification Limit (ng/L)	Achievable Laboratory Limits	
					MDL (ng/L)	RL (ng/L)
2,3,3,3-Tetrafluoro-2-[1,1,2,2,3,3,3-Heptafluoropropoxy]-Propanoic Acid (HFPO-DA)	13252-13-6	NE	N/A	6.4	1.6	6.4
4,8-Dioxa-3h-Perfluorononanoic Acid (ADONA)	919005-14-4	NE	N/A	6.4	0.376	6.4
Perfluorododecane Sulfonic Acid (PFDoDS)	79780-39-5	NE	N/A	1.6	0.24	1.6
9-Chlorohexadecafluoro-3-Oxanone-1-Sulfonic Acid (9Cl-PF3ONS)	756426-58-1	NE	N/A	6.4	0.44	6.4
11-Chloroeicosafluoro-3-Oxaundecane-1-Sulfonic Acid (11Cl-PF3OudS)	763051-92-9	NE	N/A	6.4	0.448	6.4
N-Methyl Perfluorooctane Sulfonamide (NMeFOSA)	31506-32-8	NE	N/A	1.6	0.224	1.6
N-Ethyl Perfluorooctane Sulfonamide (NEtFOSA)	4151-50-2	NE	N/A	1.6	0.352	1.6
N-Methyl Perfluorooctanesulfonamido Ethanol (NMeFOSE)	24448-09-7	NE	N/A	16	1.304	16
N-Ethyl Perfluorooctanesulfonamido Ethanol (NEtFOSE)	1691-99-2	NE	N/A	16	1.104	16
Perfluoro-3-Methoxypropanoic Acid (PFMPA)	377-73-1	NE	N/A	3.2	0.248	3.2
Perfluoro-4-Methoxybutanoic Acid (PFMBA)	863090-89-5	NE	N/A	3.2	0.36	3.2

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (ng/L)	PAL Reference[2]	Project Quantification Limit (ng/L)	Achievable Laboratory Limits	
					MDL (ng/L)	RL (ng/L)
Perfluoro(2-Ethoxyethane)Sulfonic Acid (PFEESA)	113507-82-7	NE	N/A	3.2	0.328	3.2
Nonafluoro-3,6-Dioxaheptanoic Acid (NFDHA)	151772-58-6	NE	N/A	3.2	0.544	3.2
3-Perfluoropropyl Propanoic Acid (3:3FTCA)	356-02-5	NE	N/A	8	0.536	8
2H,2H,3H,3H-Perfluorooctanoic Acid (5:3FTCA)	914637-49-3	NE	N/A	40	4.256	40
3-Perfluoroheptyl Propanoic Acid (7:3FTCA)	812-70-4	NE	N/A	40	3.184	40

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with New York State Ambient Water Quality Standards and Guidance Values for GA groundwater (NYS AWQS) and NYSDEC Interim Groundwater Screening Levels based on NYSDEC guidance as follows:

- March 2023 (PFAS and 1,4-Dioxane)

PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; NYS AWQS = New York State Ambient Water Quality Standards; PFAS = per- and polyfluoroalkyl substances; NE = not established; N/A = not applicable.

Worksheet #15-35: Reference Limits and Evaluation Table – 1,4 Dioxane in Water

Matrix: Aqueous
Analytical Method: 8270E-SIM – 1,4 Dioxane

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (ng/L)	PAL Reference[2]	Project Quantification Limit (ng/L)	Achievable Laboratory Limits	
					MDL (ng/L)	RL (ng/L)
1,4-Dioxane	123-91-1	350	NYSDEC Guidance	150	33.9	150

Notes:
[1,2] Established to align with New York State Ambient Water Quality Standards and Guidance Values for GA groundwater (NYS AWQS) and NYSDEC Interim Groundwater Screening Levels based on NYSDEC guidance as follows:

- March 2023 (PFAS and 1,4-Dioxane)

PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; NYS AWQS = New York State Ambient Water Quality Standards; PFAS = per- and polyfluoroalkyl substances; NE = not established; N/A = not applicable.

Worksheet #15-36: Reference Limits and Evaluation Table – SVOCs in Water

Matrix: Aqueous
Analytical Method: 8270E – SVOCs

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
Acenaphthene	83-32-9	20*	NY AWQS	2	0.403	2
1,2,4-Trichlorobenzene	120-82-1	5	NY AWQS	5	0.977	5
Hexachlorobenzene	118-74-1	0.04	NY AWQS	2	0.452	2
Bis(2-chloroethyl)ether	111-44-4	1	NY AWQS	2	0.392	2
2-Chloronaphthalene	91-58-7	10*	NY AWQS	2	0.35	2
1,2-Dichlorobenzene	95-50-1	3	NY AWQS	2	0.329	2
1,3-Dichlorobenzene	541-73-1	3	NY AWQS	2	0.315	2
1,4-Dichlorobenzene	106-46-7	3	NY AWQS	2	0.391	2
3,3'-Dichlorobenzidine	91-94-1	5	NY AWQS	5	1.85	5
2,4-Dinitrotoluene	121-14-2	5	NY AWQS	5	0.541	5
2,6-Dinitrotoluene	606-20-2	5	NY AWQS	5	0.845	5
Fluoranthene	206-44-0	50*	NY AWQS	2	0.411	2

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
4-Chlorophenyl phenyl ether	7005-72-3	NE	N/A	2	0.386	2
4-Bromophenyl phenyl ether	101-55-3	NE	N/A	2	0.244	2
Bis(2-chloroisopropyl)ether	108-60-1	5	NY AWQS	2	0.403	2
Bis(2-chloroethoxy)methane	111-91-1	5	NY AWQS	5	0.842	5
Hexachlorobutadiene	87-68-3	0.5	NY AWQS	2	0.355	2
Hexachlorocyclopentadiene	77-47-4	5	NY AWQS	20	1.23	20
Hexachloroethane	67-72-1	5	NY AWQS	2	0.203	2
Isophorone	78-59-1	50*	NY AWQS	5	0.862	5
Naphthalene	91-20-3	10*	NY AWQS	2	0.542	2
Nitrobenzene	98-95-3	0.4	NY AWQS	2	0.205	2
NitrosoDiPhenylAmine(NDPA)/DPA	86-30-6	50*	NY AWQS	2	0.924	2
n-Nitrosodi-n-propylamine	621-64-7	NE	N/A	5	0.906	5
Bis(2-Ethylhexyl)phthalate	117-81-7	5	NY AWQS	3	1.42	3
Butyl benzyl phthalate	85-68-7	50*	NY AWQS	5	2.61	5
Di-n-butylphthalate	84-74-2	50	NY AWQS	5	0.957	5
Di-n-octylphthalate	117-84-0	50*	NY AWQS	5	2.26	5

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
Diethyl phthalate	84-66-2	50*	NY AWQS	5	0.765	5
Dimethyl phthalate	131-11-3	50*	NY AWQS	5	0.916	5
Benzo(a)anthracene	56-55-3	0.002*	NY AWQS	2	0.323	2
Benzo(a)pyrene	50-32-8	ND	NY AWQS	2	0.368	2
Benzo(b)fluoranthene	205-99-2	0.002*	NY AWQS	2	0.533	2
Benzo(k)fluoranthene	207-08-9	0.002*	NY AWQS	2	0.621	2
Chrysene	218-01-9	0.002*	NY AWQS	2	0.222	2
Acenaphthylene	208-96-8	NE	N/A	2	0.315	2
Anthracene	120-12-7	50*	NY AWQS	2	0.467	2
Benzo(ghi)perylene	191-24-2	NE	N/A	2	0.369	2
Fluorene	86-73-7	50*	NY AWQS	2	0.439	2
Phenanthrene	85-01-8	50*	NY AWQS	2	0.419	2
Dibenzo(a,h)anthracene	53-70-3	NE	N/A	2	0.286	2
Indeno(1,2,3-cd)Pyrene	193-39-5	0.002*	NY AWQS	2	0.484	2
Pyrene	129-00-0	50*	NY AWQS	2	0.407	2
Biphenyl	92-52-4	5	NY AWQS	2	0.196	2

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
4-Chloroaniline	106-47-8	5	NY AWQS	5	0.468	5
2-Nitroaniline	88-74-4	5	NY AWQS	5	1.03	5
3-Nitroaniline	99-09-2	5	NY AWQS	5	1.16	5
4-Nitroaniline	100-01-6	5	NY AWQS	5	1.45	5
Dibenzofuran	132-64-9	NE	N/A	2	0.401	2
2-Methylnaphthalene	91-57-6	NE	N/A	2	0.372	2
Acetophenone	98-86-2	NE	N/A	5	0.917	5
2,4,6-Trichlorophenol	88-06-2	NE	N/A	5	2.09	5
P-Chloro-M-Cresol	59-50-7	NE	N/A	2	0.606	2
2-Chlorophenol	95-57-8	NE	N/A	2	0.653	2
2,4-Dichlorophenol	120-83-2	5	NY AWQS	5	1.7	5
2,4-Dimethylphenol	105-67-9	50*	NY AWQS	5	2.04	5
2-Nitrophenol	88-75-5	NE	N/A	10	1.95	10
4-Nitrophenol	100-02-7	NE	N/A	10	1.42	10
2,4-Dinitrophenol	51-28-5	10*	NY AWQS	20	5.42	20
4,6-Dinitro-o-cresol	534-52-1	NE	N/A	10	2.31	10

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
Pentachlorophenol	87-86-5	1	NY AWQS	10	2.51	10
Phenol	108-95-2	1	NY AWQS	5	0.35	5
2-Methylphenol	95-48-7	1	NY AWQS	5	2.31	5
3-Methylphenol/4-Methylphenol	106-44-5	NE	N/A	5	1.39	5
2,4,5-Trichlorophenol	95-95-4	NE	N/A	5	2.07	5
Benzoic Acid	65-85-0	NE	N/A	50	2.62	50
Benzyl Alcohol	100-51-6	NE	N/A	2	0.381	2
Carbazole	86-74-8	NE	N/A	2	0.309	2

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with New York State Ambient Water Quality Standards and Guidance Values for GA groundwater (NYS AWQS) and NYSDEC Interim Groundwater Screening Levels based on NYSDEC guidance as follows:

- March 2023 (PFAS and 1,4 Dioxane)

PAL is based on the lower regulatory criteria.
The Project Action Limit (PAL) is the regulatory screening level established for the Site. Reported concentrations above the corresponding PAL for that analyte will trigger a closer review of the data, including potential mitigation efforts at the sample location.
The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
* Indicates the value is a guidance value and not a standard.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; NYS AWQS = New York State Ambient Water Quality Standards; PFAS = per- and polyfluoroalkyl substances; NE = not established; N/A = not applicable.

Worksheet #15-37: Reference Limits and Evaluation Table – Metals in Water

Matrix: Aqueous
Analytical Method: 6020B – Metals

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (mg/L)	RL (mg/L)
Aluminum, Total	7429-90-5	NE	N/A	0.01	0.00327	0.01
Antimony, Total	7440-36-0	0.003	NY AWQS	0.004	0.000429	0.004
Arsenic, Total	7440-38-2	0.025	NY AWQS	0.0005	0.000165	0.0005
Barium, Total	7440-39-3	1	NY AWQS	0.0005	0.000173	0.0005
Beryllium, Total	7440-41-7	0.003*	NY AWQS	0.0005	0.000107	0.0005
Cadmium, Total	7440-43-9	0.005	NY AWQS	0.0002	0.0000599	0.0002
Calcium, Total	7440-70-2	NE	N/A	0.1	0.0394	0.1
Chromium, Total	7440-47-3	0.05	NY AWQS	0.001	0.000178	0.001
Cobalt, Total	7440-48-4	NE	N/A	0.0005	0.000163	0.0005
Copper, Total	7440-50-8	0.2	NY AWQS	0.001	0.000384	0.001
Iron, Total	7439-89-6	0.3	NY AWQS	0.05	0.0191	0.05
Lead, Total	7439-92-1	0.025	NY AWQS	0.001	0.000343	0.001
Magnesium, Total	7439-95-4	35*	NY AWQS	0.07	0.0242	0.07

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (mg/L)	RL (mg/L)
Manganese, Total	7439-96-5	0.3	NY AWQS	0.001	0.00044	0.001
Nickel, Total	7440-02-0	0.1	NY AWQS	0.002	0.000556	0.002
Potassium, Total	7440-09-7	NE	N/A	0.1	0.0309	0.1
Selenium, Total	7782-49-2	0.01	NY AWQS	0.005	0.00173	0.005
Silver, Total	7440-22-4	0.05	NY AWQS	0.0004	0.000163	0.0004
Sodium, Total	7440-23-5	20	NY AWQS	0.5	0.0293	0.5
Thallium, Total	7440-28-0	0.0005*	NY AWQS	0.001	0.000143	0.001
Vanadium, Total	7440-62-2	NE	N/A	0.005	0.00157	0.005
Zinc, Total	7440-66-6	2*	NY AWQS	0.01	0.00341	0.01

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with New York State Ambient Water Quality Standards and Guidance Values for GA groundwater (NYS AWQS) and NYSDEC Interim Groundwater Screening Levels based on NYSDEC guidance as follows:

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Worksheet #15-38: Reference Limits and Evaluation Table – Mercury in Water

Matrix: Aqueous
Analytical Method: 7470A – Mercury

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (mg/L)	RL (mg/L)
Mercury, Total	7439-97-6	0.0007	NY AWQS	0.0002	0.0000915	0.0002

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
[2]Established to align with New York State Ambient Water Quality Standards and Guidance Values for GA groundwater (NYS AWQS) and NYSDEC Interim Groundwater Screening Levels based on NYSDEC guidance as follows:

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The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.
* Indicates the value is a guidance value and not a standard.
MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; NYS AWQS = New York State Ambient Water Quality Standards; PFAS = per- and polyfluoroalkyl substances; NE = not established; N/A = not applicable.

Worksheet #15-39: Reference Limits and Evaluation Table – PCB Aroclors in Water

Matrix: Aqueous
Analytical Method: 8082A – PCBs – Aroclors

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
Aroclor 1016	12674-11-2	NE	N/A	0.071	0.0128	0.071
Aroclor 1221	11104-28-2	NE	N/A	0.071	0.0153	0.071
Aroclor 1232	11141-16-5	NE	N/A	0.071	0.0153	0.071
Aroclor 1242	53469-21-9	NE	N/A	0.071	0.0153	0.071
Aroclor 1248	12672-29-6	NE	N/A	0.071	0.0153	0.071
Aroclor 1254	11097-69-1	NE	N/A	0.071	0.0153	0.071
Aroclor 1260	11096-82-5	NE	N/A	0.071	0.0153	0.071
Aroclor 1262	37324-23-5	NE	N/A	0.071	0.0153	0.071
Aroclor 1268	11100-14-4	NE	N/A	0.071	0.0153	0.071
PCBs, Total	1336-36-3	0.09	NY AWQS	0.071	0.0153	0.071

Notes:

[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).

[2]Established to align with New York State Ambient Water Quality Standards and Guidance Values for GA groundwater (NYS AWQS) and NYSDEC Interim Groundwater Screening Levels based on NYSDEC guidance as follows:

- March 2023 (PFAS and 1,4 Dioxane)

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The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.

* Indicates the value is a guidance value and not a standard.

MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; NYS AWQS = New York State Ambient Water Quality Standards; PFAS = per- and polyfluoroalkyl substances; NE = not established; N/A = not applicable.

Worksheet #15-40: Reference Limits and Evaluation Table – TCL SVOCs in Water

Matrix: Aqueous
Analytical Method: 8270E – TCL SVOCs

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
Acenaphthene	83-32-9	20*	NY AWQS	0.1	0.0235	0.1
2-Chloronaphthalene	91-58-7	10*	NY AWQS	0.2	0.0227	0.2
Fluoranthene	206-44-0	50*	NY AWQS	0.1	0.0272	0.1
Hexachlorobutadiene	87-68-3	0.5	NY AWQS	0.5	0.02	0.5
Naphthalene	91-20-3	10*	NY AWQS	0.1	0.0245	0.1
Benzo(a)anthracene	56-55-3	0.002*	NY AWQS	0.1	0.0295	0.1
Benzo(a)pyrene	50-32-8	ND	NY AWQS	0.1	0.024	0.1
Benzo(b)fluoranthene	205-99-2	0.002*	NY AWQS	0.1	0.0272	0.1
Benzo(k)fluoranthene	207-08-9	0.002*	NY AWQS	0.1	0.0338	0.1
Chrysene	218-01-9	0.002*	NY AWQS	0.1	0.031	0.1
Acenaphthylene	208-96-8	NE	N/A	0.1	0.0205	0.1
Anthracene	120-12-7	50*	NY AWQS	0.1	0.0239	0.1

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
Benzo(ghi)perylene	191-24-2	NE	N/A	0.1	0.0237	0.1
Fluorene	86-73-7	50*	NY AWQS	0.1	0.0255	0.1
Phenanthrene	85-01-8	50*	NY AWQS	0.1	0.0392	0.1
Dibenzo(a,h)anthracene	53-70-3	NE	N/A	0.1	0.0235	0.1
Indeno(1,2,3-cd)Pyrene	193-39-5	0.002*	NY AWQS	0.1	0.022	0.1
Pyrene	129-00-0	50*	NY AWQS	0.1	0.0425	0.1
2-Methylnaphthalene	91-57-6	NE	N/A	0.1	0.0277	0.1
Pentachlorophenol	87-86-5	1	NY AWQS	0.8	0.0565	0.8
Hexachlorobenzene	118-74-1	0.04	NY AWQS	0.8	0.0134	0.8
Hexachloroethane	67-72-1	5	NY AWQS	0.8	0.0224	0.8
Acenaphthene	83-32-9	20*	NY AWQS	0.1	0.0235	0.1
2-Chloronaphthalene	91-58-7	10*	NY AWQS	0.2	0.0227	0.2
Fluoranthene	206-44-0	50*	NY AWQS	0.1	0.0272	0.1

Notes:

[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).

[2]Established to align with New York State Ambient Water Quality Standards and Guidance Values for GA groundwater (NYS AWQS) and NYSDEC Interim Groundwater Screening Levels based on NYSDEC guidance as follows:

- March 2023 (PFAS and 1,4 Dioxane)

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The Project Quantification Limit (PQL) is the minimum concentration to which a detection for the corresponding analyte must be reported and corresponds to the Reporting Limit for that analyte.

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Worksheet #15-41: Reference Limits and Evaluation Table – VOCs in Water

Matrix: Aqueous
Analytical Method: 8260D – TCL VOCs

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
Methylene chloride	75-09-2	5	NY AWQS	0.5	0.144	0.5
1,1-Dichloroethane	75-34-3	5	NY AWQS	2	0.65	2
Chloroform	67-66-3	7	NY AWQS	0.5	0.167	0.5
Carbon tetrachloride	56-23-5	5	NY AWQS	0.5	0.159	0.5
1,2-Dichloropropane	78-87-5	1	NY AWQS	2.5	0.7	2.5
Dibromochloromethane	124-48-1	50*	NY AWQS	2.5	0.7	2.5
1,1,2-Trichloroethane	79-00-5	1	NY AWQS	2.5	0.7	2.5
Tetrachloroethene	127-18-4	5	NY AWQS	2.5	0.7	2.5
Chlorobenzene	108-90-7	5	NY AWQS	1	0.0714	1
Trichlorofluoromethane	75-69-4	5	NY AWQS	2.5	0.7	2.5
1,2-Dichloroethane	107-06-2	0.6	NY AWQS	0.5	0.169	0.5
1,1,1-Trichloroethane	71-55-6	5	NY AWQS	2.5	0.7	2.5
Bromodichloromethane	75-27-4	50*	NY AWQS	0.5	0.175	0.5

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
trans-1,3-Dichloropropene	10061-02-6	0.4	NY AWQS	2.5	0.7	2.5
cis-1,3-Dichloropropene	10061-01-5	0.4	NY AWQS	2.5	0.7	2.5
1,3-Dichloropropene, Total	542-75-6	0.4	NY AWQS	2.5	0.7	2.5
Bromoform	75-25-2	50*	NY AWQS	2.5	0.166	2.5
1,1,2,2-Tetrachloroethane	79-34-5	5	NY AWQS	2.5	0.7	2.5
Benzene	71-43-2	1	NY AWQS	2.5	0.7	2.5
Toluene	108-88-3	5	NY AWQS	2.5	0.7	2.5
Ethylbenzene	100-41-4	5	NY AWQS	2.5	0.7	2.5
Chloromethane	74-87-3	5	NY AWQS	2.5	0.7	2.5
Bromomethane	74-83-9	5	NY AWQS	2.5	0.7	2.5
Vinyl chloride	75-01-4	2	NY AWQS	5	1	5
Chloroethane	75-00-3	5	NY AWQS	5	1.46	5
1,1-Dichloroethene	75-35-4	5	NY AWQS	5	1	5
trans-1,2-Dichloroethene	156-60-5	5	NY AWQS	5	1.94	5
Trichloroethene	79-01-6	5	NY AWQS	5	1	5
1,2-Dichlorobenzene	95-50-1	3	NY AWQS	5	1	5

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
1,3-Dichlorobenzene	541-73-1	3	NY AWQS	2.5	0.7	2.5
1,4-Dichlorobenzene	106-46-7	3	NY AWQS	2	0.65	2
Methyl tert butyl ether	1634-04-4	10*	NY AWQS	2.5	0.7	2.5
p/m-Xylene	179601-23-1	5	NY AWQS	2.5	0.7	2.5
o-Xylene	95-47-6	5	NY AWQS	2.5	0.7	2.5
Xylene (Total)	1330-20-7	NE	N/A	2.5	0.7	2.5
cis-1,2-Dichloroethene	156-59-2	5	NY AWQS	2.5	0.7	2.5
1,2-Dichloroethene (total)	540-59-0	NE	N/A	2.5	0.7	2.5
Styrene	100-42-5	5	NY AWQS	2.5	0.7	2.5
Dichlorodifluoromethane	75-71-8	5	NY AWQS	2.5	0.7	2.5
Acetone	67-64-1	50*	NY AWQS	2.5	0.7	2.5
Carbon disulfide	75-15-0	60*	NY AWQS	2.5	0.7	2.5
2-Butanone	78-93-3	50*	NY AWQS	2.5	0.7	2.5
4-Methyl-2-pentanone	108-10-1	NE	N/A	250	60.8	250
2-Hexanone	591-78-6	50*	NY AWQS	0.5	0.144	0.5
Bromochloromethane	74-97-5	5	NY AWQS	2	0.65	2

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (µg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (µg/L)	RL (µg/L)
1,2-Dibromoethane	106-93-4	0.0006	NY AWQS	0.5	0.167	0.5
n-Butylbenzene	104-51-8	NE	N/A	0.5	0.159	0.5
sec-Butylbenzene	135-98-8	NE	N/A	2.5	0.7	2.5
tert-Butylbenzene	98-06-6	NE	N/A	2.5	0.7	2.5
1,2-Dibromo-3-chloropropane	96-12-8	0.04	NY AWQS	2.5	0.7	2.5
Isopropylbenzene	98-82-8	5	NY AWQS	2.5	0.7	2.5
p-Isopropyltoluene	99-87-6	5	NY AWQS	1	0.0714	1
n-Propylbenzene	103-65-1	5	NY AWQS	2.5	0.7	2.5
1,2,3-Trichlorobenzene	87-61-6	5	NY AWQS	0.5	0.169	0.5
1,2,4-Trichlorobenzene	120-82-1	5	NY AWQS	2.5	0.7	2.5
1,3,5-Trimethylbenzene	108-67-8	5	NY AWQS	0.5	0.175	0.5
1,2,4-Trimethylbenzene	95-63-6	5	NY AWQS	2.5	0.7	2.5
1,4-Dioxane	123-91-1	0.35	NYSDEC Interim Guidance	2.5	0.7	2.5

Notes:

[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).

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Worksheet #15-42: Reference Limits and Evaluation Table – Total Cyanide in Water

Matrix: Aqueous
Analytical Method: 9012B – Cyanide, Total

Analyte	Chemical Abstracts Service (CAS) Number	Project Action Limit (PAL)[1] (mg/L)	PAL Reference[2]	Project Quantification Limit	Achievable Laboratory Limits	
					MDL (mg/L)	RL (mg/L)
Cyanide, Total	57-12-5	0.2	NY AWQS	0.005	0.0018	0.005

Notes:
[1] Based on Project Action Limits defined in the 2022 Site Characterization Report (GEI 2022).
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MDL = Method Detection Limit; RL = Reporting Limit; mg/kg = milligrams per kilogram; µg/kg = micrograms per kilogram; PAL = project action limit; RSL = regional screening level; CAS = chemical abstract service; NYS AWQS = New York State Ambient Water Quality Standards; PFAS = per- and polyfluoroalkyl substance; NE = not established; N/A = not applicable.

Worksheet #16: Project Schedule/Timeline Table

Schedules for specific phases of work will be provided in Work Plans and Field Sampling Plans for the Site.

Activity	Responsible Party	Planned Start Date	Planned Completion Date	Deliverable(s)	Deliverable Due Date
Mobilization	GEI	5/19/2025	7/1/2025	N/A	N/A
Investigation Field Work	GEI	5/19/2025	12/31/2025	RI Data Submission Report	6/1/2026
Interim Meeting	GEI	4th Wednesday of each month	11/30/2027	N/A	N/A
Preparation of Report	GEI	8/1/2025	11/1/2027	RI/FS Report	11/15/2027

Worksheet #17: Sampling Design and Rationale

GEI has designed the following sampling program to meet the objectives outlined in Worksheet #11. Tasks outlined below will be completed during RI implementation, based on the project Schedule outlined in Worksheet #16.

Subsurface Soil Investigation

Soil borings will be completed at locations shown on Figures 5 through 7 of the RI Work Plan and summarized in Table 1 of the RI/FS Work Plan to evaluate the lateral and vertical extent of DNAPL presence in the subsurface soil. A total of 28 borings are identified as either Primary or Contingency borings. Primary borings will be completed to refine the extent of known DNAPL presence and address other objectives as identified in Table 1 of the RI/FS Work Plan. If DNAPL is identified at the primary boring location, a contingency boring will be completed at a "stepped out location" to satisfy the objective of mapping the extent of DNAPL.

Primary and Secondary Soil Borings

Including primary borings to be converted to monitoring wells, twenty-one (21) primary borings (inclusive of 2 creek borings T26A and T26B to evaluate Source Control) will be completed during the investigation. As shown on Figures 5 through 7 in the RI Work Plan, groundwater monitoring wells will be installed in seven of the soil borings. As many as seven (7) contingency borings may be added to step-out locations if needed to satisfy investigation objectives in the Scajaquada Creek OU. Boring termination depth for Primary and Secondary borings are summarized in Table 1 of the RI/FS Work Plan.

Groundwater Investigation

The goal of the groundwater investigation is to assess groundwater quality in the unconfined water-bearing zone where DNAPL is known to exist. In areas where DNAPL presence is to be investigated, well screens will be positioned to monitor the bottom of the alluvial soils directly above the area-wide clay layer which forms a barrier to vertical downward migration of DNAPL. To achieve this objective, five (5) new monitoring wells (MW-RI-1 through MW-RI-5) will be installed at the proposed locations shown on Figures 5, 6, and 7 of the RI/FS Work Plan to supplement the existing network of monitoring wells in the Lower Scajaquada Creek OU. Each well will be constructed using 2-inch diameter Schedule 40 PVC with a screened section consisting of 0.02-inch sized slow opening.

The bottom of the well will terminate in the area-wide red-brown clay layer. Wells monitoring the top of the clay layer will be equipped with a 2-foot DNAPL collection sump extending into the area-wide clay layer below the well screen.

In areas where DNAPL-impacted soils were identified and shallow wells are not present, proposed monitoring wells MW-LSC-2S-RI and MW-LSC-4S-RI will be installed with well screens positioned to monitor the upper 5 feet of the saturated zone. These wells will have 5-foot screen lengths to assess groundwater quality in shallow groundwater near the water table.

Creek and Inlet Area Sediment Investigation

Inlet Creek Bank Surface Soil

Surface water sheens have historically (prior to IRMs) been observed on the creek bank due to daily fluctuations in surface water levels in the Inlet. When sheen was present on the surface water in the Inlet Area near the City of Buffalo Pumphouse, the sheen residue was observed to be deposited on the shoreline at the water/bank interface. Surface soil samples will be collected, after shallow soil probing, from the creek bank at 10 locations identified on Figure 8 of the RI/FS Work Plan.

Sediment Probing Transects

Systematic sediment probing will be performed to assess the thickness and quality of sediment within the investigation area and observe sheen for generation. Probing will be conducted along 26 transects, spaced approximately 50 to 100 feet apart, as shown on Figures 8 through 10 of the RI/FS Work Plan. The endpoints of each transect, each probing location along the transect, and the water surface elevation will be established using survey-grade GPS.

Sediment Sampling

Sediment borings will be completed in the Scajaquada Creek main channel and Inlet along 26 transects to evaluate sediment quality. Target sediment sampling locations are provided on Figures 8 through 10 of the RI/FS Work Plan. The final location of each sediment boring may be adjusted in the field based on the presence of utilities buried beneath the Creek channel, accessibility with the sampling barge, obstructions (rip-rap, large debris), or if poor sample recovery occurs. Sediment sample locations will generally be collected at the nearest (relative to target locations on Figures 8 through 10 of the RI/FS Work Plan) 10-foot probing locations along each transect, and the sediment sampling effort will replace the physical proving at those locations.

The target intervals for sampling are generally the upper two feet of sediment and the most impacted interval observed in the core. In the event the 0- to 2-foot depth interval is also the most visibly impacted, or if no depth interval appears more impacted than others, the second sample will be collected from the 2-foot interval overlying the red-brown clay. Each sediment boring will be advanced a minimum of 4 feet into the area-wide red-brown clay layer which occurs more than 15 feet below the soft sediment surface.

Sediment Analysis – COC Delineation Samples

As indicated on Figures 8 through 10 of the RI Work Plan, there are 29 locations where analyses target a list of COCs potentially affecting the Lower Scajaquada Creek area based on the findings of prior investigations in and near the creek. The primary purpose of these samples is to delineate the nature and extent of impacts. At each of those locations, two sample intervals are proposed for analysis, totaling 58 samples. Of those, 47 are proposed for analysis of the focused COC Parameters analyte list and 11 are proposed for analysis of an expanded Full Suite COC analyte list. The analytes associated with each list are indicated in Table 4 of the RI/FS Work Plan.

Sediment Analysis – Benthic Toxicity Analysis

Sediment samples will be collected from the uppermost six inches of sediment (i.e., the most biologically active zone) at a subset of 26 of the 29 locations where COC-related sampling is planned. In addition to collecting COC-related information at these locations, additional parameters will also be analyzed to better assess other factors that may affect benthic toxicity apart from identified COCs. The additional analytical parameters at these locations are indicated in Table 4 of the RI/FS Work Plan.

In addition, sediment will be collected from the upper six inches of sediment at three locations for the purpose of laboratory toxicity testing. The three target locations include T9B, T15B, and T26B (Figures 8 through 10 of the RI/FS Work Plan), although adjustments may be made based on the results of the Ecological Risk Assessment Parameters analysis indicated above.

Waste Characterization

The evaluation of waste management costs in the FS requires evaluation of chemistry characteristics for sediment that may be dredged from the creek and subject to offsite disposal. Three sediment boring locations are proposed for collection of waste characterization samples. These locations (T2B, T6B, and T23A; Figures 8 and 10 of the RI/FS Work Plan) focus on the Inlet area where the greatest volume of deposition sediment is anticipated and where prior data suggests the majority of chemical impacts are present. These

analyses, combined with the suite of area-wide chemical tests, will allow for evaluation of both landfill and thermal treatment options. Analyses for waste characterization will be performed on composite samples created from the uppermost 4 feet or full thickness of depositional material retrieved at each location, whichever is less. One composite sample will be collected at each of the three locations; three samples in total will be analyzed for waste characterization parameters.

Geotechnical

The evaluation of certain remedial technologies for the creek as part of the planned FS may require an assessment of the effects of each technology on the stability of roads and structures along the creek. Knowledge of geotechnical properties may also be pertinent to designing support facilities, sediment dewatering, excavation support, and other ancillary activities that may be needed to implement certain remedial technologies. To support this assessment, limited geotechnical testing is proposed at six locations as part of the sediment investigation scope. The six locations were selected to be adjacent to structures that may be most susceptible to being destabilized by certain remedial activities, and focus on the following areas:

- Along the bulkhead supporting highway I-190 and closing the former creek outlet (T2A, T3A, and T5A)
- Between the State Route 198 on-ramp and Niagara Street bridge (T16B)
- Adjacent to the West Avenue bridge (T26A and T26B)

Soil Boring and Monitoring Well Rationale Table

Location	Primary/Contingency*	Objective/Rationale	Boring Termination Depth
Primary Borings/Monitoring Wells			
MW-RI-1	Primary	Groundwater Quality/Verification of Source Control and Evaluate Extent of DNAPL.	AWRBCL
MW-RI-2	Primary	Groundwater Quality/Verification of Source Control and Evaluate Extent of DNAPL (combine boring information from Creek Sediment Investigation Transect T-26).	AWRBCL
MW-RI-3	Primary	Groundwater Quality/Evaluate Extent of DNAPL/Geotechnical Data (destroyed 31 Tonawanda well MW-4R).	AWRBCL
MW-RI-4	Primary	Groundwater Quality/Evaluate Extent of DNAPL and Assess Potential for Northern Migration Pathway/Geotechnical Data.	AWRBCL
MW-RI-5	Primary	Groundwater Quality/Evaluate Extent of DNAPL and Assess Potential Historic Migration Pathway (former Scajaquada Creek outlet).	AWRBCL
MW-LSC-2S-RI	Primary	Evaluate groundwater quality in upper saturated zone above DNAPL impacted soil.	Screen upper 5 feet of Saturated Zone (Elevation 565 ft)
W-LSC-4S-RI	Primary	Evaluate groundwater quality in upper saturated zone above DNAPL impacted soil.	Screen upper 5 feet of Saturated Zone (Elevation 565 ft)
Soil Borings			
T26A	Primary	Creek Boring – Evaluation of Source Control and assess extent of DNAPL (temporary well install/testing for potential mobility).	AWRBCL
T26B	Primary	Creek Boring – Evaluation of Source Control and assess extent of DNAPL (temporary well install/testing for potential mobility).	AWRBCL
SB-RI-1	Contingency	Evaluation of Source Control and assess extent of DNAPL/Step-out location if MW-RI-1 impacts are observed.	AWRBCL

Location	Primary/Contingency*	Objective/Rationale	Boring Termination Depth
SB-RI-2	Primary	Evaluate Source Control and assess extent of DNAPL. If DNAPL encountered install well to assess mobility.	AWRBCL
SB-RI-3	Contingency	Evaluation of extent DNAPL.	AWRBCL
SB-RI-4	Contingency	Evaluation of extent if DNAPL is observed at MW-RI-2.	AWRBCL
SB-RI-5	Primary	Evaluate extent of DNAPL.	AWRBCL
SB-RI-6	Contingency	Evaluation of extent if DNAPL is observed at SB-RI-5.	AWRBCL
SB-RI-7	Primary	Evaluate extent of DNAPL (install well if alluvial soils are identified and contain DNAPL).	AWRBCL
SB-RI-8	Contingency	Evaluation of extent if DNAPL is observed at SB-RI-7.	AWRBCL
SB-RI-9	Primary	Evaluate extent of DNAPL – (install well if alluvial soils are identified and contain DNAPL) Complete Evaluation of Sand below AWRBCL if no DNAPL.	BCL
SB-RI-10	Contingency	Evaluation of extent if DNAPL is observed at SB-RI-9.	AWRBCL
SB-RI-11	Primary	Evaluate Extent of DNAPL – Complete Evaluation of Sand below AWRBCL.	BCL
SB-RI-12	Contingency	Evaluation of extent if DNAPL is observed at MW-RI-5 in Historic Creek Outlet.	AWRBCL
SB-RI-13	Primary	Evaluate Northern Extent of DNAPL near Historic Creek Outlet.	AWRBCL
SB-RI-14	Primary	Assess Potential for Northern Migration Pathway.	AWRBCL
SB-RI-15	Primary	Assess Potential Migration Pathway toward Black Rock Canal.	AWRBCL
SB-RI-16	Primary	Assess Potential Migration Pathway toward Black Rock Canal.	AWRBCL
SB-RI-17	Primary	Evaluate shallow impact at MW-LSC-3	AWRBCL
SB-RI-18	Primary	Evaluate Extent of DNAPL.	AWRBCL
SB-RI-19	Primary	Evaluate Extent of DNAPL.	AWRBCL

Notes:
1. AWRBCL – Area-wide red-brown clay layer at assumed elevation between elevation 550 and 558 ft. Boring will be terminated at a minimum depth of 4 feet into the clay: beyond any visual evidence of DNAPL.
2. BCL – Below Clay Layer (AWRBCL): Boring will completely penetrate AWRBCL until till layer above bedrock encountered.

*Primary wells and borings to be completed during the RI, Contingency boring locations to completed if RI objective is not satisfied by Primary.

Sediment Investigation Transects and Objectives

Sample Location	DNAPL Delineation		Sediment-related objectives targeted							FS Support	Source Control Evaluation	Notes/Comments
	Vertical NAPL Delin (Visual)	Horizontal NAPL Delin (Visual)	Sed Vol / Thickness (Visual)	Vert Extent of Contamination (Analytical)	Eco Risk Assessment (Analytical)	Benthic Tox	Geotech Properties	Waste Profiling	Migration Pathway Eval			
Transects 1-26 Probing	X	X	X						X	X		Exploratory probing using a PVC pipe or steel rod to assess for sheen along transect.
T1A	X	X	X	X						X		
T1B	X	X	X	X						X		
T2A	X	X	X	X			X			X		
T2B	X	X	X	X	X			X		X		Benthic toxicity &/or waste profile samples in Inlet may be collected from two depth intervals
T2C	X	X	X							X		
T3A	X	X	X				X			X		
T3B	X	X	X							X		
T4A	X	X	X	X	X					X		
T4B	X	X	X							X		
T5A	X	X	X				X			X		
T5B	X	X	X							X		
T6A	X	X	X							X		
T6B	X	X	X	X	X			X		X		
T7A	X	X	X							X		
T7B	X	X	X							X		
T8A	X	X	X	X	X					X		
T8B	X	X	X							X		
T9A	X	X	X	X	X					X		
T9B	X	X	X			X				X		
T10A	X	X	X							X		

Sample Location	DNAPL Delineation		Sediment-related objectives targeted							FS Support	Source Control Evaluation	Notes/Comments
	Vertical NAPL Delin (Visual)	Horizontal NAPL Delin (Visual)	Sed Vol / Thickness (Visual)	Vert Extent of Contamination (Analytical)	Eco Risk Assessment (Analytical)	Benthic Tox	Geotech Properties	Waste Profiling	Migration Pathway Eval			
T10B	X	X	X							X		
T11A	X	X	X	X	X					X		
T11B	X	X	X	X	X					X		
T12A	X	X	X	X	X					X		
T12B	X	X	X							X		
T12C	X	X	X							X		
T13A	X	X	X	X	X					X		
T13B	X	X	X	X	X					X		
T14A	X	X	X	X	X					X		
T14B	X	X	X							X		
T14C	X	X	X	X	X					X		
T15A	X	X	X	X	X					X		
T15B	X	X	X	X	X	X				X		
T16A	X	X	X	X	X					X		
T16B	X	X	X	X	X		X			X		
T17A	X	X	X	X	X					X		
T17B	X	X	X							X		
T18B	X	X	X	X	X					X		
T18A	X	X	X							X		
T19A	X	X	X	X	X					X		
T19B	X	X	X	X	X					X		
T20A	X	X	X							X		
T21A	X	X	X	X	X					X		
T22A	X	X	X							X		
T22B	X	X	X							X		
T23A	X	X	X					X		X		
T23B	X	X	X	X	X					X		
T24A	X	X	X	X	X					X		
T24B	X	X	X							X		
T25A	X	X	X							X		
T25B	X	X	X	X	X					X		
T26A	X	X	X	X	X		X			X	X	Boring supports assessment of Source Control
T26B	X	X	X	X	X	X	X			X	X	Boring supports assessment of Source Control

Worksheet #18: Sampling Locations and Methods

Soil/Groundwater/NAPL

The following table summarizes samples to be collected for soil, groundwater, and NAPL.

Matrix	Parameter	Analytical Method		Method	Target Sample Interval*	Sampling SOP
Subsurface Soil Samples	TCL VOCs	EPA SW	8260D/5035	Grab	A	SM-001 SM-002 SM-003
	PAH-17	EPA SW	8270E			
	COC Metals (arsenic, copper, lead, mercury, nickel, and zinc)	EPA SW	6020B/7471B			
	PCBs	EPA SW	8082A			
	Grain Size analysis, Standard Penetration Testing (SPT), TOC	ASTM D422 – sieve & hydrometer, D1586 SPT, TOC – 9060A/Lloyd Kahn				
Creek Bank Surface Soil Samples	TCL VOCs	EPA SW	8260D/5035	Grab	A	SM-001 SM-002 SM-003
	PAH-17	EPA SW	8270E			
	PCBs	EPA SW	8082A			
	COC Metals (arsenic, copper, lead, mercury, nickel, and zinc)	EPA SW	6020B/7471B			
Groundwater Samples (Monitoring Wells)	TCL VOCs	EPA SW	8260D	Grab	Screen interval	GW-001 GW-003 GW-004 GW-005 GW-006 GW-007
	TCL SVOCs	EPA SW	8270E			
	Total Cyanide	EPA SW	9012B			
	TAL Metals	EPA SW	6020B/7470A			
	1,4 Dioxane	EPA SW	8270E SIM			
	PCBs	EPA SW	8082A			
	PFAS	EPA SW	1633			
Groundwater Samples (City of Buffalo Pumphouse/Conveyance Piping)	TCL VOCs	EPA SW	8260D	Grab	B	
	TCL SVOCs	EPA SW	8270E			
	Total Cyanide	EPA SW	9012B			
	TAL Metals	EPA SW	6020B/7470A			
	1,4 Dioxane	EPA SW	8270E SIM			
	PCBs	EPA SW	8082A			
	PFAS	EPA SW	1633			
NAPL Samples	Forensic Analysis	EPA SW	8270E/8015D(M)	Grab	C	GW-002

***Target Sample Interval:**

A – A minimum of two soil samples will be collected from each boring. A sample will be collected from the most apparently impacted interval based on PID screening and field observations. If impacts are not observed, a sample will be collected from the 1-foot interval immediately below the water table, and a second sample will be collected from the bottom of the boring (presumed to be from the upper red-brown clay unit).

B – Water samples will be collected directly from Manholes 21 and 23 and the Pumphouse discharge.

C – If LNAPL or DNAPL is encountered in any soil boring exhibiting properties inconsistent with prior findings of NAPL (characteristics of coal-tar-like gross contamination), a sample will be collected.

Sediment

The following table summarizes samples to be collected for sediment.

Matrix	Sample Location	Parameter	Analytical Method	Visual Assessment/Characterization	Method[1]	Target Sample Interval*	Sampling SOP
Sediment	T1A	COC Parameters	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn	X	Macrocore sampler or 2" diameter split spoon samplers	A	SM-001 SM-002 SM-003
Sediment	T1B	COC Parameters	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn	X	Macrocore sampler or 2" diameter split spoon samplers	A	SM-001 SM-002 SM-003
Sediment	T2A	COC Parameters Geotechnical Parameters	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B Grain Size – ASTM D422 TOC – 9060A/Lloyd Kahn Atterberg Limits Shelby Tubes	X	Macrocore sampler or 2" diameter split spoon samplers	A, B	SM-001 SM-002 SM-003
Sediment	T2B	COC Parameters Eco Risk Assessment Waste Characterization	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422 TCLP VOCs – 8260D TCLP SVOCs – 8270E TCLP Metals – 6020B/7470A Pesticides/Herbicides – 8081B/8151A PCBs – 8082A pH – ignitability, sulfide/cyanide, reactivity – 9045/7.3 Corrosivity – 9045 TPH-DRO/TPH-GRO – 8015D(M) Percent Sulfur – ASTM D4239 BTU – ASTM D240-87	X	Macrocore sampler or 2" diameter split spoon samplers	A, B, C, D	SM-001 SM-002 SM-003

Matrix	Sample Location	Parameter	Analytical Method	Visual Assessment/Characterization	Method[1]	Target Sample Interval*	Sampling SOP
			EPH by MassDEP Method				
Sediment	T2C	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T3A	Geotechnical Parameters	Grain Size – ASTM D422 TOC – 9060A/Lloyd Kahn Atterberg Limits Shelby Tubes	X	Macrocore sampler or 2" diameter split spoon samplers	B	SM-001 SM-002 SM-003
Sediment	T3B	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T4A	COC Parameters Full Suite of COCs Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B PAH 34 – 8270E SIM PCBs – 8082A TAL Metals – 6010D/7471B Total Cyanide – 9012B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422 EPH by MassDEP Method	X	Macrocore sampler or 2" diameter split spoon samplers	A, C, E	SM-001 SM-002 SM-003
Sediment	T4B	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T5A	Geotechnical Parameters	Grain Size – ASTM D422 TOC – 9060A/Lloyd Kahn Atterberg Limits Shelby Tubes	X	Macrocore sampler or 2" diameter split spoon samplers	B	SM-001 SM-002 SM-003
Sediment	T5B	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T6A	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T6B	COC Parameters	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B	X	Macrocore sampler or 2" diameter split spoon samplers	A, C, D	SM-001 SM-002 SM-003

Matrix	Sample Location	Parameter	Analytical Method	Visual Assessment/Characterization	Method[1]	Target Sample Interval*	Sampling SOP
		Eco Risk Assessment Waste Characterization	TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422 TCLP VOCs – 8260D TCLP SVOCs – 8270E TCLP Metals – 6010D/6020B/7471B/7470A Pesticides/Herbicides – 8081B/8151A PCBs – 8082A pH – ignitability, sulfide/cyanide, reactivity – 9045/7.3 Corrosivity – 9045 TPH-DRO/TPH-GRO – 8015D(M) Percent Sulfur – ASTM D4239 BTU – ASTM D240-87 EPH by MassDEP Method				
Sediment	T7A	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T7B	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T8A	COC Parameters Full Suite of COCs Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B PAH 34 – 8270E SIM PCBs – 8082A TAL Metals – 6010D/7471B Total Cyanide – 9012B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422 EPH by MassDEP Method	X	Macrocore sampler or 2" diameter split spoon samplers	A, C, E	SM-001 SM-002 SM-003
Sediment	T8B	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003

Matrix	Sample Location	Parameter	Analytical Method	Visual Assessment/Characterization	Method[1]	Target Sample Interval*	Sampling SOP
Sediment	T9A	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T9B	COC Parameters Full Suite of COCs Eco Risk Assessment Benthic Toxicity	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B PAH 34 – 8270E SIM PCBs – 8082A TAL Metals – 6010D/7471B Total Cyanide – 9012B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422 28-day whole-sediment tox test EPH by MassDEP Method	X	Macrocore sampler or 2" diameter split spoon samplers	A, C, E, F	SM-001 SM-002 SM-003
Sediment	T10A	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T10B	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T11A	COC Parameters Full Suite of COCs Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B PAH 34 – 8270E SIM PCBs – 8082A TAL Metals – 6010D/7471B Total Cyanide – 9012B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422 EPH by MassDEP Method	X	Macrocore sampler or 2" diameter split spoon samplers	A, C, E	SM-001 SM-002 SM-003
Sediment	T11B	COC Parameters Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH	X	Macrocore sampler or 2" diameter split spoon samplers	A, C	SM-001 SM-002 SM-003

Matrix	Sample Location	Parameter	Analytical Method	Visual Assessment/Characterization	Method[1]	Target Sample Interval*	Sampling SOP
			Sulfide – 9030B AVS/SEM Grain Size – ASTM D422				
Sediment	T12A	COC Parameters Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C	SM-001 SM-002 SM-003
Sediment	T12B	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T12C	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T13A	COC Parameters Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C	SM-001 SM-002 SM-003
Sediment	T13B	COC Parameters Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C	SM-001 SM-002 SM-003
Sediment	T14A	COC Parameters Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C	SM-001 SM-002 SM-003

Matrix	Sample Location	Parameter	Analytical Method	Visual Assessment/Characterization	Method[1]	Target Sample Interval*	Sampling SOP
Sediment	T14B	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T14C	COC Parameters Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C	SM-001 SM-002 SM-003
Sediment	T15A	COC Parameters Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C	SM-001 SM-002 SM-003
Sediment	T15B	COC Parameters Eco Risk Assessment Benthic Toxicity	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422 28-day whole-sediment tox test	X	Macrocore sampler or 2" diameter split spoon samplers	A, C, F	SM-001 SM-002 SM-003
Sediment	T16A	COC Parameters Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C	SM-001 SM-002 SM-003
Sediment	T16B	COC Parameters Full Suite of COCs	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B PAH 34 – 8270E SIM PCBs – 8082A	X	Macrocore sampler or 2" diameter split spoon samplers	A, B, C, E	SM-001 SM-002 SM-003

Matrix	Sample Location	Parameter	Analytical Method	Visual Assessment/Characterization	Method[1]	Target Sample Interval*	Sampling SOP
		Eco Risk Assessment Geotechnical Parameters	TAL Metals – 6010D/7471B Total Cyanide – 9012B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B EPH by MassDEP Method AVS/SEM Grain Size – ASTM D422 Atterberg Limits Shelby Tubes				
Sediment	T17A	COC Parameters Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C	SM-001 SM-002 SM-003
Sediment	T17B	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T18B	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T18A	COC Parameters Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C	SM-001 SM-002 SM-003
Sediment	T19A	COC Parameters Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C	SM-001 SM-002 SM-003

Matrix	Sample Location	Parameter	Analytical Method	Visual Assessment/Characterization	Method[1]	Target Sample Interval*	Sampling SOP
Sediment	T19B	COC Parameters Full Suite of COCs Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B PAH 34 – 8270E SIM PCBs – 8082A TAL Metals – 6010D/7471B Total Cyanide – 9012B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B EPH by MassDEP Method AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C, E	SM-001 SM-002 SM-003
Sediment	T20A	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T21A	COC Parameters Full Suite of COCs Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B PAH 34 – 8270E SIM PCBs – 8082A TAL Metals – 6010D/7471B Total Cyanide – 9012B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B EPH by MassDEP Method AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C, E	SM-001 SM-002 SM-003
Sediment	T21B	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T22A	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T22B	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T23A	Waste Characterization	TCL/TCLP VOCs – 8260D TCL/TCLP SVOCs – 8270E TCL/TCLP Metals – 6010D/7471B Pesticides/Herbicides – 8081B/8151A PCBs – 8082A	X	Macrocore sampler or 2" diameter split spoon samplers	D	SM-001 SM-002 SM-003

Matrix	Sample Location	Parameter	Analytical Method	Visual Assessment/Characterization	Method[1]	Target Sample Interval*	Sampling SOP
			pH – ignitability, sulfide/cyanide, reactivity – 9045/7.3 Corrosivity – 9045 TPH-DRO/TPH-GRO – 8015D(M) Percent Sulfur – ASTM D4239 BTU – ASTM D240-87				
Sediment	T23B	COC Parameters Full Suite of COCs Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B PAH 34 – 8270E SIM PCBs – 8082A TAL Metals – 6010D/7471B Total Cyanide – 9012B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B EPH by MassDEP Method AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C, E	SM-001 SM-002 SM-003
Sediment	T24A	COC Parameters Full Suite of COCs Eco Risk Assessment	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B PAH 34 – 8270E SIM PCBs – 8082A TAL Metals – 6010D/7471B Total Cyanide – 9012B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B EPH by MassDEP Method AVS/SEM Grain Size – ASTM D422	X	Macrocore sampler or 2" diameter split spoon samplers	A, C, E	SM-001 SM-002 SM-003
Sediment	T24B	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T25A	N/A	N/A	Visual Assessment/Characterization only	Macrocore sampler or 2" diameter split spoon samplers		SM-001 SM-002 SM-003
Sediment	T25B	COC Parameters Full Suite of COCs	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B PAH 34 – 8270E SIM PCBs – 8082A	X	Macrocore sampler or 2" diameter split spoon samplers	A, C, E	SM-001 SM-002 SM-003

Matrix	Sample Location	Parameter	Analytical Method	Visual Assessment/Characterization	Method[1]	Target Sample Interval*	Sampling SOP
		Eco Risk Assessment	TAL Metals – 6010D/7471B Total Cyanide – 9012B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B EPH by MassDEP Method AVS/SEM Grain Size – ASTM D422				
Sediment	T26A	COC Parameters Eco Risk Assessment Geotechnical Parameters	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B AVS/SEM Grain Size – ASTM D422 Atterberg Limits Shelby Tubes	X	Macrocore sampler or 2" diameter split spoon samplers	A, B, C	SM-001 SM-002 SM-003
Sediment	T26B	COC Parameters Full Suite of COCs Eco Risk Assessment Geotechnical Parameters Benthic Toxicity	TCL VOCs – 8260D PAH-17 – 8270E COC Metals – 6020B/7471B PAH 34 – 8270E SIM PCBs – 8082A TAL Metals – 6010D/7471B Total Cyanide – 9012B TOC – 9060A/Lloyd Kahn Ammonia – 4500NH3-BH Sulfide – 9030B EPH by MassDEP Method AVS/SEM Grain Size – ASTM D422 Atterberg Limits Shelby Tubes 28-day whole-sediment toxicity test	X	Macrocore sampler or 2" diameter split spoon samplers	A, B, C, E, F	SM-001 SM-002 SM-003

Target Sample Interval Key:
A: (0-6") samples and/or depth interval of interest = the deeper boring depth selected based on work plan criteria (approximate depth 5 to 10 feet below top of sediments).
B: Geotechnical parameters will be collected in clay.
C: Eco Risk Assessment will be collected in (0-6") samples.
D: Waste Characterization samples will be collected at the (0-4') interval or full depositional thickness of sediment, whichever is less.
E: Full Suite COC samples will be collected in (0-6") samples.
F: Benthic Toxicity samples will be collected in (0-6") samples.

Notes:

1. If sediment recovery is poor, alternate sampling methods may be used, as described in the FSP (Appendix B of the RI/FS Work Plan).

Worksheet #19, 30: Analytical SOP Requirements and Analytical Services Table

Laboratory: Pace Analytical/Alpha Analytical, **320 Forbes Blvd., Mansfield, MA** 02048-1806, Project Manager, 508-898-9220

- **Point of Contact:** Melissa Deyo

List required accreditations/certifications: National Environmental Laboratory Accreditation Program (NELAP)

Back-up Laboratory: N/A

Sample Delivery Method: Lab courier or third-party courier (FedEx, UPS, etc.)

Analyte/Analyte Group	Method/Standard Operating Procedure (SOP)	Container(s) (minimum sample size)[1]	Preservation	Preparation Holding Time[2]	Analytical Holding Time	Data Package Turnaround
Aqueous						
Total Metals	6020B/26797-4-224123-1	500mL plastic or glass container	2mL 1:1 Nitric Acid per liter of sample to pH ≤ 2, Room temperature or 4° ± 2°C	6 months	6 months	10-20 days
Mercury	7470A/25923-2-224115-1	500mL plastic or glass container	2mL 1:1 Nitric Acid per liter of sample to pH ≤ 2, Room temperature or 4° ± 2°C	28 days	28 days	10-20 days
Semivolatile Organic Compounds	8270E/31164-3-222810-1	Two 1L Amber Glass jar	Non-preserved, 4° ± 2°C	7 days	40 days	10-20 days
Polychlorinated Biphenyls	8082A/222809-2	Two 1L Amber glass bottle	Non-preserved, 4°C	7 days	40 days	10-20 days
1,4-Dioxane	8270E/2164-18-222793-1	Two 1L Amber Glass jar	Non-preserved, 4°C	7 days	40 days	10-20 days

Analyte/Analyte Group	Method/Standard Operating Procedure (SOP)	Container(s) (minimum sample size)[1]	Preservation	Preparation Holding Time[2]	Analytical Holding Time	Data Package Turnaround
PFAS	1633/4582-6-234829-4	Two 500 ml HDPE container with unlined plastic screw cap	Store at 0-6°C	28 days	28 days	10-20 days
Soil/Sediment						
Polynuclear Aromatic Hydrocarbons	8270E/32324-2-222806-1	Pre-cleaned, 250mL glass jar w/ Teflon lined screw cap	4° ± 2°C or -20° ± 5°C	14 days (Freezing extends hold time)	40 days	10-20 days
Semivolatile Organic Compounds	8270E/31164-3-222810-1	Glass jar w/Teflon lined screw cap	4° ± 2°C or -20° ± 5°C	14 days (Freezing extends hold time)	40 days	10-20 days
Total Metals	6010D/26797-4-224123-1	10g of sample in a glass jar	4° ± 2°C	6 months	6 months	10-20 days
COC Metals	6020B/26797-4-224123-1	10g of sample in a glass jar	4° ± 2°C	6 months	6 months	10-20 days
Total Mercury	7471B/7471-2139-7-224128-1	10g of sample in glass jar	4° ± 2°C	28 days	28 days	10-20 days
Polychlorinated Biphenyls	8082A/222809-2	4oz glass soil jars	4° ± 2°C or -20° ± 5°C	14 days (Freezing extends hold time)	40 days	10-20 days
Total Organic Carbon	9060A/2182-25-222834-2	Glass jar	4° ± 2°C	N/A	28 days	10-20 days
AVS-SEM	EPA-821-R-91-100/26797-4-224123-1	10g of sample in a glass jar	4° ± 2°C	14 days	14 days	10-20 days
Grain Size	ASTM D422-63/2183-10-222829-1	500g in a Ziploc bag	N/A	N/A	N/A	10-20 days
Pesticides	8081B/8081-2158-14-222800-1	Glass jar	4° ± 2°C or -20° ± 5°C	14 days (Freezing extends hold time)	40 days	10-20 days
Total Petroleum Hydrocarbons	8015D/8015-222811-2	Pre-cleaned 250mL soil jar	4°C	14 days	40 days	10-20 days

Notes:
1. Multiple analyses may be performed from the same container if the container and preservative are identical and sufficient sample volume is available.
2. Holding times begin starting from the collection date and time except for CLP methods which are based on Validated Time of Sample Receipt (VTSR).
SOP = standard operating procedure; N/A = not applicable; COC = site-specific contaminants of concern

Laboratory: Alpha Analytical, **8 Walkup Drive, Westborough, MA** 01581, Project Manager, 508-898-9220

- **Point of Contact:** Melissa Deyo

List required accreditations/certifications: NELAP

Back-up Laboratory: N/A

Sample Delivery Method: Lab courier or third-party courier (FedEx, UPS, etc.)

Analyte/Analyte Group	Method/Standard Operating Procedure (SOP)	Container(s) (minimum sample size)[1]	Preservation	Preparation Holding Time[2]	Analytical Holding Time	Data Package Turnaround
Aqueous						
Volatile Organic Compounds	EPA 8260D/32639-8-224558-1	2, 40-mL VOA vials w/ PTFE-faced silicone septum	4 ± 2°C 1:1 HCL to a pH of <2.	14 days	14 days	10-20 days
Semivolatile Organics	EPA 8270E/7471-2139-7-224128-1	2, 1-L amber glass jars	4 ± 2°C	7 days	40 days	10-20 days
Semivolatile Organics	EPA 625.1 / 31162-3-224534-1	2, 1-L amber glass jars	4 ± 2°C	7 days	40 days	10-20 days
Semivolatile Organics by SIM	EPA 8270E-SIM / 32637-4-224532-1	2, 1-L amber glass jars	4 ± 2°C	7 days	40 days	10-20 days
Polychlorinated Biphenyls	EPA 8082A / 8082-224529-2	2, 1-L amber glass jars	4 ± 2°C	7 days	40 days	10-20 days
Total Cyanide	9012B / 2210-14-224624-1	1, 250-mL plastic container, NaOH preserved	50% sodium hydroxide pH > 12, 4 ± 2°C	14 days	14 days	10-20 days
Soil/Sediment						
Volatile Organic Compounds-High Level	EPA 8260D/32639-8-224558-1	1, 40-mL vial with 15-mL of Methanol, 1, 40-mL vial without Methanol	15 mL of Methanol 4 ± 2°C	14 days	14 days	10-20 days

Analyte/Analyte Group	Method/Standard Operating Procedure (SOP)	Container(s) (minimum sample size)[1]	Preservation	Preparation Holding Time[2]	Analytical Holding Time	Data Package Turnaround
Volatile Organic Compounds-Low Level	EPA 8260D/32639-8-224558-1	2, 40-mL VOA vials with bisulfate solution or water	4 ± 2°C with bisulfate solution, < 0°C with water	14 days	14 days	10-20 days
Volatile Petroleum Hydrocarbons	MA VPH 2.1 / 2120-19-234838-1	2, 40-mL VOA vials, One unpreserved	Methanol 4 ± 2°C	N/A	28 days	10-20 days
Gasoline Range Organics	EPA 8015D(M) / 26801-6-224553	1, 40-mL VOA vial, MeOH preserved	Methanol 4 ± 2°C	14 days	14 days	10-20 days
Semivolatile Organics	EPA 8270E/31162-3-224534-1	1, 250-mL amber glass jar	4 ± 2°C	14 days	40 days	10-20 days
Semivolatile Organics by SIM	EPA 8270E-SIM / 32637-4-224532-1	1, 250-mL amber glass jar	4 ± 2°C	14 days	40 days	10-20 days
Pesticides	EPA 8081B / 8081-224528-2	1, 250-mL amber glass jar	4 ± 2°C	14 days	40 days	10-20 days
PCB	EPA 8082A / 8082-224529-2	1, 250-mL amber glass jar	4 ± 2°C	14 days	40 days	10-20 days
Herbicides	EPA 8151A / 2128-10-224526-1	1, 8-oz glass jar	4 ± 2°C	14 days	40 days	10-20 days
TPH Diesel Range Organics	EPA 8015D(M) / 2125-12-224535-1	1, 250-mL amber glass jar	4 ± 2°C	14 days	40 days	10-20 days
pH	EPA 9045D & 9040C / 2202-11-224615-1	1, 4-oz glass jar	4 ± 2°C	N/A	ASAP, within 24 hours	10-20 days
Total Cyanide	EPA 9012B / 2210-14-224624-1	1, 250-mL amber glass jar	4 ± 2°C	14 days	14 days	10-20 days
Percent Solids	SM 2540G / 2229-15-224583-1	1, 2-oz plastic container	4 ± 2°C	N/A	7 days	10-20 days
Total Sulfide	EPA 9030B / 2221-11-224632-1	1, 250-mL amber glass jar	2N zinc Acetate and 6N NaOH	N/A	7 days	10-20 days
Ignitability	EPA 1030 / 2238-6-224580-1	1, 250-mL amber glass jar	N/A	N/A	14 days	10-20 days
Reactive Sulfide & Cyanide	SW-846 Ch. 7 / 2228-6-224619-1	1, 250-mL amber glass jar	4 ± 2°C	N/A	14 days	10-20 days

Analyte/Analyte Group	Method/Standard Operating Procedure (SOP)	Container(s) (minimum sample size)[1]	Preservation	Preparation Holding Time[2]	Analytical Holding Time	Data Package Turnaround
Total Petroleum Hydrocarbons	EPA 8015D(M) /9071-9733-3-224610-1	1 8-oz glass jar	4 ± 2°C HCl	28 days	28 days	10-20 days
Ammonia	SM 4500NH3 -BH / 2206-12-224606-1	1, 250-mL amber glass jar	4 ± 2°C	N/A	28 days	10-20 days
EPH	MA-EPH / ENV-SOP-WEST-0040	1, 250 mL amber glass jar	4 ± 2°C	14 days	40 days	10-20 days

Notes:
1. Multiple analyses may be performed from the same container if the container and preservative are identical and sufficient sample volume is available.
2. Holding times begin starting from the collection date and time except for CLP methods which are based on Validated Time of Sample Receipt (VTSR).
SOP = standard operating procedure; N/A = not applicable; COC = site-specific contaminants of concern

Laboratory: Pace Analytical Services, LLC, **2225 Riverside Drive, Asheville, NC** 28804, Project Manager, 508-898-9220

- **Point of Contact:** Melissa Deyo

List required accreditations/certifications: NELAP

Back-up Laboratory: N/A

Sample Delivery Method: Lab courier or third-party courier (FedEx, UPS, etc.)

Analyte/Analyte Group	Method/Standard Operating Procedure (SOP)	Container(s) (minimum sample size)[1]	Preservation	Preparation Holding Time[2]	Analytical Holding Time	Data Package Turnaround
Soil/Sediment						
Calorific Value (BTU/lb)	ASTM Method D240-87/ ENV-SOP-ASHE-0015	Pre-cleaned 4/8 oz glass jar	0° to 6.0°C	N/A	N/A	10-20 days
Percent Sulfur	ASTM D4239/ ENV-SOP-ASHE-0009 v03_Sulfur	Pre-cleaned 4/8 oz glass jar	0° ± 6.0°C	N/A	N/A	10-20 days

Notes:
1. Multiple analyses may be performed from the same container if the container and preservative are identical and sufficient sample volume is available.

2. Holding times begin starting from the collection date and time except for CLP methods which are based on Validated Time of Sample Receipt (VTSR).
SOP = standard operating procedure; N/A = not applicable; COC = site-specific contaminants of concern

Worksheet #20: Field Quality Control Sample Summary Table

Matrix	Analytical Group	No. of Field Duplicate Pairs	No. of MS/MSD[1]	No. of Field Blanks	No. of Equipment Blanks[2]	No. of Trip Blank Samples	No. of Sampling Locations[3]	Total No. of Samples to Lab[3]
Soil/Sediment (Geotechnical)	Grain Size (ASTM D422 – Sieve and Hydrometer)	N/A	N/A	N/A	N/A	N/A	Footnote 3	Footnote 3
	TOC (9060A/Lloyd Kahn)	N/A	N/A	N/A	N/A	N/A	Footnote 3	Footnote 3
	Atterberg Limits	N/A	N/A	N/A	N/A	N/A	Footnote 3	Footnote 3
	Shelby Tubes (as applicable)	N/A	N/A	N/A	N/A	N/A	Footnote 3	Footnote 3
Soil (Analytical)	TCL VOCs (EPA Method 8260D/5035)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	PAH-16/17 (EPA Method 8270E)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	Metals (EPA Method 6010D/6020B/7471B)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	PCB-Aroclors (EPA Method 8082A)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	BTU (Calorific Value)	2 per 20	N/A	N/A	N/A	N/A	Footnote 3	Footnote 3
Sediment (Analytical)	TCL VOCs (EPA Method 8260D/5035)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	PAH-16/17 (EPA Method 8270E)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3

Matrix	Analytical Group	No. of Field Duplicate Pairs	No. of MS/MSD[1]	No. of Field Blanks	No. of Equipment Blanks[2]	No. of Trip Blank Samples	No. of Sampling Locations[3]	Total No. of Samples to Lab[3]
	Metals (EPA Method 6010D/6020B/7471B)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	PCB-Aroclors (EPA Method 8082A)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	TCL SVOCs Expanded NOAA PAH 34 (EPA Method 8270E – SIM)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	Total Cyanide (EPA Method 9012B)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	Ammonia (Method 4500NH3-BH)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	Sulfide (EPA Method 9030B)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	AVS/SEM	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	EPH (MassDEP Method)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
Groundwater (Analytical)	TCL VOCs (EPA Method 8260D)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	TCL SVOCs (EPA Method 8270E)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	Total Cyanide (EPA Method 9012B)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	TAL Metals (EPA Method 6020B/7470A)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3

Matrix	Analytical Group	No. of Field Duplicate Pairs	No. of MS/MSD[1]	No. of Field Blanks	No. of Equipment Blanks[2]	No. of Trip Blank Samples	No. of Sampling Locations[3]	Total No. of Samples to Lab[3]
	1,4-Dioxane (EPA Method 8270E SIM)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	PCBs (EPA Method 8082A)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
	PFAS (EPA Method 1633)	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3
NAPL	Forensic Analysis (EPA Method 8270E/8015D(M))	1 per 10	1 per 20	1 per 20	1 per 20	N/A	Footnote 3	Footnote 3

Notes:
1. Matrix spike (MS)/matrix spike duplicates (MSDs) will be client-provided where applicable. Laboratory may select project samples to be spiked for batch accuracy and precision. When no MS/MSDs are supplied, laboratory control sample (LCS)/laboratory control sample duplicates (LCSDs) will be performed.
2. One equipment blank per day or one per 20 samples, whichever is more frequent. Equipment blanks are not required when dedicated or disposable sampling equipment are utilized.
3. Number of sampling locations and samples will be determined by area or task in the field sampling plan.
Waste characterization samples are not included in the table due to no field QC samples bring proposed for these parameters.
N/A = not applicable; TBD = to be determined.

Worksheet #21: Project Sampling SOP References Table

SOP # or Reference[1]	Title, Revision, Date, and URL (if available)	Originating Organization	SOP Option or Equipment Type (if SOP provides options)	Modified for Project? Y/N	Comments
AR-001	General Guidance on Work Zone Monitoring Methods.	GEI	See SOP for specific equipment needs	N	Describes general guidance on work zone air monitoring methods.
AR-002	Air Sampling for Dust (Particulate Matter) Using the MIE DataRAM™ Real-Time Aerosol Monitor	GEI	MIE DataRAM™ model DR-2000 real-time aerosol monitor	N	Describes procedures for real-time monitoring of airborne particulate matter.
AR-005	Hydrogen Cyanide Work Zone Air Monitoring Procedures.	GEI	Handheld meter in conjunction with the Dräger Chip Measuring System (CMS). V-RAE or Mini-Warn used in conjunction with CMS Dräger tubes for continuous monitoring.	N	Describes real-time monitoring of hydrogen cyanide gas.
AR-006	Air Sampling for Volatile Organic Compounds (VOCs) Using Summa Canisters.	GEI	See SOP for specific equipment needs	N	Describes collection of ambient air samples for VOC analysis using Summa canisters. Typically, U.S. EPA Method TO-15 for laboratory analysis.
DM-001	General Guidance on Determination of Appropriate Drilling Methods.	GEI	See SOP for specific equipment needs	N	Describes guidance for determining appropriate drilling method with consideration of potential sample collection requirements.
DM-002	Hollow Stem Auger.	GEI	See SOP for specific equipment needs	N	Describes common hollow-stem auger drilling procedures for overburden soil borings.

SOP # or Reference[1]	Title, Revision, Date, and URL (if available)	Originating Organization	SOP Option or Equipment Type (if SOP provides options)	Modified for Project? Y/N	Comments
DM-003	Air Rotary Drilling with Casing.	GEI	See SOP for specific equipment needs	N	Not anticipated for project.
DM-004	Sonic Drilling.	GEI	See SOP for specific equipment needs	N	Not anticipated for project.
DM-006	GeoProbe® Direct Push Boring.	GEI	See SOP for specific equipment needs	N	Describes procedures for drilling overburden soil borings using Geoprobe® and Macro Core® technologies.
DM-007	Monitoring Well Construction and Installation.	GEI	See SOP for specific equipment needs	N	Describes procedures for installation of monitoring wells screened across or below groundwater table. Assumes wells will be constructed of flush-joint PVC pipe and screened section with factory-slotted openings.
DM-008	Telescoped Drilling	GEI	See SOP for specific equipment needs	N	Describe telescoped drilling used to prevent the connection of two stratigraphic layers during boring advancement and/or monitoring well installation.
DM-009	Monitoring Well Development.	GEI	See SOP for specific equipment needs	N	Describes procedures to develop monitoring wells and maximize groundwater movement.
FD-001	Field Notebook.	GEI	See SOP for specific equipment needs	N	Describes methods for documentation of field activities.
FD-002	Field Observation Report.	GEI	See SOP for specific equipment needs	N	Describes methods to generate a field observation report.
FD-003	Sample Management and Chain of Custody.	GEI	See SOP for specific equipment needs	N	Describes general methods of labelling and managing samples and chain-of-custody preparation.

SOP # or Reference[1]	Title, Revision, Date, and URL (if available)	Originating Organization	SOP Option or Equipment Type (if SOP provides options)	Modified for Project? Y/N	Comments
					Additionally addresses sample transport.
FD-004	Photo Documentation.	GEI	See SOP for specific equipment needs	N	Describes methods to document and retain photographic records.
FD-006	Handheld Global Positioning Receiver Operation.	GEI	See SOP for specific equipment needs	N	Describes procedures for conducting GPS surveys.
GW-001	Water Level and NAPL Measurement.	GEI	Water level meter or oil/water interface probe	N	Describes procedures to measure depth to water and NAPL.
GW-003	Low Flow (Low Stress) Groundwater Sampling.	GEI	See SOP for specific equipment needs	N	Describe methods to collect groundwater samples representative of aquifer conditions in wells exhibiting no continuous drawdown.
GW-004	pH and Temperature Measurement.	GEI	Combination pH/temperature meter	N	Describes methods to measure pH and temperature of liquids.
GW-005	Turbidity Measurement.	GEI	Hach nephelometer/ turbidimeter	N	Describes calibration and method to measure turbidity.
GW-006	Specific Conductance Measurement.	GEI	Field conductivity meter	N	Describes calibration and method to measure conductivity.
GW-007	Dissolved Oxygen Measurement.	GEI	Dissolved oxygen (DO) meter	N	Describes calibration and method to measure DO.
GW-008	Temporary Groundwater Sampling Points.	GEI	See SOP for specific equipment needs	N	Describes procedures for installation of temporary groundwater sampling points.
PM-001	Utility Mark Out and Clearance	GEI	See SOP for specific equipment needs	N	Describes utility mark out/clearance procedures.

SOP # or Reference[1]	Title, Revision, Date, and URL (if available)	Originating Organization	SOP Option or Equipment Type (if SOP provides options)	Modified for Project? Y/N	Comments
QA-001	Equipment Decontamination.	GEI	See SOP for specific equipment needs	N	Describes methods used to decontaminate core collection equipment and reusable sampling equipment for projects that require collection of organic and inorganic samples.
QA-002	Field Quality Control Samples.	GEI	See SOP for specific equipment needs	N	Describes procedures for field quality control (QC) samples.
SC-001	Environmental Sample Types and Sampling Strategies	GEI	See SOP for specific equipment needs	N	Describes sample types and strategic approaches to sample locations.
SC-002	Environmental Sample Handling.	GEI	See SOP for specific equipment needs	N	Describes procedures related to the collection and transportation of environmental samples.
SC-003	Investigation Derived Waste.	GEI	See SOP for specific equipment needs	N	Describes characterization and management of investigation derived waste resulting from investigation activities.
SC-004	Headspace VOC Screening.	GEI	Photoionization detector (PID) or flame ionization detector (FID)	N	Describes methods to measure total VOCs present in headspace.
SG-001	General Guidance on Soil Vapor Intrusion Evaluations.	GEI	See SOP for specific equipment needs	N	Describes methods used to evaluation soil vapor intrusion exposure pathways.
SG-002	Soil Vapor Sample Collection.	GEI	See SOP for specific equipment needs	N	Describes the general steps to collect soil vapor samples.
SG-003	Sub-slab Soil Vapor Collection.	GEI	See SOP for specific equipment needs	N	Describes general steps to collect sub-slab soil vapor samples.

SOP # or Reference[1]	Title, Revision, Date, and URL (if available)	Originating Organization	SOP Option or Equipment Type (if SOP provides options)	Modified for Project? Y/N	Comments
SG-004	Ambient Air Sample Collection.	GEI	See SOP for specific equipment needs	N	Describes method to collect ambient air samples.
SM-001	Soil Sampling Techniques Including Split-Spoon.	GEI	See SOP for specific equipment needs	N	Describes the standard procedure for the collection of surface and subsurface soil samples using traditional sampling methods.
SM-002	VOC Soil Collection and Preservation Method.	GEI	See SOP for specific equipment needs	N	Describes methods to collect and preserve soil samples for the analysis of VOCs in accordance with the EPA Method 5035.
SM-003	Classification of Soil Samples in the Field.	GEI	See SOP for specific equipment needs	N	Describes methods to classify soil samples collected in the field in a consistent manner.
SS-001	Sediment Sampling Using a Sediment Grab Sampler.	GEI	See SOP for specific equipment needs	N	Describes the use of sediment grab samplers for collection of surficial sediment.
SS-002	Sediment Sampling Using Vibracore Equipment.	GEI	See SOP for specific equipment needs	N	Describes collection of sediment samples using Vibracore methods.
SW-001	Surface Water Sampling.	GEI	See SOP for specific equipment needs	N	Describes methods to collect surface water samples.

Notes:
[1] GEI field standard operating procedures (SOPs) are presented in Appendix A of this QAPP. Revisions to GEI field sampling SOPs will be approved by the United States Environmental Protection Agency (EPA) prior to use.
Y = yes; N = no; GPS = global positioning system; ID = identification; SOP = standard operating procedure; VOC = volatile organic compound; QC = quality control.

Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection Table

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	SOP Reference	Frequency	Acceptance Criteria	Corrective Action	Responsible Person
Photo-ionization Detector	HASP and Operator's Manal	As per Operator's Manual	As per Operator's Manual	As per Operator's Manual	HASP and Operator's Manual	Daily and if instrument provides erratic results	N/A	Replace or re-calibrate	Field Team Leader
Particulate Meter	HASP and Operator's Manal	As per Operator's Manual	As per Operator's Manual	As per Operator's Manual	HASP and Operator's Manual	Daily and if instrument provides erratic results	N/A	Replace or re-calibrate	Field Team Leader
Multi-Gas Meter	HASP and Operator's Manal	As per Operator's Manual	As per Operator's Manual	As per Operator's Manual	HASP and Operator's Manual	Daily and if instrument provides erratic results	N/A	Replace or re-calibrate	Field Team Leader
GPS Unit	In accordance with manufacturer's instructions	As required by manufacturer specification	Coordinates at known benchmark	Batteries charged, acceptable memory	FD-006 – Handheld Global Positioning Receiver Operation.	Twice daily	+/- 0.5 foot horizontal; +/- 1 foot vertical	Replace or re-calibrate	Field Team Leader
Multi-probe water quality meter	In accordance with manufacturer's instructions	As required by manufacturer specification	As per Operator's Manual	As per Operator's Manual	Operator's Manual	Daily	pH ±0.3 with buffer solution true values; conductivity 10% of standard DO ±0.5 milligram per liter (mg/L) of saturation in air	Replace or re-calibrate	Field Team Leader

Field Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	SOP Reference	Frequency	Acceptance Criteria	Corrective Action	Responsible Person
Turbidity Meter	In accordance with manufacturer's instructions	As required by manufacturer specification	As per Operator's Manual	As per Operator's Manual	Operator's Manual	Daily	±10%	Replace or re-calibrate.	Field Team Leader
Oil/water interface probe and water level meter	In accordance with manufacturer's instructions	As required by manufacturer specification	As per Operator's Manual	As per Operator's Manual	Operator's Manual	Prior to use and if instrument provides erratic results	±10%	Replace or re-calibrate	Field Team Leader
Cell phone with camera	N/A	N/A	N/A	Batteries charged, acceptable memory and general camera function	FD-004 – Photo Documentation	N/A	N/A	N/A	Field Team Leader

SOP = standard operating procedure; N/A = not applicable; GPS = global positioning system; HASP = health and safety plan.

Worksheet #23: Analytical SOP References Table

SOP #[1]	Title	Date	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	Modified for Project? Y/N
2164-18-222793-1	1,4-Dioxane	1/12/2022	Definitive	Water/1,4-Dioxane	GC/MS	N
SIM-2247-12-222798	Analysis of Parent and Alkylated Polynuclear Aromatic Hydrocarbons, Selected Heterocyclic Compounds, Steranes, Triterpanes, and Triaromatic Steriods by GC/MS – SIM	11/1/2022	Definitive	Sediment/PAHs	GC/MS	N
2140-5-224120-1	Acid Volatile Sulfides and Simultaneously Extracted Metals in Sediments	1/7/2020	Definitive	Sediment/AVS-SEM	See SOP	N
D4318	Standard Test Method for Liquid Limit, Plastic Limit, and Plasticity Index of Soils	1/31/2018	Screening	Soil/Plastic Limits	See SOP	N
1633-305197-1	Preparation and Analysis of PFAS in Aqueous, Solid, and Tissue by EPA Method 1633	8/7/2024	Definitive	Soil, Sediment, Water/PFAS	LC-MS/MS	N
GTX-SVP-13	Atterberg Limit Device, Grooving Tool, Gauge, Glass Plate	10/31/2012	Definitive	Soil/Atterberg Limit	See SOP	N
2238-6-224580-1	Ignitability of Solids	10/20/2023	Definitive	Soil, Sediment/Ignitability	See SOP	N
26797-4-224114	Inductively Coupled Plasma – Mass Spectrometry 6020B	1/25/2022	Definitive	Soil, Sediment/Metals	ICP-MS	N
25923-2-224115	Mercury in Liquid Waste	1/13/2020	Definitive	Water/Mercury	Automated Cold Vapor	N
25924-4-224116	Mercury in Solid or Semisolid Waste	7/7/2022	Definitive	Soil, Sediment/Mercury	Manual Cold Vapor	N

SOP #[1]	Title	Date	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	Modified for Project? Y/N
26796-6-224118-1	Inductively Coupled Plasma – Atomic Emission Spectrometry	6/13/2023	Definitive	Water/Metals	ICP/AES	N
2206-12-224606-1	Nitrogen Ammonia	1/3/2022	Definitive	Water/Ammonia	See SOP	N
8081-224528-2	Organochlorine Pesticides by Capillary Column Gas Chromatography	1/6/2025	Definitive	Sediment, Soil/Pesticides	GC	N
2183-10-222829-1	Particle Size Analysis of Soils – with/without Hydrometer	10/25/2022	Definitive	Sediment, Soil/Particle Size	See SOP	N
8082-224529-2	PCBs	11/14/2024	Definitive	Sediment, Soil, Water/PCB	See SOP	N
2203-9-224577-1	pH, Soil and Waste	9/8/2023	Definitive	Sediment, Soil/pH	pH meter	N
32637-4-224532-1	PAHs by SIM EPA 8270E	7/13/2023	Definitive	Sediment, Soil, Water/PAH	GC/MS	N
2228-6-224619-1	Reactive Sulfide and Reactive Cyanide	1/24/2020	Definitive	Sediment, Soil/Reactivity	See SOP	N
31162-3-224534-1	Semivolatile Organic Compounds by GC/MS	7/13/2023	Definitive	Soil, Sediment, Water/SVOC	GC/MS	N
2133-19-224483-1	TCLP Extraction Metals and Semi-Volatile Organics	6/5/2023	Definitive	Soil, Sediment/TCLP Metals and SVOCs	See SOP	N
2112-12-224555-1	TCLP/SPLP Extraction – VOCs EPA 1311/1312	6/2/2023	Definitive	Soil, Sediment/TCLP VOCs	See SOP	N
2182-25-222834-2	TOC and Soot in Soil, Sediment	11/15/2024	Definitive	Soil, Sediment/TOC	CHNS/O Analyzer	N
2125-12-224535-1	TPH – Diesel Range Organics	7/27/2020	Definitive	Soil, Sediment, Water/TPH	GC	N
26801-6-224553	TPH – Gasoline Range Organics 8015 D	8/28/2023	Definitive	Soil, Sediment, Water/TPH	GC	N

SOP #[1]	Title	Date	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	Modified for Project? Y/N
32639-8-224558-1	VOC by GC/MS	11/8/2022	Definitive	Soil, Sediment, Water/VOC	GC/MS	N
ENV-SOP-ASHE-0015	Determination of Calorific Value (BTU/LB) of Waste Materials Using a Parr 6300/6400 Oxygen Bomb Calorimeter	12/14/2021	Definitive	Soil, Sediment/BTU	Calorimeter	N
ENV-SOP-ASHE-0009 v03	Sulfur by ASTM D4239	4/10/2023	Definitive	Soil, Sediment/Sulfur	Analyzer	N
2214-11-224593-1	Inorganic Anions by Ion Chromatography	2/2/2022	Definitive	Soil, Sediment, Water/Metals	See SOP	N
ENV-SOP-WES2-0062	Biochemical Oxygen Demand	1/7/2025	Screening	Water/BOD	See SOP	N
267100-1	Balance Calibration Verification	4/18/2024	Screening	Soil, Sediment, Water/Various	Balance	N
3743-3-224600-1	Free Cyanide	8/14/2015	Screening	Soil, Sediment, Water/Cyanide	See SOP	N
2204-11-224601-1	Hexavalent Chromium	2/16/2023	Screening	Soil, Sediment, Water/Metal	See SOP	N
224604-2	Nitrate, Nitrite, and Nitrate-Nitrite Nitrogen	10/17/2024	Definitive	Water/Metal	See SOP	N
2243-8-224605-1	Nitrite – Manual Colorimetric Method	5/3/2019	Definitive	Water/Metal	Spectrometer	N
2207-12-224607-1	Nitrogen, Total Kjeldahl	6/26/2023	Screening	Water/Metal	See SOP	N
2202-11-224615-1	pH, Liquid Samples	3/13/2019	Definitive	Water/pH	Electrode	N
2211-9-224582-1	Total Phenol	10/25/2023	Definitive	Soil, Water, Sediment/SVOC	See SOP	N

SOP #[1]	Title	Date	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	Modified for Project? Y/N
224621-2	Sulfate Turbidimetric Method	10/17/2024	Screening	Soil, Water, Sediment/Metal	Spectrophotometer	N
2210-14-224624-1	Total and Amenable Cyanide	3/17/2023	Definitive	Soil, Sediment, Water/CN	See SOP	N
2215-15-224626-1	TOC-DOC-TIC	4/4/2023	Definitive	Soil, Sediment, Water/TOC	TOC Analyzer	N
2226-12-224628-1	Total Phosphorus, Dissolved Phosphorus – Colorimetric	11/11/2021	Definitive	Soil, Sediment, Water/Metal	See SOP	N
2221-11-224632-1	Total Sulfide – Methylene Blue	1/13/2022	Screening	Water/Metal	See SOP	N
2237-4-224612-1	Oxidation – Reduction Potential	12/23/2015	Screening	Soil, Sediment, Water/ORP	Electrode	N
2230-9-224620-1	Specific Conductance and Salinity	2/23/2017	Screening	Soil, Sediment, Water/Wet Chem	See SOP	N
2208-15-224589-1	Chemical Oxygen Demand	¼/2022	Screening	Water/COD	See SOP	N
2216-10-224590-1	Chloride	11/11/2021	Screening	Soil, Sediment, Water/Cl	See SOP	N
222580-2	PFAS by SPE and LC/MS/MS Isotope Dilution	1/23/2024	Screening	Soil, Sediment, Water/PFAS	See SOP	N
23511-14-222579-1	PFAS by EPA 537 in Drinking Water by LC/MS/MS	2/1/2023	Screening	Water/PFAS	See SOP	N
2242-6-224578-1	Tannin & Lignin	9/11/2023	Screening	Water/Equipment Calibration	See SOP	N
7900-6-224124-1	Mercury 1631E Using the Cetac M-8000 Analyzer	2/25/2020	Definitive	Soil, Sediment, Water/Metals	Analyzer	N
2141-6-224122-1	Hydride Generation – AFS	4/19/2023	Definitive	Soil, Sediment, Water/Metals	Spectrometer	N

SOP #[1]	Title	Date	Definitive or Screening Data	Matrix/Analytical Group	SOP Option or Equipment Type	Modified for Project? Y/N
7474-2143-9-224127-1	Mercury in Soil and Tissue by CVAF 7474	1/21/2020	Definitive	Soil, Sediment/Metals	See SOP	N
7474-2142-10-224126-1	Mercury in Aqueous Samples by EPA 7474	1/14/2020	Definitive	Water/Metals	Spectrometer	N
9071-9733-3-224610-1	Oil & Grease and TPH in Soil 9071	6/19/2015	Definitive	Soil, Sediment/TPH	See SOP	N
ENV-SOP-WEST-0040	EPH by MassDEP Method	6/20/2024	Definitive	Sediment/EPH	See SOP	N
2229-15-224583-1	Total Solids in Solid and Semisolid Samples – Percent Solids	10/16/2023	Definitive	Soil, Sediment/% solid	See SOP	N
2128-10-224526-1	Herbicides EPA 8151A	3/9/2023	Definitive	Soil, Sediment, Water/Herbicides	GC/ECD	N
2120-19-234838-1	Volatile Petroleum Hydrocarbons (MA VPH)	12/8/2023	Definitive	Soil, sediment, water/VPH	See SOP	N
8015-222811-2	TPH and Saturated Hydrocarbons by GC/FID	12/2/2024	Definitive	Soil, sediment, water/TPH	GC/FID	N
8081-2158-14-222800-1	Determination of Organochlorine Pesticides By GC/ECD 8081	5/23/2018	Definitive	Soil, sediment, water/Pesticides	GC/ECD	N
222809-2	PCBs as Aroclors by GC/ECD	12/18/2024	Definitive	Soil, sediment, water/PCBs	GC/ECD	N
7471-2139-7-224128-1	Mercury in Solids by CVAA 7471	7/29/2015	Definitive	Soil, sediment/Metal	CVAA	N
4582-6-234829-4	Method 1633 Draft 2 PFAS in Aqueous, Solid Biosolids & Tissue by LCMSMS	4/4/2024	Definitive	Soil, sediment, water/PFAS	LC-MS/MS	N
31164-3-222810-1	Semivolatile Organic Compounds By GC/MS 8270E	1/14/2021	Definitive	Soil, sediment, water/SVOC	GC/MS	N

Notes:

[1] Laboratory Analytical SOPs are provided in Appendix B

Worksheet #24-1: Analytical Instrument Calibration Table – Gas Chromatography/Mass Spectrometry

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
GC/MS-SIM (PCB Homologs/ Aroclors/ Pesticides)	Perfluorotributyl-amine (PFTBA) tune	Water: 0.5-500 ng/L Solid: 0.3-400 ug/kg PUF: 5-5000 ng/cart	Prior to each Initial Calibration (ICAL)	m/e 69 – Base Peak with > 150,000 m/e 219 40% to 90% of Base Peak m/e 502 4% to 10% of Base Peak	Re-tune manually; Perform instrument/source maintenance as necessary	Analyst	2162-16-222801-1
	Decafluorotriphenyl-phosphine (DFTPP) tune		Prior to each ICAL; At the beginning of analytical sequence ; Every 12-18hrs	Perform in full SCAN mode; Maximum sensitivity criteria – see SOP; DDT breakdown < 20% (Pesticides only)	Perform instrument/injection port maintenance as necessary; Retune instrument		
	Initial Calibration [1](ICAL)		Initial instrument setup; After non-routine instrument service and; CCV/ICV criteria are not met	Minimum of 5 standards; Low standard must be ≤ RL; %RSD ≤ 20 except for 10% of compounds may be > 20 but ≤ 30% RSD, or r ≥ 0.99 (linear regression), or $r^2 \geq 0.99$ (non-linear regression); Single point calibration factors are used for PCB Aroclors	Review integrations and calculations; Perform and document remedial action as required; Repeat calibration		
	Initial Calibration[1] Verification (ICV)		Immediately after each ICAL	%D ≤ 30; exclusions apply – see SOP; Prepared using standard source	Re-analyze ICV if analytical error is suspected;		

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
				different than used for initial calibration	Recalibrate as needed		
	Continuing Calibration Verification (CCV)		At the beginning and end of every analytical sequence; Every 12-18 hours	%D ≤ 20 except for 20% of compounds may be > 20 but ≤ 30%D; Area counts of internal standards must be within 50 – 200% of the mid-level initial calibration standard.	Review integrations and calculations; Evaluate samples bracketed by failing CCV for obvious matrix interference; Re-analyze samples as needed		
GC/MS (Semi-VOA)	Decafluorotriphenyl-phosphine (DFTPP) tune	Compound specific; Water: 0.5-25 ug/L Solid: 33-1670 ug/kg PUF: 10-100 ug/cart	Prior to each ICAL; At the beginning of analytical sequence ; Every 12hrs	SW846 criteria (See SOP);	Perform instrument/injection port maintenance as necessary; Retune instrument	Analyst	31164-3-222810-1
	Initial Calibration[1] (ICAL)		Initial instrument setup; After non-routine instrument service; CCV/ICV criteria are not met	Minimum of 5 standards; Low standard must be ≤ RL; %RSD ≤20, or r ≥ 0.99 (linear regression), or if linear regression is used, recalculated concentration of the lowest calibration point is within 30% of the true value; All RFs must meet the minimum RFs listed in Table C of the SOP	Review integrations and calculations; Perform and document remedial action as required; Repeat calibration		
	Initial Calibration Verification (ICV)		Immediately after each ICAL	%D ≤ 30; Up to 5 sporadic marginal failures accepted;	Re-analyze ICV if analytical error is suspected;		

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
				Prepared using standard source different than used for initial calibration	Recalibrate as needed;		
	Continuing Calibration Verification (CCV)		At the beginning of every analytical sequence; Every 12 hours	%D ≤ 20 All RFs must meet the minimum RFs listed in Table C of the SOP; Area counts of internal standards must be within 50 – 200% of the mid-level initial calibration standard; The retention times of the internal standards must be within +0.05 RRT of the previous daily standard.	Review integrations and calculations; Evaluate samples bracketed by failing CCV for obvious matrix interference; Re-analyze samples as needed		
GC/MS-SIM (1,4-Dioxane)	Decafluorotriphenyl-phosphine (DFTPP) tune	Water: 150-100000 ng/L Solid: 8-8000 ug/kg	Prior to each ICAL; At the beginning of analytical sequence; Every 12 hrs	Perform in full SCAN mode; SW846 criteria (see SOP)	Perform instrument/injection port maintenance as necessary; Retune instrument	Analyst	2164-18-222793-1
	Initial Calibration[1] (ICAL)		Initial instrument setup; After non-routine instrument service; CCV/ICV criteria are not met	Minimum of 5 standards; Low standard must be ≤ RL; %RSD ≤ 20 or r ≥ 0.99 (linear regression)	Review integrations and calculations; Perform and document remedial action as required; Repeat calibration		
	Initial Calibration Verification (ICV)		Immediately after each ICAL	%D ≤ 30; Prepared using standard source different than used for initial calibration	Re-analyze ICV if analytical error is suspected; Recalibrate as needed		

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
	Continuing Calibration Verification (CCV)		At the beginning of every analytical sequence; Every 12 hours	%D ≤ 20 RFs > 0.05 Area counts of internal standards must be within 50 – 200% of the mid-level initial calibration standard. The retention times of the internal standards must be within +0.05 RRT of the previous daily standard.	Review integrations and calculations; Evaluate samples bracketed by failing CCV for obvious matrix interference; Re-analyze samples as needed		

[1]Calibration factor should be averaged, not forced through zero.
When batch, instrument, or sample QC fails, re-analysis shall be done. If upon reanalysis the QC item fails again, the samples shall be reextracted/reanalyzed, the laboratory shall detail the failure and corrective actions in the Case Narrative and report the data as qualified. For MS/MSD failures, re-analysis or re-extractions are not required provided that the associated LCS recovery is within the acceptance criteria, demonstrating a matrix interference.
SOP = standard operating procedure; PCB = polychlorinated biphenyl; RSD = Relative Standard Deviation; N/A = not applicable; GC = gas chromatography; MS = mass spectrometry; SIM = selected ion monitoring.

Worksheet #24-2: Analytical Instrument Calibration Table – Liquid Chromatography/Mass Spectrometry

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
LC/MS-MS (PFAS by 1633)	Initial Calibration[1] (ICAL)	0.5-250 ng/ml (ppb)	Initial instrument setup. After non-routine instrument service. CCV/ICV criteria are not met	Must meet one of the curve fit options presented in SOP	Review integrations and calculations. Perform and document remedial action as required. Repeat calibration	Analyst	ENV-SOP-MANS-0111V01
	Initial Calibration Verification (ICV)	Mid-Level Cal STD, Second Source	Immediately after each ICAL	±30% recovery of the true values Prepared using standard source different than used for initial calibration.	Re-analyze ICV if analytical error is suspected. Recalibrate as needed		
	Continuing Calibration Verification (CCV)	Alternating Low and Mid-Level Standard	Daily before sample analysis, after every 10 samples, and at end of analytical sequence.	All native and isotopically labelled compounds within ±30% recovery.	Review integrations and calculations; Evaluate samples bracketed by failing CCV for obvious matrix interference; Re-analyze samples as needed		

[1]Calibration factor should be averaged, not forced through zero.
When batch, instrument, or sample QC fails, re-analysis shall be done. If upon reanalysis the QC item fails again, the samples shall be reextracted/reanalyzed, the laboratory shall detail the failure and corrective actions in the Case Narrative and report the data as qualified. For MS/MSD failures, re-analysis or re-extractions are not required provided that the associated LCS recovery is within the acceptance criteria, demonstrating a matrix interference.
SOP = standard operating procedure; LC = liquid chromatography; MS = mass spectrometry

Worksheet #24-3: Analytical Instrument Calibration Table – Inductively Coupled Plasma Mass Spectrometry

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
ICP-MS	Tune verification ; External; multipoint	Varies by element	Daily or as required	Per SOP	Clean, inspect, adjust/recalibrate	ICP-MS Analyst, Dept. Manager	26797-4-224114
ICP	Response verification; External; single or multi-point	Varies by element	Daily or as required	Per SOP	Clean, inspect, adjust/recalibrate	ICP Analyst, Dept. Manager	26796-6-224118-1
CVAA	External; multi-point	Varies	Daily or as required	Per SOP	Clean, inspect, adjust/recalibrate	CVAA Analyst, Dept. Manager	25923-2-224115/25924-4-224116

When batch, instrument, or sample QC fails, re-analysis shall be done. If upon reanalysis the QC item fails again, the samples shall be reextracted/reanalyzed, the laboratory shall detail the failure and corrective actions in the Case Narrative and report the data as qualified. For MS/MSD failures, re-analysis or re-extractions are not required provided that the associated LCS recovery is within the acceptance criteria, demonstrating a matrix interference.
SOP = standard operating procedure; N/A = not applicable; ICP = inductively coupled plasma; MS = mass spectrometry; CVAA = cold vapor atomic absorption

Worksheet #24-4: Analytical Instrument Calibration Table – Inductively Coupled Plasma Mass Spectrometry

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
ICP-MS	Daily performance check, initial and continuing calibration per SOP	Linear Dynamic Range: Per analyte and confirmed with linear check standard	Performance check daily, ICAL daily, CCV every 10 samples	Per SOP	Inspect system, correct problem, rerun calibration and affected samples	Analyst	26797-4-224114
CVAF/CVAA (Mercury	Initial and continuing calibration per SOP	Per SOP	ICAL daily; CCV every 10 samples	Per SOP	Inspect system, correct problem, rerun calibration and affected samples	Analyst	25923-2-224115/25924-4-224116 7474-2142-10-224126-1/7474-2143-9-224127-1
Hydride (As and Se)	Initial and continuing calibration per SOP	Per SOP	ICAL daily; CCV every 10 samples	Per SOP	Inspect system, correct problem, rerun calibration and affected samples	Analyst	2141-6-224122-1
CVAF (Low-Level Mercury)	Initial and continuing calibration per SOP	Per SOP	ICAL daily; CCV every 10 samples	Per SOP	Inspect system, correct problem, rerun calibration and affected samples	Analyst	7900-6-224124-1

When batch, instrument, or sample QC fails, re-analysis shall be done. If upon reanalysis the QC item fails again, the samples shall be reextracted/reanalyzed, the laboratory shall detail the failure and corrective actions in the Case Narrative and report the data as qualified. For MS/MSD failures, re-analysis or re-extractions are not required provided that the associated LCS recovery is within the acceptance criteria, demonstrating a matrix interference.
SOP = standard operating procedure; N/A = not applicable; ICP = inductively coupled plasma; CVAF = cold vapor atomic fluorescence; CVAA = cold vapor atomic absorption

Worksheet #24-5: Analytical Instrument Calibration Table – Total Organic Carbon

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
TOC #1 (9060)	K-FACTORS	100%	Daily	+/- 3.0%	Recalibrate	Analyst	2182-25-222834-2
TOC #2 (9060)	K-FACTORS	100%	Daily	+/- 3.0%	Recalibrate	Analyst	2182-25-222834-2
TOC #3 (9060)	K-FACTORS	100%	Daily	+/- 3.0%	Recalibrate	Analyst	2182-25-222834-2
TOC #1 (Lloyd Kahn)	Linear regression	4.0%	Daily	0.995	Recalibrate	Analyst	2182-25-222834-2
TOC #2 (Lloyd Kahn)	Linear regression	4.0%	Daily	0.995	Recalibrate	Analyst	2182-25-222834-2
TOC #3 (Lloyd Kahn)	Linear regression	4.0%	Daily	0.995	Recalibrate	Analyst	2182-25-222834-2

When batch, instrument, or sample QC fails, re-analysis shall be done. If upon reanalysis the QC item fails again, the samples shall be reextracted/reanalyzed, the laboratory shall detail the failure and corrective actions in the Case Narrative and report the data as qualified. For MS/MSD failures, re-analysis or re-extractions are not required provided that the associated LCS recovery is within the acceptance criteria, demonstrating a matrix interference.
SOP = standard operating procedure; N/A = not applicable; TOC = total organic carbon

Worksheet #24-6: Analytical Instrument Calibration Table – Gas Chromatography

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
VOA (GC/MS) 8260D	4-Bromofluoro benzene (BFB) tune	Water: 5-1000ng Solid: 5-1500ng	Prior to each ICAL; At the beginning of analytical sequence; Every 12hrs	Perform in full SCAN mode; See SOP for BFB acceptance criteria;	Perform instrument/injection port maintenance as necessary; Retune instrument	Analyst	32639-8-224558-1
	Initial Calibration[1] (ICAL)		Initial instrument setup; After non-routine instrument service; CCV/ICV criteria are not met	Minimum of 5 standards; Low standard must be ≤ RL; %RSD ≤ 20 except for 10% of compounds may be > 20% but ≤ 30% RSD; r ≥ 0.99 (linear regression); r^2 ≥ 0.99 (non-linear regression)	Review integrations and calculations; Perform and document remedial action as required; Repeat calibration		
	Initial Calibration Verification (ICV)		Immediately after each ICAL	%D ≤ 30; exclusions apply – see SOP; Prepared using standard source different than used for initial calibration	Re-analyze ICV if analytical error is suspected; Recalibrate as needed		
	Continuing Calibration Verification (CCV)		At the beginning of every analytical sequence; Every 12 hours	%D ≤ 20 except for 20% of compounds may be > 20 but ≤ 30%D; Area counts of internal standards must be within 50–200% of the mid-level initial calibration standard	Review integrations and calculations; Re-analyze samples as needed		

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
SVOC (GC/MS) and (GC/MS-SIM)	Decafluorotriphenyl-phosphine (DFTPP) tune	Water / Soil (Full Scan) 1-200 ug/mL Water / Soil (SIM) 0.05-20 ug/mL	Prior to each ICAL; At the beginning of analytical sequence; Every 12 hours	Criteria listed in Table 3 of SW-846 8270E. Perform in full SCAN mode. DDT breakdown must be ≤20%. Pentachlorophenol and benzidine peak tailing factor must be ≤2.	Perform instrument/injection port maintenance as necessary. Retune instrument	Analyst	31162-3-224534-1 Full Scan 32637-4-224532-1 SIM
	Initial Calibration[1] (ICAL)		Initial instrument setup; After non-routine instrument service; CCV/ICV criteria are not met	Minimum of 5 standards, 6 if non-linear regression is used; Low standard must be ≤ RL; %RSD ≤ 20% or r ≥ 0.995 (linear regression) or r^2 ≥ 0.99 (non-linear regression); If %RSD ≤20, linear/non-linear must be used. Minimum RF's as per Table 4 of SW-846 8270E. If linear or non-linear regression used, verify the LOQ by recalculating lowest calibration standard using curve. (50-150%R)	Recalibrate if >10% of target analytes exceed %RSD, r, or r^2 criteria. If ≤10% of compounds exceed criteria, recalibration is not required as long as %RSD <40, r >0.98, or r^2 >0.98. If recalculated concentrations are outside of 50-150%R, report RL as estimated value or raise the RL to next highest calibration standard.		
	Initial Calibration Verification (ICV)		Immediately after each ICAL	70-130%, 40-160%R for difficult analytes. Prepared using standard source different than used for initial calibration	Locate source of problem. Recalibrate if >10% of all analytes are outside of criteria.		
	Continuing Calibration Verification (CCV)		At the beginning of every analytical sequence; Every 12 hours	%D ≤ 20 except difficult compounds may exhibit %D <60; Minimum RF per Table 4 in SW-846 8270E. Area counts of internal standards must be within	Recalibrate if >20% of all target analytes or >15% of analytes from a particular class (base-neutral or acid) exceed %D criteria.		

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
				50–200% of the mid-level initial calibration standard	If IS is outside of criteria, locate the source of problem and re-analyze the CCV. If ≤20% of compounds exceed criteria, recalibration is not required if %D <40 (or <60 for difficult analytes.)		
PCB, Pesticides, Herbicides (GC/ECD)	Initial Calibration[1] (ICAL)	Method specific PCBs 0.1-10 ug/mL	Initial instrument setup; After non-routine instrument service; CCV/ICV criteria are not met	Minimum of 5 standards; Low standard must be ≤ RL; %RSD ≤ 20 or r ≥ 0.99 (linear regression), If linear regression is used, recalculated concentration of the lowest and mid calibration point is within 30% of the true value	Review integrations and calculations; Perform and document remedial action as required; Repeat calibration	Analyst	8082-224529-2 8081-224528-2 2128-10-224526-1
	Initial Calibration Verification (ICV)	Pesticides 0.0005-0.2 ug/mL	Immediately after each ICAL	PCBs, Pesticides and Herbicides %D ≤ 20; Prepared using standard source different than used for initial calibration	Re-analyze ICV if analytical error is suspected; Recalibrate as needed		
	Continuing Calibration Verification (CCV)	Herbicides 0.05–2.0 ug/mL	At the beginning and end of every analytical sequence; Every 12 hours	PCBs and Pesticides %D ≤ 20; Herbicides %D ≤ 15; Area counts of internal standards must be within 50–150% of the mid-level initial calibration standard. The retention times of the internal standards must be within +0.05 RRT of the previous daily standard.	Review integrations and calculations; Evaluate samples bracketed by failing CCV for obvious matrix interference; Re-analyze samples as needed		

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
TPH/EPH (GC/FID)	Initial Calibration[1] (ICAL)	Individual Alkanes 3-500 ug/mL Range: 42-7000 ug/mL	Initial instrument setup; After non-routine instrument service ; CCV/ICV criteria are not met	Minimum of 5 standards; Low standard must be ≤ RL; %RSD ≤ 20% or r ≥ 0.99 (linear regression)	Review integrations and calculations; Perform and document remedial action as required; Repeat calibration	Analyst	2125-12-224535-1
	Initial Calibration Verification (ICV)		Immediately after each ICAL	%D ≤ 30%; Prepared using standard source different than used for initial calibration	Re-analyze ICV if analytical error is suspected; Recalibrate as needed		
	Continuing Calibration Verification (CCV)		At the beginning of every analytical sequence; Every 12 hours	%D ≤ 20%	Review integrations and calculations; Evaluate samples bracketed by failing CCV for obvious matrix interference; Re-analyze samples as needed		

[1]Calibration factor should be averaged, not forced through zero.
When batch, instrument, or sample QC fails, re-analysis shall be done. If upon reanalysis the QC item fails again, the samples shall be reextracted/reanalyzed, the laboratory shall detail the failure and corrective actions in the Case Narrative and report the data as qualified. For MS/MSD failures, re-analysis or re-extractions are not required provided that the associated LCS recovery is within the acceptance criteria, demonstrating a matrix interference.
SOP = standard operating procedure; PCB = polychlorinated biphenyl; RSD = Relative Standard Deviation ; N/A = not applicable; GC = gas chromatography; MS = mass spectrometry; FID = flame ionization detection

Worksheet #24-7: Analytical Instrument Calibration Table – Miscellaneous Laboratory Instruments

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
Lachat (Ammonia and TKN)	Blank and 10 standards. Second-source ICV.	0.05-20 mg/l	Daily	CC ≥ 0.995; low and high ICV, CCV ± 10%	Inspect system, correct problem, re-run calibration and ICV's	Analyst	2206-12-224606-1, 2207-12-224607-1
Lachat (Cyanide)	Blank and 7 standards. Second-source ICV.	0.004-0.5 mg/l	Daily	CC ≥ 0.995; low and high ICV, CCV ± 10%	Inspect system, correct problem, re-run calibration and ICV's	Analyst	2210-14-224624-1
Lachat (Nitrate and Nitrite)	Blank and 6 standards. Second-source ICV.	0.05-8.0 mg/l	Daily	CC ≥ 0.995; low and high ICV, CCV ± 10%	Inspect system, correct problem, re-run calibration and ICV's	Analyst	224604-2
Lachat (Chloride)	Blank and 6 standards. Second-source ICV.	1.0-100 mg/l	Daily	CC ≥ 0.995. 3 levels for ICV, CCV ± 10%	Inspect system, correct problem, re-run calibration and ICV's	Analyst	2216-10-224590-1
DO meter (BOD)	Per manufacturer's instructions.	NA	Daily	Per manufacturer's instructions	Inspect probe, change probe, if needed	Analyst	ENV-SOP-WES2-0062
pH electrode (pH)	Blank and 3 standards.	Buffer 4,7,10	Daily	ICV within 0.05 PH units; CCV	Inspect probe and buffers, correct problem,	Analyst	2202-11-224615-1,

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
	Second-source ICV.			±2%(waters); ±2 units for soils	re-run calibration and ICV		2203-9-224577-1
TOC analyzer (TOC, DOC)	Blank and 4 standards. Second-source ICV.	0.5 mg/l-10 mg/l	Every 6 months or after a significant change to instrument.	Correlation Coefficient ≥ 0.995. ICV/CCV ±10%	Inspect system, correct problem, re-run calibration and ICV	Analyst	2215-15-224626-1
Spectrometer (Sulfate)	Blank and 3 standards. Second-source ICV.	10-40 mg/l	Daily	Correlation Coefficient ≥ 0.995. ICV/CCV ±10%	Inspect system. Correct problem. Re-run calibration and ICV.	Analyst	224621-2
Spectrometer (Phosphorus)	Blank and 5 standards. Second-source ICV.	0.01-1.0 mg/l	Daily	Correlation Coefficient ≥ 0.995 ICV/CCV ±10%	Inspect system. Correct problem. Re-run calibration and ICV.	Analyst	2226-12-224628-1
Spectrometer (Hexavalent Chromium)	Blank and 7 standards for liquids. Blank and 4 standards for soils. Second-source ICV.	Liquids 0.005-0.5 mg/l Soils 0.01-1.0 mg/kg	Every 12 months or after a significant change to instrument	Correlation Coefficient ≥ 0.995 ICV/CCV ±15%	Inspect system. Correct problem. Re-run calibration and ICV.	Analyst	2204-11-224601-1
Spectrometer (Phenol)	Blank and 4 standards. Second-source ICV.	Phenol: 0.025-2.5 mg/L	Daily	Correlation Coefficient ≥ 0.995 ICV/CCV ±15%	Inspect system. Correct problem. Re-run calibration and ICV.	Analyst	2211-9-224582-1
Spectrometer (Free Cyanide)	Blank and 8 standards. Second-source ICV.	2.0-300 ug/L	Daily	Correlation Coefficient ≥ 0.995. ICV/CCV ±15%	Inspect system, correct problem, re-run	Analyst	3743-3-224600-1

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
					calibration and ICV.		
Spectrometer (Sulfide)	Blank and 7 standards. Second-source ICV.	0.055-1.755 mg/l	Annual, after significant instrument maintenance, or if CCVs fail	Correlation Coefficient ≥ 0.995. ICV/CCB ±25%	Inspect system, correct problem, re-run calibration and ICV	Analyst	2221-11-224632-1
Spectrometer (COD)	High-Level COD: Blank and 8 standards. Low-Level COD: Blank and 7 standards. Second-source ICV.	High-Level COD: 20-1250 mg/l Low-Level COD: 5-500 mg/l	Every 6 months, after significant instrument maintenance, or new lot of COD tubes, or if CCVs fail.	Correlation Coefficient ≥ 0.995. ICV/CCV ±10% (EPA 410.4) ICV/CCV within control limits (SM 5220D)	Inspect system, correct problem, re-run calibration and ICV	Analyst	2208-15-224589-1
Spectrometer (Nitrite)	Blank and 4 standards. Second-source ICV.	0.02-0.3 mg/l	Daily, or if CCVs fail	Correlation Coefficient ≥ 0.995. ICV/CCV ±10%	Inspect system, correct problem, re-run calibration and ICV	Analyst	2243-8-224605-1
Ion Chromatography (Anions: Fluoride, Bromide, Chloride, Nitrate, Sulfate)	Blank and 6 standards. Second-source ICV.	Fluoride, Bromide, Nitrate: 0.025-5 mg/l Chloride: 0.25-50 mg/l Sulfate: 0.5-100 mg/l	Calibrated every 6 months, or after significant instrument maintenance, if retention time shifts or CCVs fail.	Correlation Coefficient ≥ 0.995. ICV/CCV ±10%	Inspect system, correct problem, re-run calibration and ICV	Analyst	2214-11-224593-1
Balance (Oil and Grease, Total Petroleum Hydrocarbons, BTU)	The calibration of the analytical balance must be verified with Class S weights prior to and at the conclusion of the	2mg-1000mg	Daily, Prior to use	Calibration shall be within ±10% (i.e., ± 0.2 mg) at 2mg and ± 0.5 % (i.e., ± 5 mg) at 1000mg.	If values are not within these limits, check for debris around the balance, and check the bubble	Analyst	267100-1

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Responsible Person for CA	SOP Reference
	mass determinations of the HEM sample analysis.				for a level surface.		
Spectrometer (Reactive Sulfide)	Blank and 7 standards. Second-source ICV.	0.055-1.755 mg/l	Annual, after significant instrument maintenance, or if CCVs fail	Correlation Coefficient ≥ 0.995. ICV/CCB ±25%	Inspect system. Correct problem. Re-run calibration and ICV.	Analyst	2228-6-224619-1
Spectrometer (Reactive Cyanide)	Blank and 5 standards. Second-source ICV	0.004-0.5 mg/l	Annual, after significant instrument maintenance, or if CCVs fail	Correlation Coefficient ≥ 0.995. ICV/CCB ±25%	Inspect system. Correct problem. Re-run calibration and ICV.	Analyst	2228-6-224619-1
Calorific Value (BTU)	Benzoic Acid Tablet	N/A	Monthly	99% to 101% of True Value. TV=11373	Rerun once. If rerun does not pass, perform maintenance and recalibrate.	Analyst	ENV-SOP-ASHE-0015
LECO Sulfur Analyzer	Second source ICV/CCV	N/A	After every 10 samples and at the end of the analytical sequence	% Diff ±10%	Reanalyze once. If it still fails, recalibrate and reanalyze all samples after last passing CCV.	Analyst	ENV-SOP-ASHE-0009

When batch, instrument, or sample QC fails, re-analysis shall be done. If upon reanalysis the QC item fails again, the samples shall be reextracted/reanalyzed, the laboratory shall detail the failure and corrective actions in the Case Narrative and report the data as qualified. For MS/MSD failures, re-analysis or re-extractions are not required provided that the associated LCS recovery is within the acceptance criteria, demonstrating a matrix interference.
SOP = standard operating procedure; PCB = polychlorinated biphenyl; RSD = Relative Standard Deviation ; N/A = not applicable; BOD = biological oxygen demand; TOC = total organic carbon; DOC = dissolved organic carbon; COD=chemical oxygen demand; BTU=British thermal unit.

Worksheet #25-1: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[1]	SOP Reference
GC/MS (SVOC) GC/MS–SIM (PAH)	Inlet Maintenance : Septa, Injection port liner, clip column	Passing Tune/CCAL; overall chromatogram	Instrument performance and sensitivity	Frequency is dependent on degree of contamination and standard recovery	See SOP	See SOP	Analyst or Section Supervisor	WI ID No.: 2242-6-224578-1; See specific analysis SOP
GC/MS (SVOC) GC/MS –SIM (PAH)	Column	Passing Tune/ICAL/ ICV; overall chromatogram	Instrument performance and sensitivity	Frequency is dependent on degree of contamination and standard recovery	See SOP	See SOP	Analyst or Section Supervisor	WI ID No.: 2242-6-224578-1; See specific analysis SOP
GC/MS (SVOC) GC/MS –SIM (PAH)	Source cleaning: Filaments, insulators	Tuning	Instrument performance and sensitivity	Frequency is dependent on degree of contamination and standard recovery	See SOP	See SOP	Analyst or Section Supervisor	WI ID No.: 2242-6-224578-1; See specific analysis SOP
GC/MS (SVOC) GC/MS–SIM (PAH)	Pump	Complete MS pump down	Air and water check	Frequency is dependent on vacuum within instrument	See SOP	See SOP	Analyst or Section Supervisor	WI ID No.: 2242-6-224578-1; See specific analysis SOP
GC/ECD	Inlet Maintenance : Septa, Injection port liner, clip column	Passing PEM and CCAL; overall chromatogram	Instrument performance and sensitivity	Frequency is dependent on degree of contamination and standard recovery	See SOP	See SOP	Analyst or Section Supervisor	WI ID No.: 2242-6-224578-1; See specific analysis SOP
GC/ECD	Column	Passing CCAL; overall chromatogram	Instrument performance and sensitivity	Frequency is dependent on degree of contamination and standard recovery	See SOP	See SOP	Analyst or Section Supervisor	WI ID No.: 2242-6-224578-1; See specific analysis SOP

Notes:
Laboratory QC limits established at the time of analysis will be used.
All limits and procedures defer to the current lab SOPs established at the time of sample analysis.

GC = gas chromatography; MS = mass spectrometry; SIM = selected ion monitoring; SVOC = semivolatile organic compound; PAH = polycyclic aromatic hydrocarbons; ECD = electron capture detector; ICAL = initial calibration; CCAL = continuing calibration; PEM = photon emission microscopy; SOP = standard operating procedure

Worksheet #25-2: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[1]	SOP Reference
LC/MS-MS (PFAS)	Isolator Column	ICAL/CCAL; overall chromatogram	Instrument performance and sensitivity	Frequency is dependent on degree of contamination and standard recovery	See SOP	See SOP	Analyst or Section Supervisor	SOP 23511-14-222579-1 SOP 222580-223528 1633-305197-1
LC/MS-MS (PFAS)	LC Column	ICAL/ CCAL; overall chromatogram	Instrument performance and sensitivity	Frequency is dependent on degree of contamination and standard recovery	See SOP	See SOP	Analyst or Section Supervisor	SOP 23511-14-222579-1 SOP 222580-2 1633-305197-1
LC/MS-MS (PFAS)	Clean Cone assembly	ICAL/CCAL; overall chromatogram	Instrument performance and sensitivity	Frequency is dependent on degree of contamination and standard recovery	See SOP	See SOP	Analyst or Section Supervisor	SOP 23511-14-222579-1 SOP 222580-2 1633-305197-1
LC/MS-MS (PFAS)	Source/coil cleaning.	Tuning/Mass Calibration	Instrument performance and sensitivity	Annually unless contamination warrants greater frequency.	See SOP	See SOP	Analyst or Section Supervisor	SOP 23511-14-222579-1 SOP 222580-2

Notes:
Laboratory QC limits established at the time of analysis will be used.
All limits and procedures defer to the current lab SOPs established at the time of sample analysis.
LC = liquid chromatography; MS = mass spectrometry; ICAL = initial calibration; CCAL = continuing calibration; SOP = standard operating procedure; PFAS = per- and poly-fluoroalkyl substances

Worksheet #25-3: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[1]	SOP Reference
ICP-MS	Introduction system cleaning. Torch and Cones cleaning. Rough pump oil change	Nebulizer backpressure monitoring. Vacuum reading.	Software monitored. Oil reservoir.	Continuous	Per manufacturer	Clean and restore Change oil, evacuate ballast	ICP-MS Analyst	26797-4-224114
ICP	Introduction system cleaning. Torch Clean Flow adjustment. Cell conditioning	Nebulization monitoring	Visual	Daily	Flow consistent and unobstructed	Dismantle, clean	ICP Analyst	26796-6-224118-1
CVAA	Sample delivery tubing	Inspect for wear	Visual	Daily inspection Replace as needed	Uptake acceptance	Replacement	Analyst	7471-2139-7-224128-1
CVAA	Autosampler control arms	Freely moving, sample position acquired	Visual	Daily	Working condition	Replacement	Analyst	7471-2139-7-224128-1

Notes:
Laboratory QC limits established at the time of analysis will be used.
All limits and procedures defer to the current lab SOPs established at the time of sample analysis.
ICP = inductively coupled plasma; MS = mass spectrometry; CVAA = cold vapor atomic absorption; SOP = standard operating procedure

Worksheet #25-4: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[1]	SOP Reference
ICP-MS	Clean cones, replace tubing, inspect sample introduction system	Daily performance and tuning report	Instrument performance and sensitivity	Maintenance – as needed	See SOP	See SOP	Analyst or Section Supervisor	26797-4-224114
Mercury/Hydride Analyzer CVAF, CVAA, Hydride Gen.	Replace tubing, inspect sample introduction system	Calibration performance and QC recovery	Sensitivity and background check	Maintenance – as needed	See SOP	See SOP	Analyst or Section Supervisor	7471-2139-7-224128-1

Notes:
Laboratory QC limits established at the time of analysis will be used.
All limits and procedures defer to the current lab SOPs established at the time of sample analysis.
ICP = inductively coupled plasma; MS = mass spectrometry, SOP = standard operating procedure; CVAF = cold vapor atomic fluorescence; CVAA = cold vapor atomic absorption

Worksheet #25-5: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[1]	SOP Reference
TOC Analyzer	Change Columns	Blank and Analytical Standards	Gases, Column	Daily or as needed	See SOP	See SOP	Analyst or Section Supervisor	2182-25-222834-2
Sieves/Sieve Shaker	Check Shaker and Sieves	Duplicate	Check sieves for rips or tares in screens	Daily or as needed	See SOP	Replace sieves	Analyst or Section Supervisor	2183-10-222829-1

Notes:
Laboratory QC limits established at the time of analysis will be used.
All limits and procedures defer to the current lab SOPs established at the time of sample analysis.
TOC = total organic carbon; SOP = standard operating procedure

Worksheet #25-6: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[1]	SOP Reference
GC/MS	Inlet Maintenance : Septa, Injection port liner, clip column	Passing Tune/CCAL; overall chromatogram	Instrument performance and sensitivity	Frequency is dependent on degree of contamination and standard recovery	See SOP	See SOP	Analyst or Section Supervisor	31162-3-224534-1
GC/MS	Column	Passing Tune/ICAL/ ICV; overall chromatogram	Instrument performance and sensitivity	Frequency is dependent on degree of contamination and standard recovery	See SOP	See SOP	Analyst or Section Supervisor	31162-3-224534-1
GC/MS	Source cleaning: Filaments, insulators	Tuning	Instrument performance and sensitivity	Frequency is dependent on degree of contamination and standard recovery	See SOP	See SOP	Analyst or Section Supervisor	31162-3-224534-1
GC/MS	Pump	Complete MS pump down.	Air and water check	Frequency is dependent on vacuum within instrument	See SOP	See SOP	Analyst or Section Supervisor	31162-3-224534-1
GC/ECD/FID	Inlet Maintenance : Septa, Injection port liner, clip column	Passing CCAL; overall chromatogram	Instrument performance and sensitivity	Frequency is dependent on degree of contamination	See SOP	See SOP	Analyst or Section Supervisor	8015-222811-2, ENV-SOP-WEST-0040

Instrument/Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[1]	SOP Reference
				and standard recovery				
GC/ECD/FID	Column	Passing CCAL; overall chromatogram	Instrument performance and sensitivity	Frequency is dependent on degree of contamination and standard recovery	See SOP	See SOP	Analyst or Section Supervisor	8015-222811-2, ENV-SOP-WEST-0040

Notes:
Laboratory QC limits established at the time of analysis will be used.
All limits and procedures defer to the current lab SOPs established at the time of sample analysis.
GC = gas chromatography; MS = mass spectrometry; CCAL = continuing calibration; SOP = standard operating procedure; ECD = electron capture detector; FID = flame ionization detector; ICAL = initial calibration; ICV = initial calibration verification

Worksheet #25-7: Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person[1]	SOP Reference
pH electrode	Rinse; store in buffer 7.	Analytical standards	Instrument performance	Prior to use	LCS: 6.95-7.05	Recalibrate, condition probe	Analyst	2203-9-224577-1, 2202-11-224615-1
Conductivity meter	Rinse, check for cracks and scratches	Analytical standards	Instrument performance	Prior to use	LCS: ±1% of true value	Rinse; recalibrate	Analyst	2230-9-224620-1
Spectrometer	Replace lamp as needed, rinse and wipe cells	Blank and Analytical standards	Instrument performance	As needed	LCS is within control, Re-zero for Blank	Wipe, change lamp, recalibrate	Analyst	Various SOPs
TOC analyzer	Inspect tubing, scrubber, syringe, injection needle. Prime instrument	Blank and Analytical standards	Instrument performance	Daily or as needed	LCS is within control, Blank is below RL	Replace parts as needed, re-calibrate	Analyst	2215-15-224626-1
Lachat	Rinse tubing, change tubing as needed, check for blockage, O-rings changed as needed	Monitoring baseline, timing, and peak shape	Instrument performance	Daily or as needed	Baseline is flat, peaks have the correct shape, timing is correct	Replace parts as needed, recalibrate	Analyst	Various SOPs
Ion Chromato-graph	Replace column and suppressor as needed, check eluent level, check for leaks	Analytical standards	Check baseline and retention time	Daily or as needed	Flat baseline, correct retention time, LCS within ±10% of true value	Replace lines, column, or suppressor	Analyst	2214-11-224593-1
ORP meter	Rinse probe, inspect for cracks	Analytical Standards	Instrument performance	Prior to use	LCS ± 10%	recalibrate	Analyst	2237-4-224612-1
Balance	Check for debris and level working surface	Weighing samples	Visual	Daily	± 10% for daily check of certified weights	Clean, Level Balance	Analyst	Various SOPs

Notes:
Laboratory QC limits established at the time of analysis will be used.
All limits and procedures defer to the current lab SOPs established at the time of sample analysis.
LCS = laboratory control spike; TOC = total organic carbon; RL = reporting limit; ORP = oxidation-reduction potential; SOP = standard operating procedure

Worksheet #26: Sample Handling System

Sampling Organization: GEI Consultants, Inc.

Laboratories: Pace Analytical Laboratories/Alpha Analytical/Geo Testing Express

Method of Sample Delivery (shipper/carrier): Laboratory courier or third-party courier (FedEx, UPS, etc.)

Number of Days from Reporting Until Sample Disposal: As described in the attached laboratory QA Manual (Appendix C).

Activity	Organization and Title or Position of Perform Responsible for the Activity	SOP Reference[1]
Sample Collection, Packaging, and Shipment		
Sample Collection	Lead Field Geologist/Engineer/GEI Field Support Staff, GEI	FD-003 – Sample Management and Chain of Custody
Sample Labeling	Lead Field Geologist/Engineer/GEI Field Support Staff, GEI	FD-003 – Sample Management and Chain of Custody
Chain-of-custody form completion	Lead Field Geologist/Engineer/GEI Field Support Staff, GEI	FD-003 – Sample Management and Chain of Custody
Sample Packaging	Lead Field Geologist/Engineer/GEI Field Support Staff, GEI	FD-003 – Sample Management and Chain of Custody
Coordination of Shipment	Lead Field Geologist/Engineer/GEI Field Support Staff, GEI	FD-003 – Sample Management and Chain of Custody
Sample Receipt and Analysis		
Sample Receipt, Inspection, and Log-in	Laboratory Sample Receiving Technician	ENV-MAN-WES2-0001 v02 – Quality Manual, ENV-SOP-WES2-0002 – Sample Custody and Tracking
Sample Custody and Storage	Laboratory Sample Receiving Technician	ENV-MAN-WES2-0001 v02 – Quality Manual, ENV-SOP-WES2-0002 – Sample Custody and Tracking
Sample Preparation	Laboratory Sample Receiving Technician	ENV-MAN-WES2-0001 v02 – Quality Manual, ENV-SOP-WES2-0002 – Sample Custody and Tracking
Sample Determinative Analysis	Laboratory Sample Receiving Technician	ENV-MAN-WES2-0001 v02 – Quality Manual, ENV-SOP-WES2-0002 – Sample Custody and Tracking
Sample Archiving		
Sample Storage	Laboratory Analyst	ENV-MAN-WES2-0001 v02 – Quality Manual, ENV-SOP-WES2-0002 – Sample Custody and Tracking
Sample Extract/Digest Storage	Laboratory Analyst	ENV-MAN-WES2-0001 v02 – Quality Manual, ENV-SOP-WES2-0002 – Sample Custody and Tracking
Sample Disposal		
Personnel/Organization	Laboratory Analyst As Per QA Manual and Method SOP	ENV-MAN-WES2-0001 v02 – Quality Manual, ENV-SOP-MANS-0077 – Hazardous Waste Management and Sample Disposal, and Method SOPs

Activity	Organization and Title or Position of Perform Responsible for the Activity	SOP Reference[1]
Number of Days from Analysis	Laboratory Analyst As Per QA Manual and Method SOP	ENV-MAN-WES2-0001 v02 – Quality Manual, ENV-SOP-MANS-0077 – Hazardous Waste Management and Sample Disposal, and Method SOPs

[1] GEI SOPs are in Appendix A. Pace/Alpha SOPs are located in Appendix B.
[2] Examples of a chain-of-custody form and sample packaging are included in SOP FD-003 – Sample Management and Chain of Custody.
[3] Project-specific contractual agreements may require a longer retention period. Project samples should not be disposed of until authorized by the GEI Project Manager.

Worksheet #27: Sample Custody Requirements

Field Sample Custody Procedures (sample collection, packaging, shipment, and delivery to laboratory):

Each environmental sample will be properly identified and individually labeled. Label will be filled out in indelible ink with at least the following information: sample identification, date and time of sample collection, initials of sampler(s), analysis/test required, preservation (as appropriate). The sample label will be securely attached to the sample container.

Environmental samples being analyzed by the analytical laboratory will be properly packaged and shipped for analysis. Following labeling, the sample bottles will be sealed with custody deals, and enclosed in clear sealable plastic bags (i.e., Ziploc© bags), through which identifying labels is visible.

The sample containers, sealed bags, will then be placed in a hard-plastic cooler, an effort will be made to fill in voids with either ice or packaging material (i.e., bubble wrap). Samples are to be placed with sufficient ice (enclosed in double-bagged sealable plastic bags) to cool the samples to 4±2°C. Additionally, each iced cooler will be packed with a cooler temperature blank. Lastly, the cooler will be filled with adequate cushioning to minimize the possibility of container breakage.

Laboratory Sample Custody Procedures (receipt of sample, archiving, disposal):

Laboratory custody procedures are described in the attached laboratory QA Manual (Appendix C), when applicable.

Sample Identification Procedures:

The method of identification of a sample depends on the type of measurement or analysis performed. When field screening measurements are made, data will be recorded directly in logbooks or on field investigation forms. Identifying information such as project name, station number, station location, date and time, name of sampler, field observations, remarks, or other pertinent information will be recorded.

Samples collected for laboratory analysis during the field investigation will be specifically designated by GEI personnel for unique identification. Each sample will be designated by an alpha-numeric code which will identify the sampling location, type, or as necessary, depth. Information regarding sample nomenclature and identification can be found in Worksheet #14.

Chain-of-Custody Procedures:

A completed chain-of custody form must be included with sample transfers/shipments. An example chain-of custody form is included in Appendix B.

When analytical samples are being shipped by an overnight delivery service to the laboratory, the chain-of custody form and other paperwork will be checked against the sample labels and field documentation and then placed in a waterproof sealable plastic bag and taped securely to the inside lid of the cooler. The cooler must then be secured, with custody seals affixed over the lid in at least two locations, and the cooler wrapped with strapping tape (without obscuring the custody seals). Orientation "this end up" arrows will be drawn or attached on two sides of the cooler, and a completed overnight delivery service shipping label will be attached to the top or handle of the cooler. Wide, clear tape should be used to secure the label to the lid of the cooler to prevent the shipping address label from being accidentally peeled off the cooler.

Samples to be shipped by an overnight delivery service will be shipped within 24 hours of sample collection and arrive at the laboratory within 24 hours of sample shipment. A member of the field team will contact the laboratory directly, to notify them of a sample shipment, and again the next day to confirm receipt. In addition, an electronic version of the chain-of custodies will be sent to the laboratory either electronically or by mail.

If the laboratory is going to courier the samples directly from the site or the samples are being delivered directly to the laboratory by GEI, the chain-of custody form will not be placed inside the cooler. The chain-of custody form must be signed by the receiver (i.e., the laboratory courier, or the laboratory sample custodian) when he/she accepts possession of the samples.

Worksheet #28-1: Analytical Quality Control and Corrective Action – PFAS

Laboratory Facility: Alpha Analytical, Mansfield, MA
Matrix: Water, Soil, Biosolid, Tissue
Analytical Group: PFASs
Analytical Method/SOP Reference: EPA 1633 / SOP# ENV-SOP-MANS-0111 V01

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank (MB)	One per preparatory batch of up to 20 samples	No analyte greater than the reporting limit.	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >10x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst/ Laboratory Quality Assurance Officer	<RL
Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicate (LCSD)	One each per preparatory batch of up to 20 samples. Alternate spiking concentration from batch to batch at a low, medium and high concentration.	Per SOP	Correct problem, re-prep and reanalyze LCS and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst/ Laboratory Quality Assurance Officer	Per SOP
Matrix Spike (MS)	One per preparatory batch of up to 20 samples. Alternate spiking concentration from batch to batch at a low, medium and high concentration.	Per SOP	Report if associated with passing LCS and CCV. Discuss in narrative.	Laboratory Analyst/Section Supervisor	Per SOP
Field Duplicate (D) or Matrix Spike Duplicate (MSD)	One per preparatory batch of up to 20 samples.	RPD ≤ 30% for compounds > than 2x the RL. RPD ≤ 50% for compounds above the RL but < than 2x the RL.	Evaluate during data validation.	Data validation staff	RPD ≤ 30% for compounds > than 2x the RL. RPD ≤ 50% for compounds above the RL but < than 2x the RL.
Extracted Internal Standard (EIS)	All field samples and QC samples	Per SOP	Check 1) calculations to locate possible errors, 2) standard solutions for degradation, 3) contamination, and 4)	Laboratory Analyst/Section Supervisor	Per SOP

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
			instrument performance. For CCVs and QC elements spiked with all target analytes, if the recovery of the corresponding target analytes meet the acceptance criteria for the EIS in question, the data can be used but all potential biases in the recovery of the EIS must be documented in the sample report. If the associated target analytes do not meet the acceptance criteria, the data must be reanalyzed.		
Non-Extracted Internal Standard (NIS)	All field samples and QC samples	Per SOP	N/A	Laboratory Analyst/Section Supervisor	Per SOP

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection, N/A = not applicable

Worksheet #28-2: Analytical Quality Control and Corrective Action – Metals

Laboratory Facility: Alpha Analytical, Mansfield, MA
Matrix: Soil
Analytical Group: Metals
Analytical Method/SOP Reference: 6020B/SOP 26797-4-224114

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	1 per prep batch of up to 20 samples.	No target analytes >RL	Reprep and reanalyze the method blank and all samples processed with the contaminated blank. Report results if sample results are > 10x blank result or sample results < RL	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
LCS (All Targets)	One per prep batch of up to 20 samples.	80-120%	Correct problem, reprep and reanalyze LCS and all samples in associated batch for failed analytes.	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
LCSD (All Targets)	Must be requested by client. One per batch of up to 20 samples	80-120% Recovery ≤30 RPD	Reanalyze; if reanalysis is not within criteria and LCS is in control, no corrective action necessary. If LCS and LCSD are not within control, reprep and reanalyze LCS and all samples in associated batch for failed analytes.	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
Duplicates	One per batch of up to 20 samples	RPD ≤ 20%	Qualify data as needed.	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
MS	One per batch of up to 20 samples	75-125%	Flag data. Discuss in narrative. Analyze post-spike if recoveries <30% and serial dilution fails	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
MSD	Must be requested by client. One per batch of up to 20 samples	75-125% Recovery ≤ 20% RPD	Flag data. Discuss in narrative.	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
Serial Dilution	One per batch of up to 20 samples as needed	RPD ≤ 10% if analyte > 50x MDL; No criteria if < 50x MDL	Flag data. Discuss in narrative.	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
SRM (Certified Targets Only)	Must be requested by client. One per batch of up to 20 samples	Supplier Certified Limits	Correct problem, reprep and reanalyze LCS and all samples in associated batch for failed analytes.	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-3: Analytical Quality Control and Corrective Action – Total Organic Carbon

Laboratory Facility: Alpha Analytical, Mansfield, MA
Matrix: Sediments/Soil
Analytical Group: Wet Chemistry
Analytical Method/SOP Reference: 9060A/Lloyd Kahn – Total Organic Carbon/2182-25-222834-2

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	1 per prep batch of up to 20 samples.	No TOC detected at reporting limit (RL), 10x the amount measured in any sample, or 10x the regulatory limit, whichever is greater.	Reanalyze the method blank and if still fails Recalibrate inst.	Analyst	All analytes in the method blank must be ≤ 0.01%
SRM	One per analytical batch of up to 20 samples.	QC acceptance criteria specified in DoD QSM v4.2, if available. Otherwise use in-house limits.	Correct problem, reanalyze SRM	Analyst	All analytes in samples found to be within +/- 20% of the PAL's on WS#15 MUST pass method/SOP criteria.
Method Blank	1 per prep batch of up to 20 samples.	No TOC detected at reporting limit (RL), 10x the amount measured in any sample, or 10x the regulatory limit, whichever is greater.	Reanalyze the method blank and if still fails Recalibrate inst.	Analyst	No TOC detected at reporting limit (RL), 10x the amount measured in any sample, or 10x the regulatory limit, whichever is greater.
SRM	One per analytical batch of up to 20 samples.	75%-125% Recovery	Correct problem, recalibrate, reanalyze SRM and all associated samples.	Analyst	75%-125% Recovery
Sample Duplicate	1 per prep batch of up to 20 samples.	RPD ≤ 25%	Reanalyze sample and duplicate if additional sample is available	Analyst	N/A

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection; TOC = total organic carbon

Worksheet #28-4: Analytical Quality Control and Corrective Action – Nitrogen/Ammonia

Laboratory Facility: Alpha Analytical, Westborough, MA
Matrix: Water and Soil/Sediment
Analytical Group: Wet Chemistry
Analytical Method/SOP Reference: 4500NH3-BH – Nitrogen, Ammonia/SOP 2206-12-224606-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per batch of up to 20 samples	No analyte at or above the reporting limit.	Identify source and attempt to eliminate. Re-prep and reanalyze blank and affected samples (if sufficient sample remains). If results for the Distillation Blank are greater than the reporting limit, and all sample results for the batch are greater than 10x the Blank result, sample results may be reported with a narrative to be included in the final report. Otherwise, all samples must be redistilled and reanalyzed	Analyst / Supervisor/Laboratory Quality Assurance Officer	<RL
LCS	One per batch of up to 20 samples.	80-120% for method 4500 waters; 90-110% for method 350 waters, 83-115% for soils	Correct problem, reanalyze once. If still failing re-prep and reanalyze LCS and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst / Supervisor/Laboratory Quality Assurance Officer	83-155%
Matrix Spike	One per batch of up to 20 samples.	80-120% for method 4500 waters; 90-110% for method 350 waters; 55-144% for soils	Report if associated with passing LCS. Discuss in narrative.	Laboratory Analyst/Section Supervisor	55-144%
Laboratory duplicate	One per batch of up to 20 samples	<20% waters and soils	Report with narrative	Laboratory Analyst/Section Supervisor	<20%

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-5: Analytical Quality Control and Corrective Action – Ignitability

Laboratory Facility: Alpha Analytical, Westborough, MA
Matrix: Water and Soil/Sediment
Analytical Group: Wet Chemistry
Analytical Method/SOP Reference: 1030 – Ignitability/SOP 224598

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per batch of up to 20 samples	> 150°F	Identify source and attempt to eliminate. Re-prep and reanalyze blank and affected samples (if sufficient sample remains).	Analyst / Supervisor/Laboratory Quality Assurance Officer	> 150°F
LCS	One per batch of up to 20 samples.	98-106% (81 ± 3°F)	Correct problem; re-prep and reanalyze LCS and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst / Supervisor/Laboratory Quality Assurance Officer	98-106% (81 ± 3°F)
Laboratory duplicate	All samples that flash below 150°F	NA	NA	Laboratory Analyst/Section Supervisor	NA

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-6: Analytical Quality Control and Corrective Action – VOCs

Laboratory Facility: Alpha Analytical, Westborough, MA
Matrix: Water and Soil/Sediment
Analytical Group: VOCs
Analytical Method/SOP Reference: 8260D/5035 – VOCs/SOP#32639-8-224558-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	Once every 12-hour period and prior to any sample analysis.	No analyte at or above the reporting limit.	Identify source and attempt to eliminate. Reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >10x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped, and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst	Same as Method/SOP QC Acceptance Limits
LCS/LCSD	One each per batch of up to 20 samples.	Generally, 70-130% Recovery; 20% RPD – analyte specific	Correct problem; reanalyze LCS/LCSD and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst	Same as Method/SOP QC Acceptance Limits
MS/MSD	One per batch of up to 20 samples. Per Client's Request	Generally, 70-130% Recovery; 20% RPD – analyte specific	Report if associated with passing LCS/LCSD. Discuss in narrative.	Analyst	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-7: Analytical Quality Control and Corrective Action – pH

Laboratory Facility: Pace Analytical, Westborough, MA
Matrix: Water and Soil/Sediment
Analytical Group: Wet Chemistry
Analytical Method/SOP Reference: 9045 – pH/SOP 2203-9-224577-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
LCS	One per batch of up to 20 samples.	99-101% (6.95 – 7.05)	Correct problem; re-prep and reanalyze LCS and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst / Supervisor/Laboratory Quality Assurance Officer	6.95-7.05
Laboratory duplicate	One per batch of up to 20 samples.	5%	Report with narrative	Laboratory Analyst/Section Supervisor	5%

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-8: Analytical Quality Control and Corrective Action – Reactive Sulfide and Cyanide

Laboratory Facility: Pace Analytical, Westborough, MA
Matrix: Water and Soil/Sediment
Analytical Group: Wet Chemistry
Analytical Method/SOP Reference: 7.3 – Sulfide, Reactivity & Cyanide, Reactivity/SOP 2228-6-224619-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per batch of up to 20 samples	No analyte at or above the reporting limit.	Identify source and attempt to eliminate. Re-prep and reanalyze blank and affected samples (if sufficient sample remains).	Analyst / Supervisor/Laboratory Quality Assurance Officer	<RL
LCS	One per batch of up to 20 samples.	30-125% for Reactive Cyanide; 60-125% for Reactive Sulfide	Correct problem; re-prep and reanalyze LCS and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst / Supervisor/Laboratory Quality Assurance Officer	30-125% CN 60-125% Sulfide
Laboratory duplicate	One per batch of up to 20 samples.	25% for waters; 40% for soils	Report with narrative	Laboratory Analyst/Section Supervisor	25% water; 40% soil

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection; CN = cyanide

Worksheet #28-9: Analytical Quality Control and Corrective Action – PAH-17

Laboratory Facility: Alpha Analytical, Mansfield, MA
Matrix: Solid/Water
Analytical Group: SVOCs
Analytical Method/SOP Reference: PAH-17/SOP 31164-3-222810-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per preparatory batch of up to 20 samples	No analyte at or above the reporting limit.	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results > 5x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst/ Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
LCS/LCSD	One each per preparatory batch of up to 20 samples	40-140% Recovery. 30% RPD.	Correct problem, reprep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst/ Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
MS/MSD	One each per preparatory batch of up to 20 samples. Per Client's Request	40-140% Recovery, 30% RPD	Report if associated with passing LCS/LCSD. Discuss in narrative.	Laboratory Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
Field Duplicate	One per preparatory batch of up to 20 samples. Per Client's Request	RPD ≤ 30% for compounds > than 5x the RL	Evaluate during data validation.	Data validation staff	Same as Method/SOP QC Acceptance Limits
SRM	One per preparatory batch of up to 20 samples. Per Client's Request	40-140% Recovery	List outliers in project narrative.	Project Analyst/ Laboratory Staff	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection; SRM = sample ratio mismatch

Worksheet #28-10: Analytical Quality Control and Corrective Action – SVOCs

Laboratory Facility: Alpha Analytical, Mansfield, MA
Matrix: Solid/Water
Analytical Group: SVOC
Analytical Method/SOP Reference: 8270E / SOP 31162-3-224534-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per preparatory batch of up to 20 samples	No analyte at or above the reporting limit.	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >5x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
LCS/LCSD	One each per preparatory batch of up to 20 samples	40-140% Recovery for Base/Neutral compounds, 30-130% for Acid compounds. 30% RPD.	Correct problem, reprep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst/ Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
MS/MSD	One each per preparatory batch of up to 20 samples. Per Client's Request	40-140% Recovery for Base/Neutral compounds, 30-130% for Acid compounds. 30% RPD.	Report if associated with passing LCS/LCSD. Discuss in narrative.	Laboratory Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
Field Duplicate	One per preparatory batch of up to 20 samples. Per Client's Request	RPD ≤30% for compounds > than 5x the RL	Evaluate during data validation.	Data validation staff	Same as Method/SOP QC Acceptance Limits
Method Blank	One per preparatory batch of up to 20 samples	No analyte at or above the reporting limit.	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >5x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst/ Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
LCS/LCSD	One each per preparatory batch of up to 20 samples	40-140% Recovery. 30% RPD.	Correct problem, reprep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst/ Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
MS/MSD	One each per preparatory batch of up to 20 samples. Per Client's Request	40-140% Recovery. 30% RPD.	Report if associated with passing LCS/LCSD. Discuss in narrative.	Laboratory Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
Field Duplicate	One per preparatory batch of up to 20 samples. Per Client's Request	RPD ≤30% for compounds > than 5x the RL	Evaluate during data validation.	Data validation staff	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.

**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.

DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-11: Analytical Quality Control and Corrective Action – Mercury

Laboratory Facility: Pace Analytical, Westborough, MA
Matrix: Water and Soil/Sediment
Analytical Group: Mercury – CVAF
Analytical Method/SOP Reference: 7471B & 7470A/SOP 25923-2-224115 & SOP 25924-4-224116

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per prep batch of up to 20 samples.	Target analyte > RL	Reprep and reanalyze the method blank and all samples processed with the contaminated blank. Report results if sample results are >10x blank result or sample results <RL	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
LCS	One per prep batch of up to 20 samples.	80-120%	Correct problem, reprep and reanalyze LCS and all samples in associated batch for failed analytes.	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
LCSD	Must be requested by client. One per batch of up to 20 samples	80-120% Recovery ≤30 RPD	Reanalyze; if reanalysis is not within criteria and LCS is in control, no corrective action necessary. If LCS and LCSD are not within control reprep and reanalyze LCS and all samples in associated batch for failed analytes.	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
Duplicates	One per batch of up to 20 samples	RPD ≤ 20%	Qualify data as needed.	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
MS	One per batch of up to 20 samples	75-125%	Flag data. Discuss in narrative	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
MSD	Must be requested by client. One per batch of up to 20 samples	75-125% Recovery; ≤20 RPD	Flag data. Discuss in narrative.	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits
SRM (Soil & Tissue Only)	Must be requested by client. One per batch of up to 20 samples	Supplier Certified Limits	Correct problem, reprep and reanalyze SRM and all samples in associated batch for failed analytes.	Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.

DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-12: Analytical Quality Control and Corrective Action – Grain Size

Laboratory Facility: Alpha Analytical, Mansfield, MA
Matrix: Soil/Sediment
Analytical Group: Wet Chemistry
Analytical Method/SOP Reference: ASTM D422 – Grain Size/SOP 2183-10-222829-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Sample Duplicate	1 per prep batch of up to 20 samples.	RPD $\leq$ 25%	Reanalyze sample and duplicate if additional sample is available	Analyst	N/A

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; RPD = relative percent difference

Worksheet #28-13: Analytical Quality Control and Corrective Action – 1,4 Dioxane

Laboratory Facility: Alpha Analytical, Westborough, MA
Matrix: Soils/Waters
Analytical Group: VOA
Analytical Method/SOP Reference: 1,4 Dioxane 8260E-SIM/SOP 2164-18-222793-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	Once every 12-hour period and prior to any sample analysis.	No analyte at or above the reporting limit.	Identify source and attempt to eliminate. Reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >10x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped, and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst	Same as Method/SOP QC Acceptance Limits
LCS/LCSD	One each per batch of up to 20 samples.	40-140% Recovery, 30% RPD	Correct problem; reanalyze LCS/LCSD and all samples in associated batch. If problem persists, contact Project Manager.	Analyst	Same as Method/SOP QC Acceptance Limits
MS/MSD	One per batch of up to 20 samples Per Client's Request	40-140% Recovery, 30% RPD	Report if associated with passing LCS/LCSD. Discuss in narrative.	Analyst	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-14: Analytical Quality Control and Corrective Action – Pesticides

Laboratory Facility: Alpha Analytical, Westborough, MA
Matrix: Soils/Waters
Analytical Group: Pesticides
Analytical Method/SOP Reference: 8081B/SOP 2158-14-222800-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per preparatory batch of up to 20 samples	No analyte at or above the reporting limit.	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >10x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
LCS/LCSD	One each per preparatory batch of up to 20 samples	30-150% Recovery. Aqueous RPD 30%, Soil RPD 50%	Correct problem, reprep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
MS/MSD	One each per preparatory batch of up to 20 samples. Per Client's Request	30-150% Recovery, Aqueous RPD 30%, Soil RPD 50%	Report if associated with passing LCS/LCSD. Discuss in narrative.	Laboratory Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-15: Analytical Quality Control and Corrective Action – PCB Aroclors

Laboratory Facility: Alpha Analytical, Westborough, MA
Matrix: Soils/Waters
Analytical Group: PCBs
Analytical Method/SOP Reference: 8082A/SOP 8082-224529-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per preparatory batch of up to 20 samples	No analyte at or above the reporting limit.	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >10x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
LCS/LCSD	One per preparatory batch of up to 20 samples	QC acceptance criteria 40-140% Recovery, Aqueous RPD 30%, Soil RPD 50%	Correct problem, reprep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
MS/MSD	One each per preparatory batch of up to 20 samples. Per Client's Request	40-140% Recovery, Aqueous RPD 30%, Soil RPD 50%	Report if associated with passing LCS/LCSD. Discuss in narrative.	Laboratory Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-16: Analytical Quality Control and Corrective Action – Herbicides

Laboratory Facility: Alpha Analytical, Westborough, MA
Matrix: Soils/Waters
Analytical Group: Herbicides
Analytical Method/SOP Reference: 8151A/SOP 2128-10-224526-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per preparatory batch of up to 20 samples	No analyte at or above the reporting limit.	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >10x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
LCS/LCSD	One each per preparatory batch of up to 20 samples	QC acceptance criteria 30-150% Recovery, Aqueous RPD 25%, Soil RPD 50%	Correct problem, reprep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
MS/MSD	One each per preparatory batch of up to 20 samples. Per Client's Request	QC acceptance criteria 30-150% Recovery, Aqueous RPD 25%, Soil RPD 50%	Report if associated with passing LCS/LCSD. Discuss in narrative.	Laboratory Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-17: Analytical Quality Control and Corrective Action – TPH-GRO/TPH-DRO

Laboratory Facility: Alpha Analytical, Westborough, MA
Matrix: Soils/Waters
Analytical Group: TPH
Analytical Method/SOP Reference: 8015D(M)/SOP 2125-12-224535-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per preparatory batch of up to 20 samples	No analyte at or above the reporting limit.	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >5x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
LCS/LCSD	One each per preparatory batch of up to 20 samples	QC acceptance criteria 40-140% Recovery. 40% RPD.	Correct problem, reprep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
MS/MSD	One each per preparatory batch of up to 20 samples. Per Client's Request	QC acceptance criteria 60-140% Recovery. 40% RPD.	Report if associated with passing LCS/LCSD. Discuss in narrative.	Laboratory Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-18: Analytical Quality Control and Corrective Action – Total Cyanide

Laboratory Facility: Alpha Analytical, Westborough, MA
Matrix: Soils/Waters
Analytical Group: Cyanides, Total
Analytical Method/SOP Reference: 9012B/SOP 2210-14-224624-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per preparatory batch of up to 20 samples	No analyte at or above the reporting limit	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >5x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
LCS/LCSD	One each per preparatory batch of up to 20 samples	Within ±15% for liquids and ±20% for soils RPD ≤ 20% for liquids and < 35% for soils	Correct problem, reprep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
MS/MSD	One each per preparatory batch of up to 20 samples. Per Client's Request	%R within ±20% for liquids and ±35% for solids RPD ≤20% for liquids and <35% for solids	Report if associated with passing LCS/LCSD. Discuss in narrative.	Laboratory Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-19: Analytical Quality Control and Corrective Action – Total Sulfide

Laboratory Facility: Alpha Analytical, Westborough, MA
Matrix: Soils/Waters
Analytical Group: Total Sulfide
Analytical Method/SOP Reference: 9030B/SOP 2221-11-224632-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per preparatory batch of up to 20 samples	No analyte at or above the reporting limit	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >5x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
LCS/LCSD	One each per preparatory batch of up to 20 samples	%R must be within 75-125% %RPD ≤25%	Correct problem, reprep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
MS/MSD	One each per preparatory batch of up to 20 samples. Per Client's Request	%R must be within in-house control limits %RPD ≤25%	Report if associated with passing LCS/LCSD. Discuss in narrative.	Laboratory Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-20: Analytical Quality Control and Corrective Action – AVS/SEM

Laboratory Facility: Alpha Analytical, Westborough, MA
Matrix: Sediments
Analytical Group: AVS/SEM
Analytical Method/SOP Reference: 9030B/SOP 2221-11-224632-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per preparatory batch of up to 20 samples	No analyte at or above the reporting limit	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >5x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
LCS/LCSD	One each per preparatory batch of up to 20 samples	%R must be within 85-115% %RPD ≤20%	Correct problem, reprep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
MS/MSD	One each per preparatory batch of up to 20 samples. Per Client's Request	%R must be within 75-125% %RPD ≤20%	Report if associated with passing LCS/LCSD. Discuss in narrative.	Laboratory Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-21: Analytical Quality Control and Corrective Action – Metals

Laboratory Facility: Alpha Analytical, Mansfield, MA
Matrix: Solids/Water
Analytical Group: Metals
Analytical Method/SOP Reference: 6010D/SOP 26796-6-224118-1

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per preparatory batch of up to 20 samples	Less than ½ of the LLOQ for all analytes of concern	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >5x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
LCS/LCSD	One each per preparatory batch of up to 20 samples	%R must be within ±20% %RPD ≤20%	Correct problem, reprep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
MS/MSD	One each per preparatory batch of up to 20 samples. Per Client's Request	%R must be within 75-125% %RPD must be within in-house control limits	Report if associated with passing LCS/LCSD. Discuss in narrative.	Laboratory Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; QA = quality assurance; QL = quantitation limit; RL = reporting limit; PAL = project action limits; MS/MSD = matrix spike/matrix spike duplicate; LCS/LCSD = laboratory control spike/laboratory control spike duplicate; S = sampling; A = analytical; RPD = relative percent difference; %R = percent recovery; LOQ = limit of quantitation; LOD = limit of detection

Worksheet #28-22: Analytical Quality Control and Corrective Action – Percent Sulfur

Laboratory Facility: Pace Analytical, Asheville, NC
Matrix: Sediments
Analytical Group: Percent Sulfur
Analytical Method/SOP Reference: ASTM D4239/ENV-SOP-ASHE-0009

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
N/A	N/A	N/A	N/A	N/A	N/A

N/A = not applicable

Worksheet #28-23: Analytical Quality Control and Corrective Action – BTU

Laboratory Facility: Pace Analytical, Asheville, NC
Matrix: Sediments
Analytical Group: Calorific Value (BTU)
Analytical Method/SOP Reference: ASTM D240-87/SOP ENV-SOP-ASHE-0015

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	Two per batch of 20 samples	RPD ≤10%	Report results with appropriate footnote.	Analyst	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; RPD = relative percent difference; SOP = standard operating procedure

Worksheet #28-24: Analytical Quality Control and Corrective Action – EPH

Laboratory Facility: Pace Analytical, Westborough, MA
Matrix: Sediment
Analytical Group: EPH
Analytical Method/SOP Reference: MA-EPH / ENV-SOP-WEST-0040

QC Sample	Number/Frequency	Method/SOP Acceptance Criteria	Corrective Action	Title/Position of Person Responsible for Corrective Action	Measurement Performance Criteria
Method Blank	One per preparatory batch of up to 20 samples	No analyte at or above the reporting limit.	Identify source and attempt to eliminate. Re-extract and/or reanalyze blank and affected samples (if sufficient sample remains). Qualify data as needed. Report data if sample results >10x blank or sample results ND. If contamination is widespread or reoccurring, analyses must be stopped and the source of contamination must be eliminated or reduced before analyses can continue.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
LCS/LCSD	One each per preparatory batch of up to 20 samples	QC acceptance criteria 40-140% Recovery (except nonane 30%-140%). 25% RPD.	Correct problem, reprep and reanalyze LCS/LCSD and all samples in associated batch for failed analytes. If problem persists, contact Project Manager.	Analyst / Laboratory Quality Assurance Officer	Same as Method/SOP QC Acceptance Limits
MS/MSD	One each per preparatory batch of up to 20 samples. Per Client's Request	QC acceptance criteria 40-140% Recovery (except nonane 30%-140%). 50% RPD.	Report if associated with passing LCS/LCSD. Discuss in narrative.	Laboratory Analyst/Section Supervisor	Same as Method/SOP QC Acceptance Limits

*Internally derived limits are updated periodically; current limits will be used at the time of analysis.
**The lab EDD must identify sample QC and batch identifier to relate individual samples to the batch and QC samples.
DQI = data quality indicators; QC = quality control; RPD = relative percent difference; SOP = standard operating procedure

Worksheet #29: Project Documents and Records

Sample Collection and Field Records

Record	Generation	Verification	Storage Location/Archival
Field logbooks or data collection sheets[1]	GEI Field Team Lead	GEI Project Manager	Project File
Chain-of-Custody Forms	GEI Field Team Lead	GEI Project Manager	Project File
Shipping Records	GEI Field Team Lead	GEI Project Manager	Project File
Photographs	GEI Field Team Lead	GEI Project Manager	Project File
Custody Seals	GEI Field Team Lead	GEI Project Manager	Project File
Deviations	GEI Field Team Lead	GEI Project Manager	Project File
Corrective Action Reports	GEI Field Team Lead	GEI Project Manager	Project File
Correspondence	GEI Field Team Lead	GEI Project Manager	Project File
GPS Raw Data	GEI Field Team Lead	GEI Project Manager	Project File

On-Site Analysis Documents and Records

Record	Generation	Verification	Storage Location/Archival
Equipment Calibration Logs	GEI Field Team	GEI Quality Assurance Manager	Project File
Field Data Records	GEI Field Team	GEI Quality Assurance Manager	Project File
Field Instrument Maintenance Logs	GEI Field Team	GEI Quality Assurance Manager	Project File

Off-Site Documents and Records

Record	Generation	Verification	Storage Location/Archival
Sample Receipt, Custody, and Tracking Records	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File
Standard Traceability Logs	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File
Instrument Calibration Logs	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File
Instrument Maintenance Logs	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File
RTK and Post-Processed GPS Data	GEI Technical Team	GEI Quality Assurance Manager	Project File

Sample Preparation Worksheets/Logs	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File
Sample Analysis Worksheets/Run Logs	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File
Chromatograms/Raw Data/Instrument Printouts	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File
Sample Results/Forms	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File
Corrective Action Documentation	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File
Data Package Completeness Checklists	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File
Sample Disposal Records	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File
Raw Data	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File
Analytical Report	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File
EDDs[2]	Analytical Laboratory	GEI Quality Assurance Manager	Laboratory QA File

Data Assessment Document and Records

Record	Generation	Verification	Storage Location/Archival
Data Validation Report	GEI Data Validator	GEI Quality Assurance Manager	Project File

Other

Record	Generation	Verification	Storage Location/Archival
Health and Safety Plan	GEI Technical Team	GEI Safety Officer	Project File
Daily Safety Briefing Form	GEI Field Team Lead	GEI Project Manager	Project File
Incident Report Form	GEI Field Team	GEI Safety Officer	Project File
Near Miss Form	GEI Field Team	GEI Safety Officer	Project File
Utility Clearance Form	GEI Field Team	GEI Project Manager	Project File
Risk Assessment	GEI Field Team Lead	GEI Project Manager	Project File
Telephone/Email Logs	Various	GEI Project Manager	Project File
Corrective Action Documentation	GEI Field Team Lead	GEI Quality Assurance Manager	Project File

Laboratory Data Deliverables

Soil Samples						
Record	**VOCs**	**SVOCs**	**Metals**	**PCBs**	**Total Organic Carbon**	**Grain Size Analysis/SPT**

Narrative	X	X	X	X	X	X
COC	X	X	X	X	X	X
Summary Results	X	X	X	X	X	X
QC Results	X	X	X	X	X	X
Chromatograms	X	X				

Groundwater Samples							
Record	**VOCs**	**SVOCs**	**Total Cyanide**	**Metals**	**1,4-Dioxane**	**PCBs**	**PFAS**
Narrative	X	X	X	X	X	X	X
COC	X	X	X	X	X	X	X
Summary Results	X	X	X	X	X	X	X
QC Results	X	X	X	X	X	X	X
Chromatograms	X	X			X	X	X

Sediment Samples										
Record	**VOCs**	**SVOCs**	**TOC**	**Metals**	**Total Cyanide**	**PCBs**	**Geotech Parameters**	**Ammonia**	**Sulfide**	**Benthic Toxicity**
Narrative	X	X	X	X	X	X	X	X	X	X
COC	X	X	X	X	X	X	X	X	X	X
Summary Results	X	X	X	X	X	X	X	X	X	X
QC Results	X	X	X	X	X	X	X	X	X	X
Chromatograms	X	X	X			X				

Waste Characterization Samples											
Record	**VOCs**	**SVOCs**	**BTU**	**Metals**	**Total Cyanide**	**TPH**	**PCBs**	**Pesticides/Herbicides**	**Percent sulfur**	**pH, Ignitability, Reactivity**	**Corrosivity**
Narrative	X	X	X	X	X	X	X	X	X	X	X
COC	X	X	X	X	X	X	X	X	X	X	X
Summary Results	X	X	X	X	X	X	X	X	X	X	X
QC Results	X	X	X	X	X	X	X	X	X	X	X
Chromatograms	X	X				X	X				

1 Data collection sheets may include, but are not limited to, field sampling forms, soil boring logs, test pit logs, monitoring well installation forms, monitoring well development forms, and groundwater sampling forms.

2 Electronic data will be submitted in accordance with the GLLA Project Agreement and in a format specified by the Environmental Protection Agency (USEPA) remedial project manager. Electronic data will be uploaded to the GLLA electronic data upload submission site on GLNPO.net. Electronic files and project deliverables will be maintained in a centralized location and retained for a minimum of 10 years. In addition, electronic data will be backed up on a nightly basis. Hard copy project files and data, such as original field forms and calculations, will be stored in a centralized project file location in the GEI offices in Buffalo, NY and Raleigh, NC. Hardcopy files will be maintained for a minimum of 10 years.

Worksheet #31: Assessments and Corrective Action

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Data review	Upon Receipt of data	Internal	Analytical Laboratory	Jason Hebert/QA Lab Manager Michael Pratt/QA Lab Manager	Senior Chemist for affected laboratory procedure	Melissa Deyo/Laboratory Project Manager	Jason Hebert/QA Lab Manager Michael Pratt/QA Lab Manager
Data review	Upon Receipt of Data	External	GEI	Jennifer Bash/GEI Quality Assurance Manager[1]	Rick Frappa/GEI Project Manager	Jennifer Bash/GEI Quality Assurance Manager	Jennifer Bash/GEI Quality Assurance Manager
GPS Data Review	Upon receipt of data	External	GEI	Jennifer Bash/GEI Quality Assurance Manager[1]	Rick Frappa/GEI Project Manager	Jennifer Bash/GEI Quality Assurance Manager	Jennifer Bash/GEI Quality Assurance Manager
Field Audit	Discretionary	Internal	GEI	Jennifer Bash/GEI Quality Assurance Manager[1]	Rick Frappa/GEI Project Manager	Mike Cummings/GEI Field Team Lead	Rick Frappa/GEI Project Manager Jennifer Bash/GEI Quality Assurance Manager
	Discretionary	External	Regulatory Agency	Regulatory Agency Remedial Project Manager	Rick Frappa/GEI Project Manager	Mike Cummings/GEI Field Team Lead	Rick Frappa/GEI Project Manager Jennifer Bash/GEI Quality Assurance Manager

Assessment Type	Frequency	Internal or External	Organization Performing Assessment	Person(s) Responsible for Performing Assessment (Title and Organizational Affiliation)	Person(s) Responsible for Responding to Assessment Findings (Title and Organizational Affiliation)	Person(s) Responsible for Identifying and Implementing Corrective Actions (CA) (Title and Organizational Affiliation)	Person(s) Responsible for Monitoring Effectiveness of CA (Title and Organizational Affiliation)
Laboratory Audit	As needed based on laboratory performance	External	GEI	Jennifer Bash/GEI Quality Assurance Manager[1]	Rick Frappa/GEI Project Manager	Jason Hebert/QA Lab Manager Michael Pratt/QA Lab Manager	Rick Frappa/GEI Project Manager Jennifer Bash/GEI Quality Assurance Manager
Field Inspections	Intermittent	Internal	GEI	Mike Cummings/GEI Field Team Lead	Rick Frappa/GEI Project Manager	Mike Cummings/GEI Field Team Lead	Rick Frappa/GEI Project Manager Jennifer Bash/GEI Quality Assurance Manager
Safety Audits	Annually and Field Assessment based on > 400 field hours/ quarter	Internal	GEI	Corporate Health and Safety (H&S) Staff, Regional H&S Manager or Regional H&S Specialist	Rick Frappa/GEI Project Manager	Mike Cummings/GEI Field Team Lead	Rick Frappa/GEI Project Manager

[1]The Quality Assurance Manager will operate independently of all other entities involved with field work, laboratory analysis, and reporting. The QAM will independently review all data and documents prior to signing or approving any reports.

Worksheet #32: Assessment Findings and Corrective Action Responses

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, Organization)	Timeframe for Response
Qualitative laboratory data review	Inappropriate analytical/testing methods yield unusable result	Tanya Alexander/ National Fuel Project Manager Regulatory Remedial Project Manager	14 days after findings	In writing via e-mail	Tanya Alexander/ National Fuel Project Manager Regulatory Remedial Project Manager	As indicated in CA response
Field Sampling Technical Systems Audit	Written Audit Report	Rick Frappa/GEI Project Manager Jennifer Bash/GEI Quality Assurance Manager[1]	72 hours after audit	Letter	Rick Frappa/GEI Project Manager Jennifer Bash/GEI Quality Assurance Manager	48 hours after notification
Contract Laboratory Technical Audit (external)	Written Audit Report	Melissa Deyo/Lab Project Manager Rick Frappa/GEI Project Manager Jennifer Bash/GEI Quality Assurance Manager[1]	1 week after audit	Letter	Jason Hebert/QA Lab Manager Michael Pratt/QA Lab Manager	48 hours after notification
Field Inspection	Memorandum	Rick Frappa/GEI Project Manager Jennifer Bash/GEI Quality Assurance Manager[1]	2 days	Memorandum	Rick Frappa/GEI Project Manager Jennifer Bash/GEI Quality Assurance Manager	48 hours after notification

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (Name, Title, Organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (Name, Title, Organization)	Timeframe for Response
Contract Laboratory Technical Audit (internal)	Memorandum	Melissa Deyo/Lab Project Manager	2 days	Memorandum	Jason Hebert/QA Lab Manager Michael Pratt/QA Lab Manager	48 hours after notification

[1]The Quality Assurance Manager will operate independently of all other entities involved with field work, laboratory analysis, and reporting. The QAM will independently review all data and documents prior to signing or approving any reports.

Worksheet #33: QA Management Reports Table

Type of Report	Frequency (daily, weekly, monthly, quarterly, annually, etc.)	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation (Title and Organizational Affiliation)	Report Recipient(s) (Title and Organizational Affiliation)
Data Summary Reports	As needed	3 months after receipt of analytical data	Rick Frappa/GEI Project Manager	Client Project Manager Regulatory Remedial Project Manager EPA Project Manager
Data Validation Reports	As specified in the Data Assessment section based on intended use of the data and required percentage to meet project DQOs	As generated throughout project	Bethany Russel and Darionna Malone, GEI Data Validators	Rick Frappa/GEI Project Manager Jennifer Bash/GEI Quality Assurance Manager[1] EPA Project Manager
Field Sampling Technical Systems Audit Report	1/year	NA	Mike Cummings/GEI Field Team Lead	Rick Frappa/GEI Project Manager Jennifer Bash/GEI Quality Assurance Manager[1] EPA Project Manager
Contract Laboratory Technical Audit Report	As needed during project	NA	Bethany Russel and Darionna Malone, GEI Data Validators	Rick Frappa/GEI Project Manager Jennifer Bash/GEI Quality Assurance Manager[1] EPA Project Manager

GEI Consultants, Inc. will perform corrective action measures, including deviations from plans and procedures that are of an immediate nature that may require work stoppage in accordance with GEI's Quality Management Plan (GEI, 2018).

[1]The Quality Assurance Manager will operate independently of all other entities involved with field work, laboratory analysis, and reporting. The QAM will independently review all data and documents prior to signing or approving any reports.

Worksheet #34: Data Verification and Validation Inputs

Item	Description	Verification (completeness)	Validation (conformance to specifications)
	Planning Documents/Records		
1	Approved QAPP	X	X
2	Field SOPs	X	
3	Laboratory SOPs	X	
	Field Records		
4	Field notes and logbooks	X	X
5	Field data collection sheets	X	X
6	Instrument calibration records	X	X
7	Instrument testing and maintenance records	X	X
8	Chain-of-custody records	X	X
9	Sampling diagrams/maps	X	X
10	Drilling logs	X	X
11	Sediment core and grab logs	X	X
12	Photographs	X	X
13	Subcontractor reports	X	X
14	Communication records	X	X
15	Change orders/deviations	X	X
16	Field audit reports	X	X
17	Field CA reports	X	X
	Laboratory Data Package		
18	Cover sheet (laboratory identification, job number, etc.)	X	X[1]
19	Cross reference (field sample ID to laboratory ID)	X	X[1]
20	Case narrative	X	X[1]
21	Definition of laboratory qualifiers	X	X[1]
22	Internal and external chain-of-custodies	X	X[1]
23	Sample receipt records	X	X[1]
24	Communication records	X	X[1]
25	Sample Chronology (dates and times of sample receipt, preparation, analysis)	X	X[1]
26	Sample results, including units, sample-specific detection limits, dilution factors, laboratory qualifiers	X	X[1]
27	QC Results	X	X[1]
28	RAW data, including chromatograms and ion spectra, as applicable	X	X[1]
29	EDD	X[2]	X[1,2]

[1]Refer to Worksheet #36 for level validation for each parameter.
[2]Refer to Worksheet #29 for applicability.

Worksheet #35: Sampling and Analysis Validation (Step Iia and Iib) Process Table

Step of Validation	Validation Input	Description	Responsible for Validation (Name, Organization)
Iia	Sampling methods and procedures	Establish that required sampling procedures and field measurements meet performance criteria and that deviations were documented. Verify that sampling methods/procedures were followed (in accordance with GEI Field SOPs (Appendix A), and that deviations were noted/approved in accordance with Worksheets #31 and 32.	Jennifer Bash/GEI Quality Assurance Manager Bethany Russel and Darionna Malone, GEI Data Validators
Iib	SOPs	Determine potential impacts from noted/approved deviation, regarding project quality objectives (DQOs; Worksheets #10 and #11).	Jennifer Bash/GEI Quality Assurance Manager
Iia	Chain-of-custody	Examine chain of-custody forms against QAPP and laboratory contract requirements (i.e., analytical methods, sample identification, etc.)	Jennifer Bash/GEI Quality Assurance Manager Bethany Russel and Darionna Malone, GEI Data Validators
Iia	Laboratory data package	Examine packages against QAPP and laboratory contract requirements, and against chain-of-custody forms (i.e., holding times, sample handling, analytical methods, sample identification, data qualifiers, QC samples, etc.)	Jennifer Bash/GEI Quality Assurance Manager Bethany Russel and Darionna Malone, GEI Data Validators
Iib	Analytical method and procedures	In addition to the required review items listed in Stage 2A/2B, the following review is performed: Verification and validation checks for the compliance of instrument-related QC: initial and continuing calibration verification standard frequency and result criteria, instrument blank results, and method specific instrument performance checks.	Bethany Russel and Darionna Malone, GEI Data Validators
III (Modified)	Analytical method and procedures	Verification of instrument outputs of sample quantitation report versus laboratory reports results, and appropriateness of calibration model applied. Manual recalculations are not required.	Bethany Russel and Darionna Malone, GEI Data Validators
IV	Analytical method and procedures	In addition to the required review items listed in Stage 3, the following review is performed:	Bethany Russel and Darionna Malone, GEI Data Validators

Step of Validation	Validation Input	Description	Responsible for Validation (Name, Organization)
		Verification and validation of instrument and sample results and comparison of recalculated results to those reported and review of the actual instrument outputs is performed on a subset of data.	
Iia, Iib, III, and IV	Issue resolution	Data issues which may adversely impact the data will be reported to the GEI PM, client, regulator, and laboratory in an effort to mitigate the impact on the project data.	Bethany Russel and Darionna Malone, GEI Data Validators

Notes:
Step Iia – Compliance with methods, procedures, and contracts.
Step Iib – Compliance with QAPP quality objectives.
Guidelines for reporting data verification/validation efforts are discussed in Worksheet #37 of this QAPP.

Worksheet #36: Sampling and Analysis Validation (Stage 3) Summary Table

Validation Level	Matrix	Analytical Group	Concentration Level	Validation Criteria	Data Validator (Title and Organizational Affiliation)
Stage 3	Inclusive	Inclusive	Inclusive	Qualitative data review according to Worksheets No. 11, 12, 19, 28. Level III data validation according to Worksheet No. 37 EPA National Functional Guidelines for Organic and Inorganic Superfund Methods Data Review, EPA-540-R-2017-002 (Organic) and EPA-540-R-2017-001 (Inorganic), January 2017 Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use, EPA-540-R-08-005, January 2009	Bethany Russel and Darionna Malone, GEI Data Validators

Notes:
Guidelines on reporting data verification/validation efforts are discussed in Worksheet #37 of this QAPP.
100 percent of project chemical data will undergo data validation at Stage 3.

Worksheet #37: Data Usability Assessment

A Data Usability Assessment will be performed by GEI for all data associated with the RI investigation and FS. These usability assessments will consist of data validation checklists and overall usability summaries captured within a Data Validation Report.

The data usability assessment will be performed at the conclusion of data collection activities, using the outputs from data verification and data validation. It is the data interpretation phase, which involves a qualitative and quantitative evaluation to determine if the project data are of the right type, quality, and quantity to support the decisions to be made.

Personnel (organization and position/title) responsible for participating in the data usability assessment.

Rick Frappa, GEI Project Manager

Jennifer Bash, GEI Quality Assurance Manager

Jaimie Wargo, GEI Data Manager

Bethany Russel and Darionna Malone, GEI Data Validators

The data usability assessment process will include the following to be completed following investigation and document in the report.

Step 1	Review of the project objectives and sampling design to determine if the project data are of the right type and quantity to support the decisions that need to be made.
Step 2	Review of the field data verification and data verification and validation outputs: Verification is the process by which sampling information and laboratory data are reviewed for completeness and adherence to the project requirements. Data validation, as outlined in Worksheets #35 and #36, entails a review of the sample collection, handling, laboratory QC data, and the raw data to verify that the laboratory operated within required method and project specified limits; analytical results were correctly transcribed from the instrument read-outs; and which (if any) environmental samples were related to non-conforming QC samples and the effect (uncertainty/bias) of these results on the data.
Step 3	Data usability assessment report preparation: The data usability assessment evaluates whether the sources of uncertainty have been accounted for and are acceptable and the results can be used as intended and are sufficient to meet the project needs and data quality objectives. A data assessment report will be issued by the project manager or designee documenting the review of the project team.

Each assessment will include field data verification and laboratory data verification and validation. Data verification involves review of laboratory results to confirm that the quality control steps outlined in this QAPP, and appropriate SOPs were performed to meet the project QC objectives.

The key elements of the field data verification process will include the following:

- Field sampling, sample handling, and analysis activities documentations (e.g., signatures, QC checklists).
- Internal verification of sampling, handling, on-site analytical and off-site laboratory data at the data-generator level.

The key elements of the laboratory verification and validation process will include the following:

- Sample collection, handling, and analysis procedures.
- Laboratory data (e.g., laboratory-qualified data).
- Data package deliverable completeness.
- Case narrative review.
- Analytical data reporting.
- QC sample data summaries.
- Applicable raw data (when required).

Required data deliverables must be present in the data packages to proceed to the next step of data validation.

Data validation entails a review of the sample collection, handling, field analysis and QC data, and the raw data to verify that the laboratory operated within required limits; analytical results were correctly transcribed from the instrument read-outs; and which (if any) environmental samples were related to non-conforming QC samples. The objective of data validation is to identify suspect or invalid laboratory measurements.

The data quality indicators (DQIs) used to evaluate conformance with the project data quality objectives (DQOs) are presented below.

DQIs are generally defined in terms of seven (7) parameters:

1. Precision
2. Accuracy
3. Representativeness
4. Comparability
5. Completeness
6. Sensitivity
7. Selectivity

Each parameter is defined below. Specific objectives for the site actions are presented in other sections of this QAPP, as referenced below.

Precision

Precision is a measure of agreement among individual measurements of the same property, under prescribed conditions. Sampling precision is measured for this project through the collection of field duplicates and is expressed in terms of Relative Percent Difference (RPD). Precision is expressed in terms of analytical variability and will be calculated intra-laboratory. For this project, analytical variability will be measured as RPD between analytical replicates/duplicates (i.e., field or laboratory) and between the MS MSD analytical results. Analytical precision is measured by evaluation of MS/MSD pairs, dual column PCB results, LCS/LCSD, and/or laboratory-generated sample replicates.

The RPD is calculated using the following equations:

$$RPD = \frac{(C1-C2)*100\%}{(C1+C2)/2}$$

Where:

C1 = The larger of the two observed values
C2 = The smaller of the two observed values.

The RPDs will be reviewed during the data quality review, deviations from specified limits will be notes, and the effect on reported data will be commented upon by the data reviewed.

Accuracy

Accuracy is the amount of agreement between the measured value and the true value. It will be measured as the percent recovery (%R) of the MS/MSD, LCS, and surrogate spike compounds. It will also be measured using the analytical results of instrument calibration and other laboratory internal standards.

Accuracy will be calculated as the %R of analytes as follows:

$\%R = (y/x) *100$

Where:

%R = percent recovery

Y = measured analyte concentration (measured – original sample concentration)

X = known analyte concentration

The resultant percent recoveries will be compared to acceptance criteria, and deviations from specified limits will be reported. If the objective criteria are not met, the laboratory will supply a justification of why the acceptability limits were exceeded and implement the appropriate corrective actions. Percent recoveries will be reviewed during the data quality review, deviations from the specified limits will be noted, and the effect on reported data will be comments upon by the data reviewer.

Representativeness

Representativeness is the degree to which data is accurately and precisely represents a parameter variation at a sampling point or an environmental condition. Results from analyses will be used to evaluate the data to determine whether the samples were collected in a manner that the results appropriately describe the area investigated.

Comparability

Comparability is the degree to which data from one study can be compared with data from other similar studies, reference values (such as background), reference materials, and screening values. This goal will be achieved by using standard techniques to collect and analyze representative samples, reporting analytical results in appropriate units, and comparing past and future results.

Completeness

Completeness is defined as the percentage of usable data from the total amount of data generated. Analytical completeness is a measure of the number of overall accepted analytical results (valid results), including estimated values, compared to the total number of analytical results requested on samples submitted for analysis after review of the analytical data. Less than 100% completeness could result if samples require dilution, resulting in an increase in project-required detection/quantitation limits for some parameters. Highly contaminated environments can also be sufficiently heterogeneous to prevent the achievement of specified precision and accuracy criteria. Therefore, the target goal for completeness as a whole shall be 90% for both field and laboratory analytical methods. Completeness is calculated on a per sample delivery group basis for this project. Project-specific data needs will be defined on an individual batch basis and will consist of data for which all QC criteria were net.

Completeness will be calculated as follows:

$$\%C = (A/I)*100\%$$

Where:

%C = percent completeness (analytical)
A = actual number of samples collected/valid analyses obtained
I = intended number of samples/analyses requested

Invalid data (i.e., data qualified as "R" rejected) will be identified during the QA review.

Sensitivity

As used in this context, sensitivity refers to the ability of project analytical procedures to identify and quantify target analytes at concentrations low enough to meet project data needs. Specific indicators of sensitivity in analytical measurements include the method detection limit (MDL) and the reporting limit (RL) for organics and inorganics.

The MDL is a purely statistical value, which is defined by EPA as the concentration at which an analytical system has a 99 percent probability that the analyte is present, but there is a high probability of false non-detections at or around the MDL. MDLs are determined by preparation and analysis of a minimum of seven replicate portions of a low-level standard. EPA revised their definition of MDLs in December 2016 (EPA, 2016) to: The MDL is defined as the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results. Per the new definition (and procedure), the MDL is determined using multiple replicates of low-level standards and method blanks throughout the year; the MDL is higher of the two values; the MDL lies in a region of high quantitative uncertainty; and results near the MDL must be considered as estimates.

The RL is normally set at a factor of 5 to 10 times the MDL. The exact number depends on the lowest concentration that a laboratory can successfully use as a low calibration standard. The RL is considered the lowest concentration that a laboratory can report with reasonable quantitative accuracy, although results less than 5 times the RL can still be highly variable.

Selectivity

Selectivity is the ability of an analytical procedure to accurately identify an analyte, and to distinguish that analyte from interferences. The complexity of the project sample matrix and the presence of multiple, closely related compounds make selectivity an important data quality issue for this project. So that project data needs are met, the subcontracted laboratories will discuss compound identification issues with the designated project personnel as soon as they are identified.

Data Validation and Usability

GEI will validate data generated referencing the most recent versions of the EPA National Functional Guidelines (EPA, 2020) where applicable. These procedures and criteria may be modified, as needed, to address project-specific method-specific criteria, control limits, and procedures. Data validation will consist of data screening, checking, reviewing, editing, and interpretation to document analytical data quality and to determine whether the quality is sufficient to meet DQOs.

The data validator will verify that reduction of laboratory measurements and laboratory reporting of analytical parameters is in accordance with procedures specified for the analytical method and/or specified in this QAPP. Deviations from the analytical method or special reporting requirements apart from those specified in this QAPP will be detailed on COC forms.

Upon receipt of laboratory data, the following information will be checked by the data validator:

- Evaluate completeness of data package.
- Verify that field COC forms were completed and that samples were handled properly.
- Verify that holding times were met for requested parameters. Holding time exceedances, should they occur, will be documented. Data for samples exceeding holding time requirements will be flagged as either estimated or rejected. The decision as to which qualifier is more appropriate will be made on a case-by-case basis.
- Verify that parameters analyzed according to the methods specified.
- Review QA/QC data (i.e., confirm that duplicates, blanks, and spikes were analyzed on the required number of samples as specified in the method, and verify that duplicate and MS recoveries are acceptable).
- Investigate anomalies identified during review. When anomalies are identified, they will be discussed with the Project Manager and/or Laboratory Manager, as appropriate.
- If data appear suspect, investigate the specific data of concern. Calculations will be traced back to raw data. If calculations do not agree, the cause will be determined and corrected.

Deficiencies discovered as a result of the data review, as well as the corrective actions implemented in response, will be documented and submitted in a written report addressing the following topics, as applicable to the methods:

- Assessment of the data package.
- Description of protocol deviations.
- Failures to reconcile reported and/or raw data.
- Assessment of compromised data.
- Overall appraisal of the analytical data.
- Table of site name, sample quantities, matrix, and fractions analyzed.

Quantified results do not necessarily invalidate data. The goal to produce the best possible data does not necessarily mean that data must be produced without QC qualifiers. Qualified data can provide useful information.

During the validation process, laboratory qualified, and unqualified data are verified against the supporting documentation. Based on this evaluation, validation qualifiers are applied to the project data. Examples of codes that will be used to qualify results include:

U	The analyte was analyzed for but was not detected above the reported sample quantitation limit.
J	The result was positively identified; however, the associated numerical value is an estimated concentration only.
J+	The analyte was positively identified; the associated numerical value is an approximate concentration of the analyte but may be biased high.
J-	The analyte was positively identified; the associated numerical value is an approximate concentration of the analyte but may be biased low.
JN	The analysis indicates the presence of a compound for which there is presumptive evidence to make a tentative identified. The associated numerical value is an estimated concentration only.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a tentative identification.
R	The data are unusable. A sample result is rejected when associated QC analysis results exceed the expanded control limits of the QC criteria. The rejected data are known to contain significant errors based on documented information. The data user must not use the rejected data to make environmental decisions. The presence or absence of the analyte cannot be verified.
UJ	The analyte was not detected. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation.

Resolution of issues regarding laboratory performance or deliverables will be handled between the laboratory and the data validator. Suggestions for re-analysis may be made by the QA lead.

Validation Reports

The data validation reports will identify deficiencies and the potential impact on the results. The Validation and Data Project team will amend qualifiers generated during the validation process to the database. The validation checklists and the database will be the primary location of applicable data qualifiers.

Qualifiers will not be applied to the hard copy analytical reports.

Field Data Review

Field data are generated from in-field measurements, which may include geophysical survey activities, water quality monitoring, and air quality monitoring. The quality objective for the in-field measurement activities is to obtain accurate measurements of sample characteristics, including pH, conductivity, temperature, turbidity, dissolved oxygen, and/or redox potential, using appropriate equipment. Data are recorded in field logbooks or on field sampling sheets and calibration logs.

Calibration logs will be reviewed with other field documentation to identify potential impacts to data quality and usability.

Field logbooks/field forms are reviewed as part of the QC inspections.

Reconciliation with Data Usability Requirements

The data results will be examined to determine the performance that was achieved for the data usability criteria. The performance will then be compared with the project objectives and DQOs. Deviations from objectives will be noted. Additional action may be warranted when performance does not meet performance objectives for critical data. Options for corrective action relating to incomplete information, questionable results, or inconsistent data may include the following:

- Retrieval of missing information.
- Request for additional explanation or clarification.
- Reanalysis of sample from extract (when appropriate).
- Recalculation or reinterpretation of results by the laboratory.

These actions may improve data quality, reduce uncertainty, and eliminate the need to qualify or reject data. If these actions do not improve the data quality to an acceptable level, the following actions may be taken:

- Extrapolation of missing data from existing data points.
- Use of historical data.
- Evaluation of the critical/noncritical nature of the sample.

If the data gap cannot be resolved by these actions, the data bias and potential for false negatives and positives can be evaluated. If the resultant uncertainty level is unacceptable, the following action must be taken:

- Additional sample collection and analysis.

References

The following is a list of relevant work plans and reports cited in the introduction to this QAPP. Additional sources for secondary information are listed in Worksheet #13.

Empire GeoServices, Inc. (2015). "Sediment Sampling – Scajaquada Creek Slip behind 1654 Niagara Street," Prepared for NYSDEC. February 2015.

Empire GeoServices, Inc. (2017). "Summary Report for Sediment Screening Assessment Within the Lower Scajaquada Creek Corridor and Black Rock Canal," Prepared for NYSDEC. June 2017.

GEI Consultants, Inc. (2018). "GEI Quality Program," Prepared for GEI Consultants, Inc. March 2018.

GEI Consultants, Inc. (2025). "DRAFT Remedial Investigation/Feasibility Study Work Plan," Prepared for the Scajaquada Creek Operable Unit RI/FS- GLLA Program Site. February 2025.

GEI Consultants, Inc. (2022). "Site Characterization Report – Former Buffalo Gaslight Iroquois Corporation Site and Lower Scajaquada Creek," Prepared for National Fuel Gas Distribution Corporation. June 2022; Revised October 2022.

GEI Consultants, Inc. (2023). "Pre-Design Investigation and Interim Remedial Measures Report – Lower Scajaquada Creek City of Buffalo Pumphouse," Prepared for National Fuel Gas Distribution Corporation. March 2023.

Labella Associates (2016). "Phase II Environmental Site Assessment, 1660 Niagara Street," Prepared for Buffalo Niagara Riverkeeper et al. February 2016.

USEPA. (2005a). Intergovernmental Data Quality Task Force. Uniform Federal Policy for Quality Assurance Project Plans: Evaluating, Assessing, and Documenting Environmental Data Collection and Use Programs. Part 1: UFP-QAPP Manual. Version 1, March 2005. Publication Numbers: USEPA: EPA-505-B-04-900A, Department of Defense: DTIC ADA427785. March.

USEPA. (2005b). Intergovernmental Data Quality Task Force. Uniform Federal Policy for Quality Assurance Project Plans. Part 2B, Quality Assurance/Quality Control Compendium: Minimum QA/QC Activities. Version 1, March 2005. Publication Numbers: USEPA: EPA-505-B-04-900B, Department of Defense: DTIC ADA 426957. March.

USEPA. (2012). Intergovernmental Data Quality Task Force. Uniform Federal Policy for Quality Assurance Project Plans: Optimized UFP-QAPP Worksheets. USEPA: EPA-2106-G-05. March.

USEPA. (2016). "U.S. EPA National Functional Guidelines for High Resolution Superfund Methods Data Review." EPA-542-B-16-001. OSRTI. Washington, D.C. April.

USEPA. (2020). "U.S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review." EPA-542-R-20-006. OSRTI. Washington, D.C. November.

USEPA. (2020). "U.S. EPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review." EPA-540-R-20-005. OSRTI. Washington, D.C. November.

Appendix A GEI Field SOPs

STANDARD OPERATING PROCEDURES

AR-001 General Guidance on Work Zone Monitoring Methods

1. Objective

Protect human health by measuring air quality at the perimeter of a work area.

2. Execution

Walk-around perimeter and work zone monitoring for Total Volatile Organic Compounds (TVOC), Respirable Particulate matter less than 10 microns (RPM_{10}), and odor will occur along the perimeter of the project site on a regular and as-needed basis. Specific site conditions that will trigger walk-around perimeter or work zone monitoring include:

- visible dust
- odor complaints
- detection of TVOCs and/or RPM_{10} at levels approaching or exceeding action levels
- direction by the site oversight consultant or client

Perimeter air monitoring and work zone monitoring, in the absence of any specific triggering criteria, will be conducted on a regular basis during the normal work day. Particular attention will be given to the direction of any residences or other sensitive receptors.

At the time when a triggering condition is observed, the walk-around perimeter and work zone monitoring will occur continuously until the conditions that triggered the monitoring have subsided. Additional temporary monitoring points may be established due to changing site or meteorological conditions.

TVOC concentrations will be monitored and recorded using a Rae Systems MiniRAE 2000 Portable Ionization Detector (PID) or equivalent. RPM_{10} will be measured and recorded using a MIE personal DataRAM 1200 (PDR-1200) portable real-time aerosol monitor equipped with a PM-10 monitor and Gilian personal air sampling pump. AR-002 Suspended Particulate Matter in Ambient Air using the MIE DataRAM Real-time Aerosol Monitor (Portable) provides guidance on the use of this meter. Odors will be noted based on the n-butanol scale, as adapted from ASTM E544-99.

At each monitoring point, the 15-minute average value of TVOC and RPM_{10}, sample time, and sample location will be collected and recorded. The odor intensity based on the n-butanol scale will be monitored over a 15-minute period and recorded. At each location, air temperature, wind direction, and wind speed may be monitored and recorded using a hand-held wind meter.

Odors as a function of naphthalene concentration will be monitored over a 15-minute period and recorded. To measure naphthalene concentrations, the zNose™ Model 4200 system will be used. The zNose™ is an ultra-fast gas chromatograph (GC) that is capable of analyzing airborne concentrations of VOCs and SVOCs in less than one minute. The zNose™ uses a surface acoustic wave (SAW) detector that changes in vibration frequency as compounds elute from the column and condense onto the surface of the detector.

The zNose™ is a portable instrument and will be positioned downwind of the remedial activities. Up to five samples will be analyzed for naphthalene concentrations over a 15-minute period. The concentrations will be averaged to produce a 15-minute result. The calibration will be checked at the start of the day, at mid-day, and at the end of the day. An air blank will be run every two hours. A blank will also be run if a reading exceeds the calibration range of the instrument.

The zNose™ also has the capability of generating fingerprint images of the chemical constituents in the vapor called VaporPrints™. A VaporPrint™ of an air sample can be collected through a headspace analysis. This VaporPrint™ can be compared to others generated at the perimeter and off-site to see if remedial operations are the source of the odors. VaporPrints™ can allow for identification of odors that may not be affiliated with remedial operations.

To monitor cyanide (as hydrogen cyanide gas), a real-time hand-held meter in conjunction with the Dräger Chip Measuring System (CMS) will be used. Types of continuously monitoring equipment include the V-RAE by Rae Systems and the Mini-Warn by Dräger Safety Systems and are available from rental equipment suppliers. Due to potential interference from sulfur compounds, hydrogen sulfide gas (H_2S) will also be monitored for comparison to the hydrogen cyanide gas levels detected. Hydrogen cyanide gas detections will also be confirmed with CMS Dräger tubes due to this interference. The Dräger CMS can quantify other gases that could potentially provide false positives for hydrogen cyanide gas (including sulfur dioxide, hydrogen sulfide, phosphine gas, chlorine, and nitrogen dioxide) detected by the real-time meter.

At each location, air temperature, wind direction, and wind speed will be monitored and recorded using a hand-held wind meter.

3. References

New York State Department of Environmental Conservation. 2010. DER-10 / Technical Guidance for Site Investigation and Remediation. Division of Environmental Remediation. May 3, 2010. Appendix 1A. pp. 204-206

ASTM E544 - 99(2004) Standard Practices for Referencing Suprathreshold Odor Intensity

4. Attachments

None

5. Contact

Brian Skelly

STANDARD OPERATING PROCEDURE

AR-002 Air Sampling for Dust (Particulate Matter) using the MIE DataRAM™ Real-Time Aerosol Monitor

1. Objective

Describe standard procedures for the real-time monitoring of airborne particulate matter using a MIE DataRAM™ model DR-2000 real-time aerosol monitor.

The MIE DataRAM™ is a real-time, portable monitor that measures airborne particulate matter. It is capable of monitoring total suspended particulate matter, particulate matter less than 10 microns (PM_{10}), and particulate matter less than 2.5 microns ($PM_{2.5}$) by using an appropriate orifice to control the size of the particles being measured. The DataRAM can be programmed to collect continuous real-time data, or to record time averaged data.

2. Materials

- MIE DataRAM™ model DR-2000
- Shelter/Enclosure - a pre-constructed enclosure capable of protecting the instrumentation from severe weather conditions during sample collection
- Omnidirectional inlet
- Inlet heater
- $PM_{2.5}/PM_{10}$ impactor
- IBM compatible computer loaded with MIE DataRAM™ software
- Field notebook

3. Meter Calibration

- Assemble all necessary DataRAM™ attachments.
- Make sure the power selector switch on the rear panel is in the “on” position. Turn on the power by pressing the “on” button on the front display panel of the DataRAM™. “Main Menu I” will appear on the screen.
- Activate the zero mode by pressing the button indicating zero.
- When the screen indicates the zero mode is complete, activate the span check mode by pressing the button indicated, and follow the prompts that appear on the screen.
- Record the time of the zeroing and the calibration difference percent in the field notebook. If the zeroing procedure takes longer than 5 minutes press “exit” and then “off,” and then put the power switch on the rear panel of the instrument in the “off” position. Wait several minutes, turn on the power and zero the instrument again. If the calibration difference percent is more than ±5% then follow the “Calibr Diff” Resetting Procedure in the DataRAM™ Instruction Manual.
- Set the DataRAM™ to automatically log data. Refer to the Instruction Manual for details on how to set the data logging function.

- Record the instrument flow rate from the parameters menu in the field notebook.

4. Sample Collection

- Set the DataRAM™ at the predetermined sampling location in a rain or weatherproof containment, with only the inlet tubing exposed to ambient air.
- Start the run.
- When sampling is completed, terminate run and download data using the RS-232 cable connector and the MIE DR-COM software.
- Once the data file has been successfully downloaded and saved in an appropriate location, clear the data from the instrument memory.

5. Limitations

Each instrument must be calibrated using the internal reference standard and zeroed at the start of each sampling event and at a frequency of once per day throughout the duration of the sampling event.

The DataRAM™ can be programmed to collect continuous real-time data, or can collect "averaged" real-time data. These various options should be addressed prior to field operations and must be referenced in an approved work plan. This work plan must be available to all field personnel.

Project objectives will usually dictate the sampling location. In general, for air monitoring, meters are usually placed near breathing height and away from objects that can interfere with air motions. Since the DataRAM™ is measuring particulate matter, placement of the instrument directly on the ground should be avoided to prevent the sampling of dust concentrations that may not be representative of the air that is intended to be sampled (e.g. air at breathing height).

6. References

Code of Federal Regulations, 40 CFR 50, Appendix J, Reference Method for the Determination of Particulate Matter as PM_{10} in the Atmosphere.

Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Quality Monitoring Program, US Environmental Protection Agency, Office of Air Quality Planning and Standards, Washington, D.C. EPA-454/B-08-003 December, 2008.

7. Contacts

Brian Skelly
Mark Ensign

STANDARD OPERATING PROCEDURE

AR-005 Hydrogen Cyanide Work Zone Air Monitoring Procedures

1. Objective

Describe real-time monitoring of hydrogen cyanide gas during field activities.

2. Equipment

To monitor cyanide (as hydrogen cyanide gas), the GEI field representative should utilize a real time handheld meter in conjunction with the Dräger Chip Measuring System (CMS) during subsurface activities including subsurface excavations, borings and monitoring well installation, materials handling, and groundwater sampling in areas with confirmed or suspected cyanide impacts.

Continuous monitoring equipment includes the V-RAE by Rae Systems and the Mini-Warn by Dräger Safety Systems, and are available from rental equipment suppliers. Due to potential interference from sulfurs, hydrogen sulfide gas (H_2S) should also be monitored for comparison to the hydrogen cyanide gas levels detected. Hydrogen cyanide gas detections will also be confirmed with CMS Dräger tubes due to this interference. The Dräger CMS can quantify other gases that could potentially provide false positives for hydrogen cyanide gas (including sulfur dioxide, hydrogen sulfide, phosphine gas, chlorine, and nitrogen dioxide) detected by the real time meter.

3. Calibration

Prior to commencing work on-site, the real-time cyanide meter should be calibrated in accordance with the equipment manufacturer's specifications. If the meter is calibrated in the field, the daily calibration results should be recorded in the field notebook.

4. Execution

Cyanide will be monitored around the perimeter of the work zone on a regular basis. Continuous monitoring should be completed every fifteen minutes if sulfur odor or suspected purifier waste material (former MGP sites only) is encountered. Measurements should be monitored in the breathing zone and should be recorded into the field notebook or on an applicable form. In the event that hydrogen cyanide is detected, the GEI field representative should proceed as follows:

4.1. Action Level: HCN ≤1 ppm for 15-minute average using real time meter

- Run CMS Dräger tube.
- Continue monitoring with real time meter.
- Continue work if CMS Dräger tube for hydrogen cyanide reads <2 ppm.

4.2. Action Level: 1 ppm < HCN < 2ppm for 15-minute average using real time meter.

- Run CMS Dräger using hydrogen cyanide gas chip and confirm <2 ppm concentration.
- Continue monitoring with real time meter.
- Run CMS Dräger tube using sulfur dioxide, hydrogen sulfide phosphine chip to evaluate potential interference.
- Recalibrate the real time meter and continue to monitor the work zone.

4.3. Action Level: HCN 2 ppm on CMS Dräger tube

- Stop work and move (with continuous monitoring meter) at least 25 feet upwind from excavation or until continuous monitoring meter registers <1 ppm.
- Run CMS Dräger hydrogen cyanide chip and re-evaluate activities
- Continue monitoring with real time meter.
- Allow area to ventilate and continue to monitor while returning to the work zone.
- Do not move into an area when readings are >1 ppm without confirming with additional CMS Dräger measurement.
- May resume work if Dräger tube for cyanide reads <2 ppm.

5. Limitations

No air purifying respiratory protection is available for hydrogen cyanide gas.

The American Conference of Industrial Hygienists (ACGIH) Threshold Limit Value (TLV) for Hydrogen Cyanide is 4.7 ppm.

6. References

Code of Federal Regulations, 40 CFR 50, Appendix J, Reference Method for the Determination of Particulate Matter as PM_{10} in the Atmosphere.

Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II: Ambient Air Specific Methods (Interim Edition), Addendum to Section 2.11 Reference Method for the Determination of Particulate Matter as PM10 in the Atmosphere (High-Volume Sampler Method), US Environmental Protection Agency, Office of Research and Development, Washington, D.C. EPA/600/R-94/038b April 1994.

7. Contacts

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Ryan Hoffman

STANDARD OPERATING PROCEDURE

AR-006 Air Sampling for Volatile Organic Compounds (VOCs) using Summa Canisters

1. Objective

Describe standard procedures for the collection of ambient air samples to be analyzed for volatile organic compounds (VOCs) using Summa canisters. Typically, U.S. EPA Method TO-15 is used for laboratory analysis. The site-specific Work Plan should be consulted for proposed sample locations and sampling duration.

2. Materials

- Sampling canister
- Flow controller
- Vacuum gauge
- Wrench for removing fittings and assembling the sample train
- Data sheets for recording the sampling location, date, duration, starting and stopping times, and calculated sample volume
- Camera and measuring tape
- Weather station data
- PID

3. Sampling

This section details the sampling methodology and the media preparation by the analytical laboratory.

3.1. Sampling Equipment Overview

- The laboratory prepares the canister for sampling by cleaning and then evacuating the contents to a vacuum of approximately 29.9 inches of Mercury (in. Hg). Opening the stainless steel bellows valve allows the air sample to enter the canister. When the target volume of sample is collected, close the valve and return the canister to the laboratory.
- A flow controller is used as part of the sample train to control the amount of air allowed to flow into the container over time. Flow controllers are typically set to a flow rate that collects a sample continuously over a 1-hour (hr), 8-hr, or 24-hr interval. The sampling duration needs to be communicated to the laboratory prior to sampling, so that the laboratory can provide the appropriate flow controller.
- Summa canisters are typically used and named after the “Summa” process which describes the electro polishing of the interior surface of the canister to prepare it for sampling.
- The holding time for a standard VOCs list of EPA Method TO-15 is 30 days after sample collection, although some projects may require a shorter hold time.

3.2. Document Field Conditions

Document pertinent field conditions prior to sample collection:

- Record weather information, if available (such as precipitation, temperature, barometric pressure, relative humidity, wind speed, and wind direction) at the beginning of the sampling event. Record substantial changes to these conditions that may occur during the course of sampling. The information may be measured with on-site equipment or obtained from a reliable source of local measurements (e.g., a local airport). Data should be obtained for at least the past 12 hours.
- Sketch the site, area streets, neighboring commercial or industrial facilities (with estimated distance to the site), outdoor air sampling locations (if applicable), and compass orientation (North).
- Record pertinent observations, such as odors and readings from field instrumentation.

3.3. Sample Collection

- Collect samples in a clean Summa canister (or equivalent) using a flow controller calibrated for the anticipated sample duration (e.g. 8-hour, 24-hour, etc.). The flow controller flow rate should not exceed 0.2 liters per minute.
- Verify the initial vacuum of the canister using the vacuum gauge. If the canister vacuum is less than 25 in. Hg, do not use it. The procedure to verify the initial pressure is simple, and a missed step can compromise the validity of the sample media.
    i. Confirm the canister's bellows valve is closed by turning the knob clockwise to tighten.
    ii. Remove the brass cap from the canister inlet.
    iii. Attach the vacuum gauge.
    iv. Open and close the bellows valve quickly (a few seconds).
    v. Read and record the vacuum on the gauge as 'Initial vacuum' on the chain-of-custody (COC).
    vi. Confirm the bellows valve is closed by turning the knob clockwise to tighten.
    vii. Remove the vacuum gauge and replace the brass cap.
- <u>Begin Sampling</u>
    i. Confirm the bellows valve is open by turning the knob counter-clockwise to loosen.
    ii. Remove the brass cap.
    iii. Attach the flow controller.
    iv. Attach a "J"-shaped sampling cane to prevent precipitation from entering the canister.
    v. Place the canister at the sampling location open the bellows valve. If the sample is collected from breathing height (e.g., 3 to 5 feet above ground), then mount the canister on a stable platform such that the sample inlet should be at the proper height.

vi. Record the start date and time on the COC.
vii. Record the identification numbers for the canister and flow controller and the vacuum gage.

- Begin Sampling (with a field duplicate)
  i. Confirm the bellows valve is closed by turning the knob clockwise to loosen on both canisters.
  ii. Remove the brass cap from both canisters.
  iii. Attach a flow controller on both canisters.
  iv. Attached a "T"-shaped sample train designed for field duplicates to both canisters.
  v. Attach a "J"-shaped sampling cane to the common end of the sampling "T" to limit precipitation entering the canisters.
  vi. Place the attached primary and duplicate canisters at the sampling location open the bellows valve. If the sample is collected from breathing height (e.g., 3 to 5 feet above ground), then mount the canister on a stable platform such that the sample inlet should be at the proper height.
  vii. Record the start date and time on the COC.
- Monitoring Sample progress
  i. At regular intervals, record the vacuum on the flow controller to confirm that the vacuum is decreasing in the canister. If the vacuum reads 5 in. Hg or less the bellows valve should be closed and the sample interval ended.
  ii. Some residual vacuum is important to maintaining sample integrity. If there is no vacuum remaining, call the laboratory and discuss the sample viability with them. Evaluate whether another sample will be taken after sharing the laboratory's opinion with your project manager.
- End Sampling
  i. Sampling will end when the time interval (e.g., 8-hr period) is completed, or when the canister vacuum reads 5 in. Hg or less.
  ii. Close the bellow valve by turning the knob clockwise to tighten.
  iii. Remove the "J"-shaped sampling cane.
  iv. Remove the flow controller.
  v. Attach the vacuum gauge.
  vi. Open and close the bellows valve quickly (a few seconds).
  vii. Read and record the vacuum on the gauge as 'Final vacuum' on the chain-of-custody (COC).
  viii. Confirm the bellows valve is closed by turning the knob clockwise to tighten.
  ix. Remove the vacuum gauge and replace the brass cap.
- Sample Transport
  i. Return the canister, flow controller, and sampling cane to the laboratory in the boxes provided.
  ii. Fill out the COC and relinquish samples properly with flow controller and canister numbers on the COC.

iii. Place the COC in the box and retain a copy of the COC for your records.
iv. Tape the box shut.
v. Deliver or ship the samples to the laboratory as soon as practical to adequately meet the holding time of the sample.

4. General Guidance

- This method may be modified for indoor air sampling.
- Field Blank: Do not collect a field blank.
- Trip Blank: Do not collect a trip blank. The canister is prepared for sampling by evacuating the contents to a vacuum of, so no air exists for a trip blank to provide meaningful information.

5. References

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-15, Determination of Volatile Organic Compounds (VOCs) in Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/ Mass Spectrometry (GC/MS), US Environmental Protection Agency, Office of Research and Development, Center for Environmental Research Information, Cincinnati, OH, EPA/625/R-96/010b, January 1999.

Guide To Air Sampling & Analysis, Air Toxics, Ltd., Folsom, CA.

6. Contacts

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STANDARD OPERATING PROCEDURES

DM-001 General Guidance on Determination of Appropriate Drilling Methods

1. Objective

There are multiple drilling methods which can be employed based on the type of stratum (e.g. overburden or bedrock) and the end use of borehole. End uses include geotechnical investigation, subsurface soil sampling, and monitoring well installation or a combination thereof.

The following text describes different methods of drilling with considerations for their use to collect groundwater and/or subsurface soil samples. Profiles of subsurface conditions encountered and well installation details must be recorded on logs. Procedures for field documentation are provided in Section 4 - Field Documentation.

2. Hollow-Stem Augers (HSAs)

Borings can be installed in unconsolidated formations using solid-stem or hollow-stem augers (HSAs). The augers are advanced by rotation and the drill cuttings are brought to the surface by travelling up the outside of the auger flights in a screw-like manner. HSAs have the advantage of allowing the well to be installed inside the hollow stem of the auger, which prevents the borehole from collapsing. Upon reaching the planned well depth, the casing and screen are placed inside the HSAs and the flights are individually removed while the annular space around the well is filled with the filter pack and grout, as appropriate. Conversely, solid-stem augers must be completely removed from the borehole before well installation, which can lead to collapse of the borehole. For this reason, solid stem augers are seldom used for installation of monitor wells.

HSAs come in a variety of sizes and allow collection of soil samples utilizing split spoons or Shelby tubes. Samples are collected ahead of the augers for determining soil/sediment type, stratigraphy, depth to the water table, and for collecting soil samples for chemical analysis. During this process, the standard penetration test (SPT, ASTM Method D 1586) can also be performed. The HSA method also has an advantage over mud-rotary drilling techniques in that drilling mud is not used. Drilling mud can contaminate the soil samples and potentially reduce the yield of the wells.

A disadvantage of the method is that HSAs cannot be used to drill into competent bedrock or through large boulders. Also, "heaving or running sands" can be forced up inside the augers as a result of strong vertical groundwater gradients, which can hamper efforts to collect soil samples or complete well installation. Furthermore, the maximum depth achievable using HSAs, which is generally shallower than other methods, is dependent not only on the ability of the rig (e.g., horsepower, rig-torque, weight of augers etc.), but also the lithology of the material drilled.

3. Rotary Drilling

Rotary drilling methods include both direct rotary and reverse-circulation rotary. Direct rotary is more commonly used in environmental investigations, whereas reverse-circulation rotary is used in drilling large-diameter water supply wells. In direct rotary drilling the borehole is advanced by rotating the drill pipe (rods) and bit to produce a cutting action. The cuttings are removed from the borehole by continuous circulation of a drilling fluid. The fluid or "mud" is pumped down the inside of the drill pipe and is circulated back to the surface on the outside of the pipe. The fluid removes the drill cuttings from the borehole and cools and lubricates the bit. Mud used during direct rotary consists of additives (e.g., bentonite), water, or air.

Reverse-circulation rotary drilling is similar to direct rotary except the drill rigs are larger and the flow of the drilling fluid is reversed. The drilling fluid moves upward inside the drill pipes and circulates back to the borehole via settling pits. The drilling fluid returns to the borehole via gravity and moves downward in the annular space between the drill pipe and borehole wall. Drilling fluids for reverse-circulation rotary are generally water and any suspended particles picked up from the surrounding formations.

Mud-rotary methods can be used to drill in both unconsolidated and consolidated (bedrock) formations. In addition, drilling mud stabilizes the borehole and limits the potential for borehole collapse. Disadvantages of using the mud-rotary method include the difficulty in determining the depth to the water table, the potential for drilling mud to impact soil samples and dragging of contamination into deeper zones since the drill cuttings are re-circulated in the borehole. Wells installed using this method typically take longer to develop than wells installed using the HSA or air-rotary methods due to the invasion of mud filtrate into the formation.

In air-rotary drilling, compressed air is directed down the inside of the drill pipe. As in mud-rotary drilling, air removes the cuttings and lubricates the bit. However, since air has no viscosity, it cannot be used to stabilize a borehole therefore, casing must be advanced in unconsolidated formations to keep the borehole open. This is why air rotary methods are best suited for drilling in bedrock formations. The percussion-type air-rotary "hammer" bit provides the best penetration rate when drilling bedrock consisting of crystalline rock. However, when drilling above the water table, an air-rotary bit can grind the soil and bedrock to a fine powder which is blown out of the hole with air and which has the potential to be inhaled. Therefore, drilling above the water table using air-rotary methods requires the addition of potable water to the borehole for dust control. In addition, the air compressor should be of the oil-less variety, or have a filter to prevent any oil from entering the borehole.

A disadvantage of using rotary methods while drilling in unconsolidated formations is the requirement of pulling the drill pipe out of the hole each time a split-spoon soil sample is collected (and the SPT is performed). This adds up to considerable amounts of time when deep wells are being installed or when continuous split-spoon

sampling is being performed. As stated above, split-spoons used to collect soil samples can become contaminated when they are advanced down a mud-filled borehole.

A special type of rotary drilling is bedrock coring, wherein a special core bit and barrel are used to retrieve relatively undisturbed core samples of the bedrock. Coring allows better characterization of bedrock lithology and other features including orientation of fractures and bedding planes, which can control contaminant migration. Core barrels can either be unoriented or oriented. An oriented core is scribed with respect to magnetic north. Although more expensive than collecting an unoriented core, this method gives the true orientation of the features encountered in the core.

Drilling fluids are generally air (air-rotary) or bentonite and/or water (mud-rotary). Water added to a borehole must be of potable quality. The source of the potable water used during the installation (and development) of monitor wells should be documented (e.g., in the Remedial Investigation Report).

Bentonite is high swelling clay with sodium montmorillonite as its primary clay mineral. Bentonite is added to water to increase the viscosity of the drilling fluid so that drill cuttings can be removed from the borehole more effectively. At the same time, the viscosity must be low enough to allow cuttings and coarse-grained particles to settle out once they are circulated out of the hole. Bentonite also adds weight to the drilling fluid, which helps to maintain borehole stability.

4. Sonic Drilling

The method involves driving a core barrel using vibration, rotation, and a downward force to collect soil samples. A sonic drill rig looks and operates very much like a conventional top-drive rotary or auger rig. The main difference is that a sonic drill rig has a specially designed, hydraulically powered drill head or oscillator, which generates adjustable high-frequency vibrational forces. The oscillator uses two eccentric, counter-rotating balance weights or rollers that are timed to direct 100 percent of the vibrational energy at 0 and 180 degrees. There is an air spring system in the drill head that insulates or separates the vibration from the drill rig itself. The sonic head is attached directly to the drill pipe or outer casing, sending the high-frequency vibrations down through the drill pipe to the bit.

A core barrel is advanced using vibration, rotation, and downward force to collect continuous soil cores up to 20 feet in length. The bit at the end of the core barrel contains carbide teeth allowing the core barrel to be advanced through most overburden, soft bedrock, and minor obstructions such as bricks and boulders. Once the core barrel has been advanced, a secondary or "over-ride" casing is advanced down to the same depth as the inner core barrel. The over-ride casing keeps the borehole from collapsing while the inner core barrel is removed. Once the core barrel is removed, the soil core is pushed out of the core barrel through the use of

vibration and either air or water pressure. Soil core diameters are dependent on the size of core barrel used and range from 3 to 12 inches. The use of multiple over-ride casings of increasing diameter allows the borehole to be telescoped down through multiple confining units. The setup used in sonic drilling makes this drilling method amendable to collecting soil cores and installing wells in angled boreholes. With only the bottom of the inner and outer core barrel exposed to the aquifer at any given time, determining the location of the water table can be difficult.

While this drilling method has the capability of drilling through and providing samples of coarse gravels, boulders, and tight clays, these situations will result in slow drilling or advancement of the core barrel. The result is a hotter core barrel and a longer contact time between the core barrel and the encased soil core. The aforementioned conditions will increase the probability that the sonic method will raise the temperature of the soil core and facilitate VOC and SVOC loss.

The ability to quickly install deep borings and wells, while generating a large-diameter continuous soil core, makes this drilling technique invaluable when continuous soil sampling is needed to assess deep or complex geological situations. However, sonic drilling's high cost, relative to other drilling methods, may be prohibitive for small projects or shallow boreholes. The higher cost of the drilling method should be weighed against the cost savings incurred due to its faster drilling rate and high quality of the soil core produced.

5. GeoProbe®-Direct Push

The method involves hydraulically pushing hollow rods into the subsurface for the purpose of collecting soil and/or groundwater samples (e.g., Geoprobe®). The method can be used to collect discrete soil samples or install small-diameter wells used to collect groundwater samples.

Advantages of the direct-push method include the relatively quick collection of groundwater samples and, when used along with a mobile laboratory, collection of data in “real” time. The method allows for collection of multiple samples in a day with the potential for achieving contaminant delineation in one mobilization of the field equipment. The data can also be used to select locations of permanent monitor wells.

Disadvantages of the method include the fact that the data quality achieved is often suitable only for screening purposes. Direct-push methods typically result in very turbid samples since an oversize borehole is not produced and a filter pack is not used. Turbid samples can produce higher metal concentrations in groundwater samples since metals are typically adsorbed onto soil particles. Use of direct-push methods can also cause cross-contamination since contamination from shallow zones may be driven down to deeper zones. Due to the narrow diameter of the direct-push rods, samples are often collected with peristaltic pumps. When samples are collected for volatile organic compounds (VOCs) using peristaltic pumps, some

of the volatiles may be lost due to the pressure drop produced by the suction lift. In such cases, the VOC data must be qualified accordingly. For this reason, use of the peristaltic pump for collecting groundwater samples for VOC analysis is not recommended and approval for its use should first be obtained from the project manager or geologist.

Another disadvantage of using direct-push technology for collecting groundwater samples is the potential to breech confining units. To prevent this, soil sampling using direct-push technology or conventional split-spoon sampling techniques should first be performed to identify the presence, depth and lateral extent of confining units. Pushing through confining units should be avoided if the presence of dense, non-aqueous-phase liquid (DNAPL) or very soluble compounds such as Methyl Tertiary Butyl Ether (MTBE) are suspected or the contaminant plume appears to be diving in the aquifer.

6. Contact

Gary Fuerstenberg

STANDARD OPERATING PROCEDURE

DM-002 Hollow-Stem Auger

1. Objective

Describe standard operating procedures for drilling of overburden soil borings using hollow-stem augers.

2. Execution

- Confirm that the appropriate measures have been taken for clearance of potential subsurface utilities. The responsibility for clearance may vary, depending on the client.
- Inspect the drilling rig to make sure it is clean and that the down-hole equipment has been steam-cleaned or pressure-washed. Record observations in the field notebook (See SOP FD-001).
- Observe that the augers are vertical when the first section is advanced into the ground.
- Use a 140-lb hammer to drive the sampler, unless conditions necessitate using a 300-lb hammer (see SOPs SM-001, *Split-Spoon Sampling* and SM-0003, *Soil Classification,* for details). Count and record the number of blows per 6-inch increments, confirming blow counts with driller if necessary).
- Decontaminate the split-spoon sampler after each use (see *Equipment Decontamination,* SOP QA-001) or use another decontaminated split-spoon sampler.
- Ensure that the drillers advance the augers only after they have inserted the auger plug (to prevent soil from entering the augers while advancing to the next sample interval).
- Request that the drillers remove the auger cutting bit/plug and insert the split-spoon sampler into the interior of the augers. Measure the stick-up of the rods attached to the sampler to ensure that the nose of the spoon is in virgin soil below the augers.
- Watch for signs of a soil strata change at depth during drilling (i.e., change in blow counts, change in soil color, soil wetness, soil contamination, bouncing of the drill rig, etc.). If important to the investigation, stop drilling and collect a soil sample.
- If subsurface soil samples are being collected with split-spoon samplers, ensure that the drillers use a 30-inch drop of the 140-pound hammer. The number of blow-counts for each 0.5 foot penetration provides important geotechnical data.
- Repeat until the borehole has been drilled to the desired depth.
- If a monitoring well is not installed in the soil boring, fill the boring with either cement/bentonite grout or properly-tamped and hydrated bentonite. Check with Project Manager and/or the appropriate regulatory personnel before using drill cuttings to backfill the boring.

- If a monitoring well will be installed, refer to SOP DM-007.
- Complete boring log and, if necessary, well installation logs (SOP SM-003, *Soil Classification*).
- Record boring locations on a site map and in a field notebook sketch. If the boring location will not be surveyed, measure each location from on-site reference points and record the information in the field notebook so that the location can be plotted on site figures.

3. Limitations

- In areas of significant soil contamination, hollow-stem augers may cross-contaminate upper soil layers as contaminated cuttings move up the auger flights. The potential also exists for contaminated augers to carry contamination to deeper soil strata
- If significant unanticipated contamination is encountered during drilling, stop drilling to confer with the project manager and evaluate health and safety conditions. If the borehole is to be advanced below the contaminated strata, use telescoping techniques (see SOP DM-008 *Monitoring Well Telescoping Techniques*) to avoid cross-contaminating underlying geologic strata.
- When drilling below the groundwater table in fine to medium sands, the potential exists for the phenomenon of “running sands” or “blow in” to occur. Frequent measurements inside the hollow-stem augers after the drill bit/plug is removed will indicate if running sands are present. If sands start to flow into the auger, pour clean water into the augers and keep the augers filled during sampling.
- If necessary, arrange for the storage of contaminated soil cuttings and water in drums or other appropriate containers in a secure place at the site. Containers should be labeled.
- Plan the drilling program to drill borings from the least- to most-contaminated areas. Be prepared in advance and know where alternative drilling locations are in the event that problems are encountered at each planned soil boring location. Alternative locations will need to have utility clearance.
- Down-hole drilling equipment should be steam cleaned or pressure-washed between holes unless otherwise directed by the project manager.
- Record when standard operating procedures are deviated from. The drilling inspector should also record any detected odor from the boring and depth encountered.

4. References

Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers (October 1990), American Society for Testing and Materials [ASTM] D5092-90

Nielsen, D.M. (1993), “Correct Well Design Improves Monitoring,” Environmental Protection, July, pp. 38-49

Standard References for Monitoring Wells (April 1991), Commonwealth of Massachusetts Department of Environmental Protection, WSC-310-91

5. Attachments

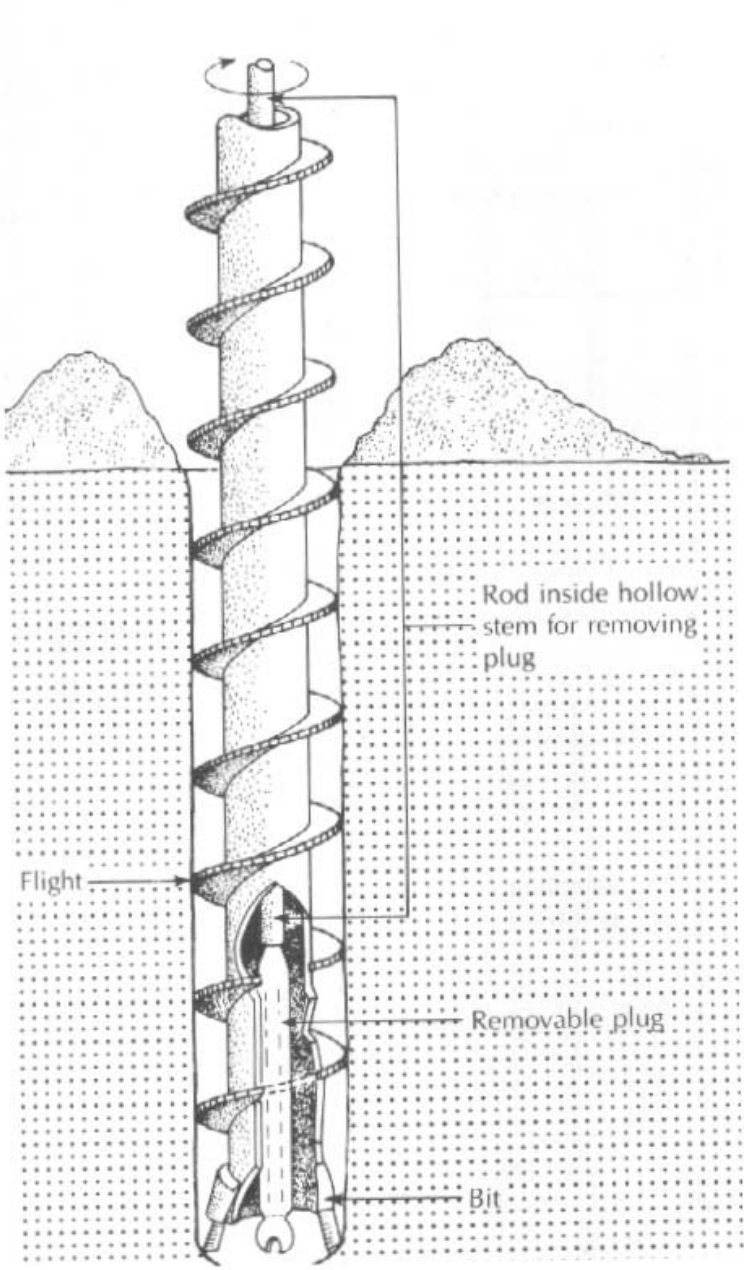
Attachment A – Hollow-Stem Auger

6. Contact

Gary Fuerstenberg
Cathy Johnson

SOP DM-002

Attachment A – Hollow Stem Auger



GEI Consultants

STANDARD OPERATING PROCEDURE

DM-003 Air Rotary Drilling with Casing

1. Objective

Describe standard operating procedures (SOP) for drilling of soil borings using casing by either the rotary method or air method.

2. Execution

- Confirm that appropriate measures have been taken for clearance of potential subsurface utilities. The responsibility for clearance may vary, depending on the client.
- Inspect the drilling rig to make sure it is clean and that the down-hole equipment has been steam-cleaned or pressure-washed. Record condition of down-hole drilling equipment. Record all observations in the field notebook (See SOP FD-001).
- Confirm that the casing (i.e., the borehole) is vertical when the first section of casing is advanced into the ground.
- For all soil samples, use a 140-lb hammer to drive the sampler, unless conditions necessitate using a 300-lb hammer (see SOP SM-001 *Split-Spoon Sampling* and SOP SM-003 *Soil Classification*–for details). Count and record the number of blows per 6-inch increments, confirming blow counts with driller if necessary.
- Decontaminate the split-spoon sampler after each use (see SOP QA-001 *Equipment Decontamination).*
- Advance the casing with the drive hammer and periodically clean out cuttings using a pneumatic hammer. A water spray and shield may be used for dust control and to control rock chips.
- Potable water should be used as the drilling fluid. If subsurface soil is sufficiently permeable to require a thicker fluid, drilling mud may be used to increase fluid viscosity, with prior approval from the project manager.
- To continue advancing the boring, additional lengths of casing are added one at a time, repeating this sequence until the required depth is reached.
- If an obstruction is encountered, the driller should attempt to penetrate the obstruction with a pneumatic hammer.
- If a monitoring well is not installed in the soil boring, fill the boring with either cement/bentonite grout or properly-tamped and hydrated bentonite. Check with Project Manager before using drill cuttings to backfill the boring.
- If a monitoring well will be installed, follow SOP DM-007.

- If the boring location is not going to be surveyed, measure each location from on-site reference points in the field notebook so that it can be relocated and plotted on figures.

3. Limitations

- To advance the boring, it may be necessary to use casing to maintain air pressure. Additional lengths of casing are then added one at a time, repeating this sequence until the required depth is reached.
- If unanticipated contamination is encountered, stop drilling to confer with the project manager and evaluate health and safety conditions.
- If the borehole is to be advanced below the contaminated strata, use telescoping techniques (see DM-008 Monitoring Well Telescoping *Techniques)* to avoid cross-contaminating underlying geologic strata.
- When drilling below the groundwater table in fine to medium sands, the potential exists for the phenomenon of "running sands" to occur. A head should be kept on the borehole at all times.
- Arrange for the storage of contaminated soil cuttings and water in drums or other appropriate containers in a secure place at the site (see SOP SC-003, *Investigation Derived Waste Management*).
- Plan the drilling program to drill borings from the least to most contaminated areas. Be prepared in advance and know where alternative drilling locations are in the event that problems are encountered at each planned soil boring location. These locations must also have been cleared by the state utility service prior to drilling.
- Document variations from standard operating procedures.

4. References

ASTM D 5782 – Guide for Use of Direct Air Rotary Drilling for Geoenvironmental Exploration and Installation of Subsurface Water-Quality Monitoring Devices

5. Contact

Gary Fuerstenberg
Cathy Johnson

STANDARD OPERATING PROCEDURE

DM-004 Sonic Drilling

1. Objective

Describe common sonic drilling procedures.

Prior to drilling confirm that utility clearance has been completed and that the drilling rig has been appropriately decontaminated.

2. Execution

- Collect soil cores in runs of 5 to 10 feet. Some sonic rigs can collect a 20 foot sample, but the process generates a significant amount of heat that may degrade sample quality.
- Classify and sample the soil located within the liner.
- Excess soil should be placed in a 55-gallon drum for disposal.
- The core barrel should be cleaned with tap water following each use.
- The core barrel is then advanced within the isolation casing to collect the next soil core interval.
- Add water between the inner core barrel and the outer override casing. This will reduce friction between the casings and adsorb heat.
- Maximize drilling advance rate. The faster the core barrel is advanced, the less likely the core barrel will heat up. Drilling with a 3-inch diameter core barrel and a 5-inch diameter override casing, instead of the standard 4-inch core barrel and 6-inch over-ride casing, may increase advance rates and reduce the potential for soil core heating.
- If a significant decrease in drilling advance rate is observed, stop drilling and remove soil that has accumulated in the core barrel. Resume drilling through the resistant material (gravel, boulder, hard clay, etc.). When the resistant material has been penetrated and the drilling advance rate increases, stop drilling and remove what material has accumulated in the core barrel.
- Wash down the core barrel with cool water to cool the core barrel and associated casing, and resume drilling.
- If a well is to be installed in the borehole, the sandpack and grout are placed as the core-barrel and over-ride casing(s) are selectively vibrated out of the ground. The vibratory action should facilitate settlement of the sandpack and grout. Upon completion, no casing is left in the ground other than the well casing and screen.

3. Limitations

- Disturbance of the soil core is most likely to occur during removal of the soil core from the core barrel. The soil cores are usually vibrated out of the core barrel into plastic bags approximately 5 feet in length. As the plastic bags are

a little larger than the soil core itself, fragmentation of the soil core may occur as the core is extruded into the bag or while the bagged core is being moved in an unsupported manner. Soil conditions that are prone to disturbance include wet or dry zones that contain little or no fines, and well graded sands that contain significant volumes of water.

- If integrity of the soil core is of concern, the following procedures should be implemented:
  - Measures should be taken to ensure that the core, from the time it is extruded from the core barrel, is rigidly supported through the use of some type of cradle or carrying device.
  - The core should not be removed from its cradle until all sampling of the core has been completed. Acrylic liners are available for some core sizes and can be used to hold the core together upon removal from the core barrel.
  - If the soil is to be sampled for volatile organic compounds (VOCs), acrylic liners must be used.
  - Sampling of the soil core for VOCs or semi-volatile organic compounds (SVOCs) must be approved on a case by case basis. Proposals for VOC or SVOC soil core sampling must include provisions to minimize core fragmentation and heat generation, such as:
    - Acetate liners in the core barrel so that the soil core does not have to be extruded out of the core barrel.
    - Limit the length of soil core generated during a given downhole run.
    - Implement practices to reduce the residence time of the soil core in the core barrel.
- For the analysis of SVOCs, the use of the acetate liners is not required.
- The large diameter of the core barrel enables ground water sampling equipment to be placed inside the core barrel so that discrete depth groundwater samples can be collected during borehole advancement.

4. References

Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers (October 1990), American Society for Testing and Materials [ASTM] D5092-90

5. Contact

Melissa Felter

STANDARD OPERATING PROCEDURE

DM-006 Geoprobe® Direct Push Boring

1. Objective

Describe standard operating procedures (SOP) for drilling of overburden soil borings using Geoprobe® and MacroCore® technologies.

2. Execution

- Confirm that appropriate measures have been taken for clearance of potential subsurface utilities. The responsibility for clearance may vary, depending on the client.
- Inspect the drilling rig to make sure it is clean and that the down-hole equipment has been decontaminated (QA-001). Record condition of all down-hole drilling equipment.
- Make sure the sampler is fitted with a piston rod assembly to block the sample tube until the desired subsurface sample interval is attained. Upon reaching the target sample depth, the piston tip will be released and the discrete sampler device is then advanced to collect the representative sample. This reduces the volume of slough that is collected.
- When the sampler is brought to the ground surface, it should be opened immediately, and the length of recovery should be measured and recorded.
- Log the soil sample using USCS procedures (SOP SM-003). Collect analytical samples if necessary (SOP SM-001).
- Decontaminate the cutting shoe if necessary (SOP QA-001 Equipment Decontamination) and have driller reassemble the parts with a new liner.
- Repeat the procedure described above until refusal or the boring is terminated.
- Periodically verify that depths cited by drillers are accurate.

3. Limitations

- If significant unanticipated contamination is encountered during drilling, stop drilling to confer with the project manager and re-evaluate health and safety conditions.
- Arrange for the storage of contaminated soil cuttings and water in drums or other appropriate containers in a secure place at the site (see SOP SC-003, *Investigation Derived Waste Management*).
- If possible, plan the drilling program to drill borings from the least to most contaminated areas. Be prepared in advance and know where alternative drilling locations are in the event that problems are encountered at soil boring locations. These locations must also have been cleared by the state or local utility service prior to drilling.

4. References

ASTM D6001-05 Guide for Direct Push Water Sampling for Geoenvironmental Investigations, April 2005

Geoprobe Systems, "Geoprobe MacroCore MC-5 1.25-inch Light Weight Center Rod Soil Sample System SOP", Technical Bulletin No. MK 3139, November 2006

5. Attachments

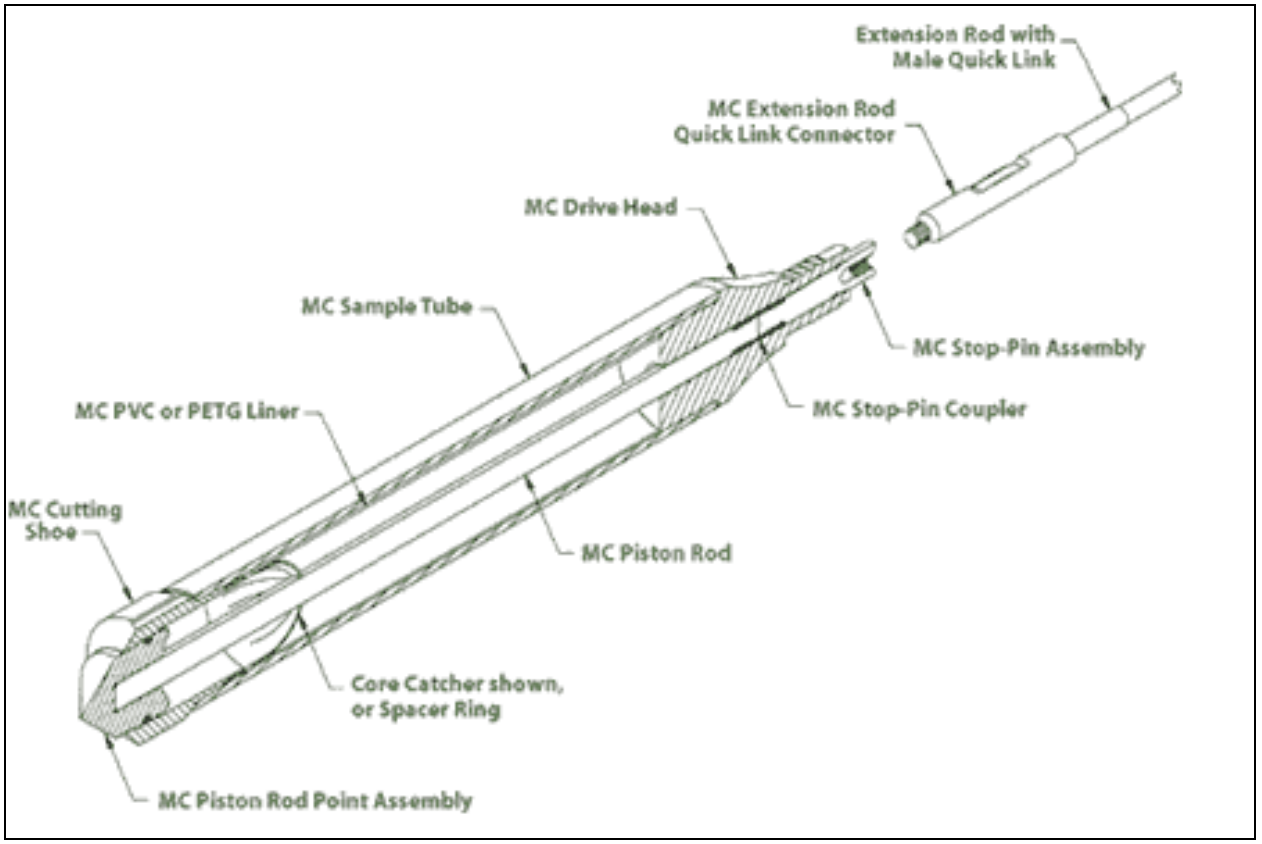
Attachment A – Geoprobe® with Macrocore® Sampler Assembly

6. Contact

Melissa Felter
Cathy Johnson

SOP DM-006

Attachment A – GeoProbe® with Macrocore® Sampler Assembly



Above: Diagram of a Macrocore® sampler

Right: A track-mounted GeoProbe ® Rig

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STANDARD OPERATING PROCEDURE

DM-007 Monitoring Well Construction and Installation

1. Objective

Describe installation procedures for overburden monitoring wells screened across or below the groundwater table.

Well dimensions (well diameter, screen length, and screen slot-diameters) will be specified in the Work Plan. This SOP assumes the monitoring wells will be constructed of flush-joint PVC pipe and the screened section will have factory-slotted openings.

2. Execution

Attachment A provides a diagram of typical shallow, intermediate, and deep groundwater monitoring well construction detail. A Groundwater Monitoring Well Installation Log is in Attachment B.

- Measure and record the depth of the completed soil boring before beginning the well installation.
- If possible, measure the depth to groundwater in the borehole over a 10 to 15 minute period to ensure that the groundwater elevation has approximately stabilized. Compare the saturated soil depth estimated from split-spoon samples to the measured water level in the borehole. If drilling water has been used during boring advancement, pump the water out of the borehole to the static water depth, based on examination of the soil samples, and monitor the recovery of groundwater until the level has stabilized.
- If it is not possible to accurately measure the depth to groundwater in the borehole due to low permeability in the formation, use the saturated soil depth observed in the collected samples or measured water depth in a nearby existing monitoring well to estimate the depth to water in the borehole.
- For shallow monitoring wells, select the monitoring well screen and riser lengths so that the slotted section of the screen intersects the groundwater table. Screen lengths of 15 feet or less are preferred and 10 foot screens are most common. If the water table is seasonally high or low or if the well is in a location where the water table is likely to be tidally influenced, appropriately place the screened section to allow for the screen to intersect likely future water tables.
- For intermediate or deep wells screened entirely below the water table, select the monitoring well screen and riser lengths as described in the Work Plan. Screen lengths of 10 feet or less are preferred.
- If the borehole is deeper than the desired well depth or the bottom of the well is close to a change in soil strata, then fill the base of the borehole with bentonite. Keep in mind that bentonite swells when hydrated, and that filter

sand should be placed at the bottom of the borehole above the bentonite before installing the well.

- Prevent well materials from contacting foreign substances during installation. Precautions may include requiring the driller to wear clean gloves while handling well materials and requiring that well materials not be placed onto the ground or pavement without a protective barrier such as polyethylene sheeting being present
- Confirm that the driller installs a minimum one-inch sump with a bottom cap to the bottom of the well screen. See the Work Plan for locations that may require larger sumps.
- Monitoring wells can be constructed of either 1, 1.5, 2 or 4 inch inner diameter (ID) Schedule 40 threaded flush-jointed PVC. Refer to the work plan for the site-specific requirements. Flush-threaded well materials should be used. Do not allow the driller to use glues, as they typically contain solvents that could affect on groundwater quality.
- Stainless steel well materials may be used if required in the Work Plan. Select slot size based on grain size of the formation and on requirements in the Work Plan.
- Confirm that the driller places at least 12 inches of clean uniformly graded medium quartz filter sand pack into the base of the borehole, if required in the Work Plan.
- The driller should remove the drilling casing/augers from the borehole slowly, at a maximum of 2-foot intervals, at the same time that filter sand is added. The drillers should take frequent measurements of the depth to sand.
- Confirm that the driller has added adequate sand to surround the area around the slotted section. The filter sand should extend at least 2 feet above the top of the slotted section.
- The driller should place a bentonite seal above the filter pack. If the seal is above the water table, use at least 5-gallons of potable water to hydrate the bentonite before grouting the remaining annular space, or otherwise backfilling the remaining annular space as discussed with the Project Manager. Tamp seal. It should extend 1 to 2 feet above the filter sand.
- If required by the Work Plan, the driller should use bentonite-cement and grout the annular space from the top of the bentonite seal to the ground surface. Bentonite cement grout should be placed using tremie methods. Grout should be mixed in approximately the following proportions: 7.5 gallons water to one 94-lb bag of cement to 2-4 lbs of pulverized bentonite. The grout must be mixed using a pump (such as one on the rig) to ensure proper mixing.
- The drillers should cut the monitoring well riser at an angle or make "V"-notch in the riser pipe as a benchmark for surveying and groundwater measurements. The driller should cut the well riser so that the top of the well will be approximately 3 inches below the top of protective casing. The top of

the riser should be close enough to the top of the surface casing to allow reading of depth markings on a water level indicator tape.

- The protective surface casing is either a flush-mounted roadbox or a steel "stick up" pipe. The base of either type of casing should extend at least 1 foot into the grout below the ground surface (below the frost line) whenever possible.
- The protective casing should be set by placing cement in the annular space between the protective casing and the borehole up to the ground surface. If possible, the driller should slope the cement radially away from the protective casing at the ground surface to promote surface water runoff.
- In areas of high traffic or areas of parking lots and/or roadways where plowing occurs, set the roadbox flush with the ground surface to avoid damage to the well.
- If the well is installed in a high-traffic area and is completed with a steel "stick up" pipe, additional protection such as steel pole bumpers around the steel "stick up" pipe may be necessary.
- If possible a locking cap should be placed on the steel "stick up" pipe. If the surface casing is flush mounted, a locking expansion plug should be placed, if possible, inside the top of the well riser pipe.
- All well locations should be photodocumented in accordance with SOP FD-004 Photodocumentation.
- Label the outside of the protective well casing with a paint pen. If the well is not going to be surveyed, measure the location to nearby landmarks so that the well may be located in the future and plotted on figures. Make sure to enter this information in the field notebook). If possible, place a brightly colored stake or other identifier adjacent to the well.
- Develop the well (see SOP DM-009, *Monitoring Well Development*).

3. Limitations

- Do not screen across different hydrostratigraphic units (for example, outwash sands, confining layers or till) unless specified in the Work Plan or approved by the Project Manager.

- If the formation is composed of a material that is uniformly coarser than the filter sand, the grain size of the filter sand should be increased. Consideration should also be given to changing the slot size on the well screen. Differences in average grain size should generally not be greater than a factor of two to four times.

- Do not use drill cuttings to backfill during monitoring well installation unless specified by the work plan or project manager.

4. References

Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers (October 1990), American Society for Testing and Materials [ASTM] D5092-90

Nielsen, D.M. (1993), "Correct Well Design Improves Monitoring," Environmental Protection, July, pp. 38-49

Standard References for Monitoring Wells (April 1991), Commonwealth of Massachusetts Department of Environmental Protection, WSC-310-91.

5. Attachments

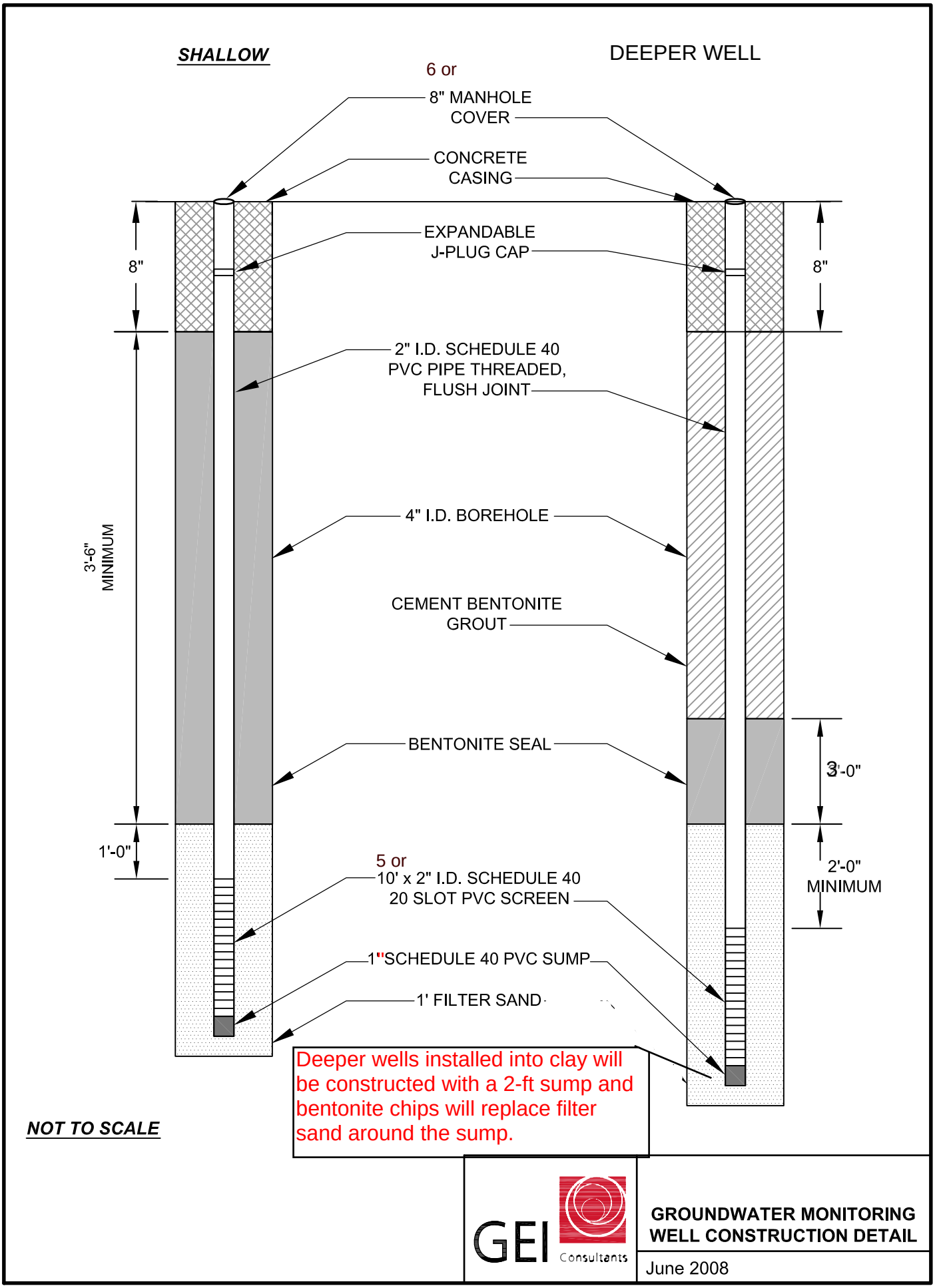
Attachment A – Typical Shallow, Intermediate, and Deep Groundwater Monitoring Well Construction Detail

Attachment B – Groundwater Monitoring Well Installation Log

6. Contact

David Terry
Anne Leifer

Attachment A - Well Detail



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Groundwater Well Installation Log

Well ID

Project _____ **GEI Proj. No.** _____

City / Town _____ **Location** _____

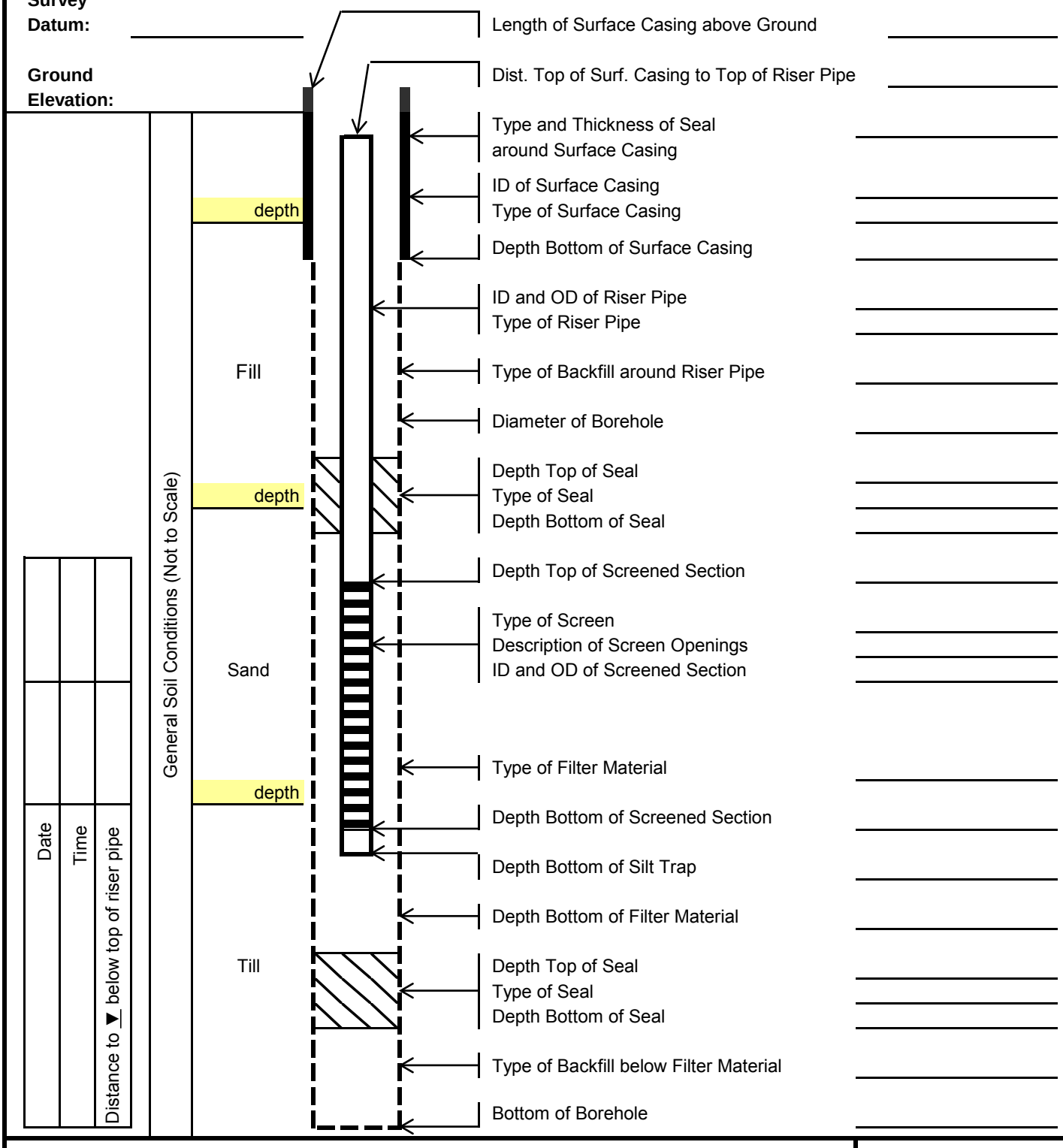
Client _____ N _____

Contractor _____ E _____

Driller _____ **GEI Rep.** _____ **Install Date** _____

Survey Datum: _____

Ground Elevation: _____



Notes:

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STANDARD OPERATING PROCEDURE

DM-008 Telescoped Drilling

1. Objective

Describe telescoped drilling used to prevent the connection of two stratigraphic layers during boring advancement and/or monitoring well installation. This type of drilling is commonly used to prevent higher density contaminants (such as in coal tar or chlorinated solvents) from migrating downward in a boring.

Telescoped drilling is used with air rotary drilling (SOP DM-003) and drive-and-wash drilling (SOP DM-005). Refer to these SOPs for procedures to be performed prior to installing casing.

2. Execution

- Confirm that drilling tools have been appropriately decontaminated.
- Drill or core at least four feet into the stratigraphic unit (bedrock, till, etc.) to confirm the unit and provide a "socket" into which permanent casing can be grouted.
- The socket diameter should be at least two inches larger that the casing to be installed.
- The casing that will be installed should be large enough to allow smaller diameter drilling tools inside it to advance deeper, without the possibility of cross-contamination or hydraulic connection between the shallower and deeper units. For example, a six-inch casing is large enough for use of 4-inch tools inside it.
- Freshly mixed grout is required to set the large-diameter casing into the unit to be isolated. Depending on application, a mixture of Portland cement and bentonite meets most grouting requirements. For proper consistency, use no more than 6 gallons of water per 94-pound sack of cement. Add a few pounds of bentonite or hydrated lime per sack of cement for better flow.
- The driller should use a tremie pipe to deliver grouting outside the casing. Follow these steps to complete grouting using this method:
    i. Lower the casing to the bottom of the borehole. Make sure that the lower end of the casing is tightly seated at the bottom.
    ii. Mix a sufficient quantity of grout and pump it through the tremie pipe. As the grout is placed, lift the tremie pipe slowly, but keep the lower end submerged in the grout.
    iii. Fill the casing with water as the grout is placed to balance the fluid pressure inside and outside the casing. Doing so prevents grout from leaking under the bottom of the casing.

iv. Allow the grout to set for a minimum of 24 hours. Some regulatory agencies require 48 hours.
v. Using smaller diameter drilling tools, advance through the existing casing into the underlying unit to continue investigation. Install additional casing or PVC into the unit, or leave it as an open borehole.

3. Limitations

- If dense non-aqueous phase liquid (DNAPL) and/or dissolved contamination is suspected in the shallow (weathered) bedrock, then installation of casing in the rock may not be wise. It could hide the DNAPL from detection and/or result in cross-contamination of deeper rock. A shallow bedrock well may be called for to characterize the impacts. Consult with the Project Manager.
- Other casing installation methods, such as pressure-grouting may be more appropriate. Consult with the Project Manager.

4. References

Environmental Protection Agency, Region 4, "Environmental Investigation Standard Operating Procedures and Quality Assurance Manual, Chapter 6 – Design and Installation of Monitoring Wells," November 2001.

5. Contact

Gary Fuerstenburg
Catherine Johnson

STANDARD OPERATING PROCEDURE

DM-009 Monitoring Well Development

1. Objective

Describe standard procedures to remove fluids from monitoring wells (introduced during drilling) and maximize the movement of groundwater into the well by removing fine particles in the well and sand pack around the screen.

2. Execution

To prevent cross contamination between monitoring wells, use dedicated equipment and/or appropriately decontaminated equipment to perform monitoring well development. See SOP QA-001 Equipment Decontamination and the Work Plan for more information.

For deep or large diameter monitoring wells, it may be necessary to use a re-usable pump system, such as a Grundfos pump, to develop monitoring wells.

- Calculate the volume of water in the monitoring well (one well volume) using the following table:

Well diameter (inches)	Volume (gal/ft)
1	0.04
1.5	0.09
2	0.16
3	0.36
4	0.65
6	1.50

  The equation used to establish these volumes is presented in Section 4.

- Calculate or estimate the amount of water introduced to the borehole during drilling. At a minimum, this is the amount of water that should be removed during development. Removing less water than was introduced and allowing additional time for the surrounding formation to clear of injected drilling fluids may be considered as an alternative if the volume of introduced water was large.

- Record the volume of water purged in the field notebook or on the Monitoring Well Sampling Form (Attachment A).

- Collect a sample of water from the monitoring well with the selected submersible pump (e.g. 12-volt whale pump or Grundfos pump), a bailer, or a

Waterra system. Record the physical properties (color, turbidity, odors, etc.) of the sample.

- The volume of water that should be removed will depend on the work plan, local regulatory guidance, and/or the volume of water that was introduced during drilling and well installation. Typical guidance for the removal volume includes:

    - Ten well volumes.
    - The volume of fluid added during drilling.
    - The volume required to remove enough suspended particles so that the turbidity of the water is less than 50 nephelometric turbidity units.

If needed, pump the ground water into a 5-gallon pail so that the volumetric flow rate and total water volume from the pump or bailer can be calculated.

Measure the groundwater level in the well during development to assess if the pumping rate is sufficient to create a drawdown in the well.

Observe the groundwater every few well volumes during the pumping and record the physical properties (color and turbidity).

If required by the Work Plan, conduct surging in the monitoring well. See the Work Plan for the method of well surging to be used. If surging is necessary, do so only after initial pumping at the well has occurred and fine sediments have been removed.

Slowly move the surge block up and down in the well. Periodically remove the surge block and purge the groundwater until it is relatively clear again. Start at a slow pace and progress to a faster surging action through time.

3. Limitations

Always remove groundwater with fine particles from the well before surging. The fine particles may be forced into the well screen by the surging action. They may also damage the pump.

If the ground water in the monitoring well is contaminated, the water removed during well development may need to be placed in a properly-labeled drum and disposed of in accordance with local, state, and federal regulations (see SC-003 Investigation Derived Waste).

If the soils around the well screen are composed of fine-grained silts and clays, over-pumping and mechanical surging is not recommended since these more vigorous

techniques can cause mixing of the fines into the filter pack. To develop these wells, use of a bailer is recommended.

There are occasions when the turbidity of groundwater cannot be meaningfully reduced. On these occasions, a minimum of ten volumes should be removed, and the Project manager should be consulted.

Sampling of groundwater should generally not occur within one week after development. In some regions or regulatory jurisdictions, a minimum of two weeks may be required before sampling. If no water was introduced to the formation during drilling, this waiting period may be shortened if required by the project. See the Work Plan for additional information.

4. References

Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers (October 1990), American Society for Testing and Materials [ASTM] D5092-90.

Nielsen, D.M. (1993), "Correct Well Design Improves Monitoring," Environmental Protection, July, pp. 38-49.

"The Methods & Mechanics of Well Development, Part 2 of 5," National Drillers Buyers Guide, March 1993, p. 17.

Massachusetts Department of Environmental Protection, "WSC-310-91Standard References for Monitoring Wells, Section 4.5 Decommissioning of Monitoring Wells", January 1991
.

U. S. EPA Environmental Response Team Standard Operating Procedure SOP: 2044 ," Monitor Well Development" REV: 0.1, 10/23/01

5. Attachments

Attachment A - Monitoring Well Sampling Form

6. Contact

Gary Fuerstenberg
Anne Leifer

Monitoring Well Development / Sampling Log

GEI Consultants

Site Name:	Well ID:

Date_____

Field Personnel_____

Method of Purging/ Sampling_____

Well Depth (ft btoc)_____

Depth to Water (ft btoc)_____

Casing type/dia._____

Well Volume (g)_____

Time	Total Volume (g)	Temp (°F)	pH	Cond (us/cm)	ORP (mV)	D.O. (mg/L)	Turbidity (NTU)	Comments

STANDARD OPERATING PROCEDURE

FD-001 Field Notebook

1. Objective

Describe methods for documentation of field activities.

Documentation of site activities is a crucial part of the field investigation process. The field notebook serves as the record of field activities performed or observed during the project. It provides a factual basis for preparing field observation reports, if required, and reports to clients and regulatory agencies. Example field notes are provided in Attachment A.

2. Execution

- Use a separate all-weather bound notebook for each site/location/project number. Spiral notebooks should not be used because pages can be easily removed.
- Write neatly using black or blue pen, preferably a waterproof pen. Use of pencil is also acceptable only with approval of the project manager, such as in but not limited to, certain field conditions [e.g., cold or wet weather].
- Write the project name, project number, book number (i.e., 1 of 3), and date on the front cover. On the inside cover, identify the project name, project number, and "Return Book To:" the office address of the project manager.
- Number all of the pages of the field book starting with the first entry.
- Record activities as they occur. Record only facts and observations, regardless of whether they appear to be relevant at that time.
- Identify conditions or events that could affect/impede your ability to observe conditions (e.g. snow-covered ground surface, inability to access areas of interest).
- Neatly cross out mistakes using a single line and initial them. Erasures are not permitted.
    - If an error is made on an entry in the field notebook, the individual who made the entry should make the corrections. The corrections must be initialed and dated by the person making the correction.
- Sign or initial and date the bottom of every page with an entry if the project requires such documentation.
- Place a diagonal line through unused portions of a page.
- Record the following information upon each arrival at the site:
    - Date/time/weather.
    - GEI personnel.
    - Purpose of visit/daily objectives.
    - People (client, contractor, landowners, etc.) present upon GEI arrival.

- Record the following information during the course of the day:
  - Conversations with contractors/subcontractors, clients, visitors, GEI staff, landowners (site or abutters). If possible, record complete names, titles, and affiliations.
  - Time of arrival and departure of individuals.
  - Activities as they occur.

- Additional examples of observations to record may include and are not limited to:
  - Type and quantity of monitoring well construction materials used.
  - Use of field data sheets or electronic logging equipment (e.g. boring logs, monitoring well sampling logs, etc.).
  - Ambient air monitoring data.
  - Field equipment calibration information.
  - Locations and descriptions of sampling points.
  - Contractor/Subcontractor progress.
  - Sample media (soil, sediment, groundwater, etc.).
  - Sample collection method.
  - Number and volume of sample(s) collected and sample bottle preservatives used.
  - Sample identification number (s) and date and time of sample collection.
  - Approximate volume of groundwater removed before sampling.
  - Any field observations made such as pH, temperature, turbidity, conductivity, water level, etc.
  - References for maps and photographs of the sampling site(s).
  - Information pertaining to sample documentation: bottle lot numbers/ dates, method of sample shipments, chain-of custody record numbers, and overnight shipping numbers.
  - Surveying data (including sketches with north arrows).
  - Changes in weather.
  - Rationale for critical field decisions.
  - Recommendations made to the client representative and GEI Project Manager.
  - Site sketch of conditions at the end of the day.
  - Summary of work completed/work remaining.
  - Allow time at the end of the day to complete entries in the notebook.

3. References

New Jersey DEP Field Sampling Procedures Manual, August 2005.

ASFE Daily Field Report for Geotechnical Field Observation, 2nd Edition (2001), ASFE, Inc.

4. Attachments

Attachment A - Example Field Notes

5. Contact

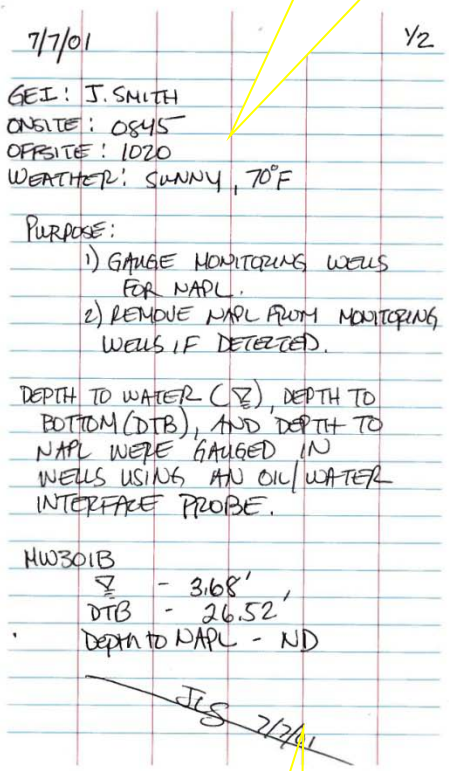
Melissa Felter
Leslie Lombardo

SOP FD-001

Attachment A – Example Field Notes

Start of each day includes:
- **Date**
- **Project Number**
- **People on site**
- **Purpose of Work**
- **Weather Conditions**

Each page is numbered



7/7/01 1/2

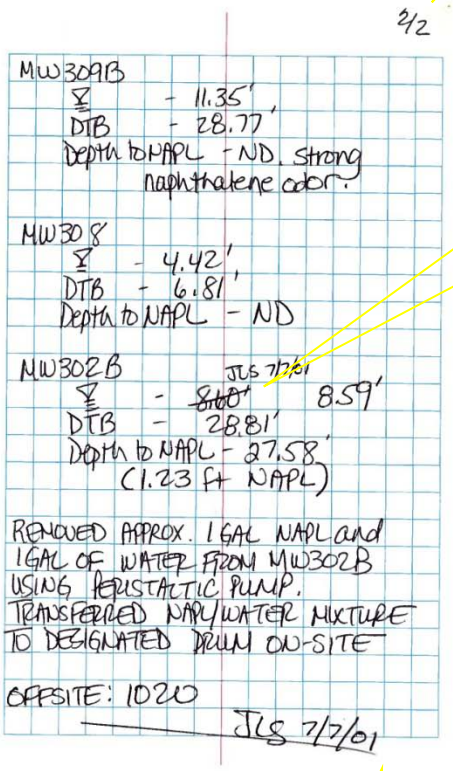
GEI: J. SMITH
ONSITE: 0845
OFFSITE: 1020
WEATHER: SUNNY, 70°F

PURPOSE:
1) GAUGE MONITORING WELLS FOR NAPL.
2) REMOVE NAPL FROM MONITORING WELLS IF DETECTED.

DEPTH TO WATER (▽), DEPTH TO BOTTOM (DTB), AND DEPTH TO NAPL WERE GAUGED IN WELLS USING AN OIL/WATER INTERFACE PROBE.

MW301B
▽ - 3.68'
DTB - 26.52'
Depth to NAPL - ND

JLS 7/7/01



2/2

MW309B
▽ - 11.35'
DTB - 28.77'
Depth to NAPL - ND. Strong naphthalene odor.

MW308
▽ - 4.42'
DTB - 6.81'
Depth to NAPL - ND

MW302B JLS 7/7/01
▽ - ~~8.60'~~ 8.59'
DTB - 28.81'
Depth to NAPL - 27.58'
(1.23 ft NAPL)

REMOVED APPROX. 1 GAL NAPL and 1 GAL OF WATER FROM MW302B USING PERISTALTIC PUMP. TRANSFERRED NAPL/WATER MIXTURE TO DESIGNATED DRUM ON-SITE

OFFSITE: 1020

JLS 7/7/01

Errors are single line crossed out and initialed

Blank Space crossed out and initialed

Bottom of each page signed and dated

GEI Consultants

STANDARD OPERATING PROCEDURE

FD-002 Field Observation Report

1. Objective

Describe methods to generate a Field Observation Report.

The Field Observation Report is used to record a summary of activities, observations, and decisions made during the day's field work. The daily field observation report serves as a permanent record of the day's activity for the Project Manager (PM), In-House Consultant (IHC), and/or client.

2. Execution

- If required, at the close of the day's field work, a Field Observation Report should be prepared by the individual responsible for the field notebook. This report should be completed before leaving work for the day. Contents of the report should include, at a minimum, the following information:
    - A record of person(s) present at the site, time of arrival, departure times (e.g., GEI, contractor(s), client, etc.).
    - A record of the daily objective(s) and the activities performed (e.g., drilled five borings in the overburden).
    - A summary of deviation(s) from the field plan or objectives.
    - A summary of field decisions made, who made them, and the basis for such decisions.
    - A diagram, sketch, and/or map showing the location and extent of the work or other significant observation(s) made during the day.
    - Recommendations that may result from field observations and actions that may result from implementation of those recommendations.
    - A summary listing and field sketch showing location(s) of field activity.
- Submit a draft report to the PM/IHC for review. Complete any editorial changes, sign, date, and submit the report to PM/IHC for approval/signature. Field Observation Reports should be written neatly. They are not required to be typed unless specifically requested by the PM.

3. Limitations

- The Field Observation Report is not a substitute for the field notebook.
- Not all projects require daily Field Observation Reports.
- The Field Observation Report should be based primarily on factual information. Opinions, if necessary, should be identified as such. Any speculation should be clearly noted in the report as such.

- The Field Observation Report should never be released to anyone other than the PM/IHC prior to review and sign-off unless explicitly authorized by the PM/IHC.

4. References

New Jersey DEP Field Sampling Procedures Manual, August 2005

ASFE Daily Field Report for Geotechnical Field Investigations, 2nd Edition (2001), ASFE, Inc.

5. Attachments

Attachment A - Example Field Observation Report

6. Contact

Melissa Felter
Leslie Lombardo

FIELD OBSERVATION REPORT

Project :	Guard Booth Upgrades	**Date:**	November 8, 2006
Client :	ACME Industries	**Report No.**	1
Contractor:	ABC Contracting	**Page:**	1 of 2
Subcontractor:	NA	**GEI Proj. No.**	99999-0

SOP FD-002 - Attachment A – Example Field Observation Report

Time of Arrival: 0700 **Departure: 1440** **Weather: Overcast, Raining, 55^{0}F**

Persons Contacted, Company
Jane Doe, ABC Contracting

GEI Representatives
Bill Smith

Purpose of Site Visit: To observe excavation of soils for new guard booth and sidewalk.

Observations:

1. **Excavation**
   a. Areas for guard booth and sidewalk were laid out by ABC with stakes, string, and spray paint. Locations were between the pavement and wetland area; no excavation occurred in the wetland area.
   b. Staging area for soil stockpile was located to the west of the excavation, along the fenceline; polyethylene sheeting was placed beneath the pile.
   c. HDPE membrane delivered to site; stored in garage area through the inside fence.
   d. ABC crew began hand digging area for sidewalk and guard booth. Sidewalk area measured 22 feet long by 4 feet wide by 4 inches deep. Guard booth area measured 12 feet long by 10 feet wide by 9 inches deep. Utility pole and bollard locations started today.
   e. Rain continued to get worse in the afternoon; ABC covered the entire excavation and soil stockpile with poly sheeting and secured the sheeting with grade stakes.

2. **Subgrade Preparation**
   a. Subgrade preparation for the sidewalk and guard booth areas at the site is complete.

3. **Dewatering**
   a. No dewatering occurred today.

4. **Air Monitoring**
   a. During excavation, I monitored the breathing zone of the workers with an organic vapor meter (OVM). No headspace readings were measured in soil samples S-1 through S-8.

FIELD OBSERVATION REPORT

Project :	Guard Booth Upgrades	**Date:**	November 8, 2006
Client :	ACME Industries	**Report No.**	1
Contractor:	ABC Contracting	**Page:**	2 of 2
Subcontractor:	NA	**GEI Proj. No.**	99999-0

Picture 1: Sidewalk excavation and bollard layout

By: Bill Smith	**Reviewed By:**

STANDARD OPERATING PROCEDURE

FD-003 Sample Management and Chain of Custody

1. Objective

Describe methods to label sample containers, manage the samples, and prepare Chain of Custody documentation for the samples. Sample transport is also addressed.

2. Project Setup

When setting up a sampling event, inform the recipients of the samples (laboratories) and recipients of laboratory results (data group and project managers). Discuss with the laboratory the sampling media, turnaround times, and reporting limits for appropriate regulatory criteria for the site. Include the data group on correspondence so that turnaround times, data validation, and project deliverable schedules can be tracked successfully.

- Laboratory - Number of samples, analyses needed: bottle orders and holding times, turnaround times needed, reporting limits needed for regulatory criteria.
- Data group - Number of samples, analyses requested, turnaround times and reporting limits requested, data validation needed, regulatory criteria to use for tabulating results, deliverables needed, and project name and number.
- Schedule - Inform the laboratory and Data Group of schedule delays, changes to analyses, and expediting.

3. Sampling Execution

- Review the work plan prior to sampling to determine the following:
  - Sample matrix and sampling method.
  - Required analysis and sample volumes.
  - Sample container type and preservative requirements.
  - Required analysis methods and/or report formats.
  - The turnaround time required by the project.
  - If the data will be sent directly from the laboratory to the data validator, Project Manager, or Data Group.
  - Holding time restrictions for sampling media and analytical methods.
  - Sample naming convention used for this project site.
- Sample labels should be filled out using a waterproof or permanent marker or pen. Required information includes:
  - Sample ID.
  - Date and time (military time) of sample collection.
  - Project number.
  - Sample preservatives.
  - Sampler's initials.
  - Laboratory analytical methods.

- Place the label on the jar or bottle, not on the cap. Sample custody begins at this time.
- Record the above information in the field notebook.
- Individually wrap sample jars with packing material, if needed. See SOP SC-002 for guidance on packaging samples for shipment to the laboratory by way of common carrier. Place samples in a cooler with bagged ice or freezer packs (blue ice) immediately after collection. Add sufficient ice or freezer packs to cool samples to approximately 4°C.
- Complete a chain of custody (COC) for the samples as described below. GEI or laboratory COCs may be used as long as they contain fields for all required sample information as described in Section 2.1.

3.1. Chain-of-Custody (COC) Completion

- Fill out COC neatly and in permanent ink. Alternatively, an Excel version of the GEI COC is available and can be filled out electronically.
- Certain analyses (i.e. air analysis by TO-15) require specialized, laboratory issued COCs. Make sure any specialized COCs are available before sample collection.
- Record the project name and number, the sampler's name(s) and the state where the samples were collected.
- For each sample, enter the sample identification number, date and time (military time) collected, the number of sample containers, and any additional information to fulfill project, client or regulatory requirements.
- Record the type of analysis (including laboratory method; e.g. EPA-SW846 Method XX) requested and the preservative (if appropriate) in the vertical boxes.
- Field duplicates should be anonymous to the laboratory, but must be recorded for use by the Data Group. To keep track of this information, link the field duplicate with the proper sample in the field notebook. If required by the Project Manager or Data Group, also document this information on or attach a note to the GEI copy of the COC.
- Trip blanks for large sites should be named similar to the samples they are collected with so that there are not two of the same sample name for the same site. For example, "OU1TB-122509" and "OU3TB-122509" would avoid any mistakes.
- Strike incorrect entries on the COC with a single line, followed by the initials of the person making the correction, the date, and the correct entry.
- When sample custody is ready to be relinquished, complete the bottom of the form with date and time (military time) and signatures of relinquisher and receiver of samples as indicated. The sample collector is always the first signature while the analytical laboratory is the final signature. Theoretically, all individuals handling the samples between collection and laboratory should sign the form; however, if a common carrier (i.e., Federal Express, UPS) is used for shipping, GEI must identify the carrier in the 'Received by' box on the

COC. If the sampler hand delivers the samples to the laboratory, the received box must be signed by the laboratory.

- If the samples are placed in a designated secure area (e.g. GEI sample fridge), note this location in the "Received by" box on the COC.
- GEI uses both single sheet and triplicate COCs. If using the triplicate COCs (white, yellow, and pink copies), the pink copy should be retained by the sampling personnel and provided to the Data Group for proper filing. The white and yellow copies should accompany the samples to the laboratory.
- If you are using the single sheet COC, make a copy of the COC after it has been signed by the lab courier and forward it to the Data Group.
- Prior to sample shipment by common carrier, the COC must be placed inside the cooler in a Ziplock bag or other watertight package.
- If a common carrier such as FedEx is used to transport the samples to the laboratory, include the carrier tracking number and identify the carrier in the "Received by" box on the COC.
- If a courier is used to transport samples to the laboratory (lab courier or GEI personnel), the courier signs the COC in the "Received by" box.
- Place a custody seal on the cooler if shipping via common carrier.
- Transport samples to the laboratory as soon as possible. It is preferable to transport the samples directly to the laboratory from the field. Samples brought back to the office for storage prior to submission to the laboratory must be kept cold (4° C).
- Unused sampling containers/media that are sent back to the lab should be included on a separate COC.
- After the samples are sent to the laboratory, the GEI copy of the COC must be forwarded to the Data Group: datagroup@geiconsultants.com.

4. Limitations

- Keep the number of people involved in handling samples to a minimum.
- Where practical, only allow people associated with the project to handle the samples.
- Always document the transfer of samples from one person to another on the COC.
- The COC should always accompany the samples.
- Give samples positive identification at all times that is legible and written with waterproof or permanent ink.
- When sending samples via a common carrier, use one COC per package.
- Where practical, avoid sending samples from more than one site with separate COCs in a single package.

5. References

New Jersey Department of Environmental Protection, Field Sampling Procedures Manual, August 2005.

Connecticut Department of Environmental Protection, Guidance for Collecting and Preserving Soil and Sediment Samples for Laboratory

6. Attachments

Attachment A - Example Chains of Custody
Attachment B - Shipping Info Pics

7. Contact

Brian Skelly
Leslie Lombardo

Chain of Custody Record

Example COC

STL Connecticut
128 Long Hill Cross Road
Shelton, CT 06484
Tel: 203-929-8140

Severn Trent Laboratories, Inc.

STL-4124 (0901)

Client: GEI	Project Manager: Dave Terry	Date: 12-31-07	Chain of Custody Number: 00452
Address: 455 Winding Brook Dr	Telephone Number (Area Code)/Fax Number: 860 368 5300 / 860 368 5307	Lab Number	Page 1 of 1
City: Glastonbury / State: CT / Zip Code: 06033	Site Contact: M. Felter	Lab Contact: Paul Mobart	
Project Name and Location (State): Carroll Gardens NY	Carrier/Waybill Number: FedEx 9383 7603 0879		
Contract/Purchase Order/Quote No.			

Sample I.D. No. and Description (Containers for each sample may be combined on one line)	Date	Time	Matrix: Air	Aqueous	Sed.	Soil	Containers & Preservatives: Unpres.	H2SO4	HNO3	HCl	NaOH	ZnAc/NaOH	Analysis: VOC 8260B	SVOC 8270C	TO-15 + NAPHTHALENE	Special Instructions/ Conditions of Receipt
CGSB-01 (0-2)	12-31-07	1130				X	X						X	X		
CGSB-02 (3-4)	12-31-07	1250				X	X						X	X		
CGSB-02 (3-4) MS	12-31-07	1250				X	X						X	X		
CGSB-02 (3-4) MSD	12-31-07	1250				X	X						X	X		
CGSB-XX (5-6)	12-31-07	0800				X	X						X	X		
CGTB-123107	12-31-07	1400				TB				X			X			
CGGW-01	12-31-07	1430		X			X			X			X	X		
CGSG-01	12-31-07	0700-1500	X												X	CANISTER # 2613 REGULATOR # 779

Per NEW JERSEY: "CLP data package deliverables"

Possible Hazard Identification
☐ Non-Hazard ☐ Flammable ☐ Skin Irritant ☐ Poison B ☒ Unknown

Sample Disposal
☐ Return To Client ☒ Disposal By Lab ☐ Archive For _____ Months (A fee may be assessed if samples are retained longer than 1 month)

Turn Around Time Required
☐ 24 Hours ☐ 48 Hours ☐ 7 Days ☐ 14 Days ☐ 21 Days ☒ Other See Contract

QC Requirements (Specify): NYS Cat B ASP deliverable send to Lorie Mackinnon

Send EDD to datagroup@geiconsultants.com

1. Relinquished By: Melissa J Felter	Date: 12-31-07	Time: 1600	1. Received By: FedEx Waybill 923117-4432	Date: 12-31-07	Time:
2. Relinquished By	Date	Time	2. Received By	Date	Time
3. Relinquished By	Date	Time	3. Received By	Date	Time

Comments: USED FLOW CONTROLLER FOR AIR SAMPLE INCLUDED

DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

EXAMPLE COC

Chain-of-Custody Record

Laboratory: Accutest

Laboratory Job # (Lab use only)

GEI Consultants
400 Unicorn Park Dr.
Woburn, MA 01801
PH: 781.721.4000
FX: 781.721.4073

Project Information

Project Name: MWRA - Low Service Storage Tank
Project Location: Stoneham, MA
Project Number: 093400
Project Manager: D. Aghjayan

Send Report to: rseigener@geiconsultants.com
Send EDD to: labdata@geiconsultants.com

MCP PRESUMPTIVE CERTAINTY REQUIRED – YES NO

If Yes, Are MCP Analytical Methods Required? YES NO NA
If Yes, Are Drinking Water Samples Submitted? YES NO NA
If Yes, Have You Met Minimum Field QC Requirements? YES NO NA

Lab Sample Number	GEI Sample ID	Collection Date	Collection Time	Matrix	No. of Bottles	Sampler(s) Initials	Preservative	PCBs	VPH Fractions Only	EPH Fractions Only	SVOCs	VOCs, % Solids**	Conductivity	RCRA 8 Metals*	TPH by GC/FID DRO	Sample Specific Remarks
							None		MeOH	None	None	MeOH	None	None	None	
							Analysis									
	093400-LS6-S5(19'-21')	12/29/2009	9:30	SO	3	JMR			x			x				
	093400-LS6-COMP (FILL)	12/29/2009	9:30	SO	1	JMR		x		x	x		x	x	x	
	093400-LS6-COMP (NATIVE)	12/29/2009	15:00	SO	1	JMR		x		x	x		x	x	x	
	093400-LS8-COMP	12/29/2009	14:00	SO	1	JMR		x		x	x		x	x	x	
	093400-LS9-S4 (8'-8'-5")	12/30/2009	14:30	SO	3	JMR			x			x				
	093400-LS9-COMP	12/30/2009	15:00	SO	1	JMR		x		x	x		x	x	x	

Sample Handling

Samples Field Filtered
YES NO NA

Sampled Shipped With Ice
YES NO

MCP Level Needed: GEI requires the most stringent Method 1 MCP standard be met for all analytes whenever possible.

Relinquished by sampler: (signature)	Date:	Time:	Received by: (signature)
1. Joseph M. Reyes	12/30/09	16:30	1. GEI FRIDGE
Relinquished by: (signature)	Date:	Time:	Received by: (signature)
2. GEI FRIDGE	1/4/10	1310	2. Naomi Stegman
Relinquished by: (signature)	Date:	Time:	Received by: (signature)
3. Naomi Stegman	1/4/10	1310	3. W. R. Odell
Relinquished by: (signature)	Date	Time	Received by: (signature)
4.			4.

Turnaround Time (Business days):

Normal _X_ Other ____
10-Day ____ 7-Day ____
5-Day ____ 3-Day ____

Before submitting rush turnaround samples, you must notify the laboratory to confirm that the TAT can be achieved.

Additional Requirements/Comments/Remarks:

Please use MA Landfill List

* Please run TCLP analysis for RCRA 8 Metals results that exceed the 20 times rule.

** Please use % solids sample for VOC and VPH analysis of 093400-LS6-COMP (FILL), 093400-LS6-COMP (NATIVE), 093400-LS8-COMP, and 093400-LS9-COMP

Chain-of-Custody Record

Laboratory:

Laboratory Job # (Lab use only)

GEI Consultants

455 Winding Brook Drive, Suite 201
Glastonbury, CT 06033
PH: 860.368.5300
FX: 860.368.5307

Project Information

Project Name/Number:

Project Location:

Project Contact:

Phone Number:

Page __ of __

Send Report to:

Send EDD to: datagroup@geiconsultants.com

Preservative

Analysis

Sample Handling

Samples Field Filtered

YES NO NA

Samples Shipped With Ice

YES NO

Deliverables:

If Yes, Have You Met Minimum Field QC Requirements? YES NO NA

Lab Sample Number	GEI Sample ID	Collection		Matrix	No. of Bottles	Sampler(s) Initials	Sample Specific Remarks
		Date	Time				

Relinquished by sampler: (signature)	Date:	Time:	Received by: (signature)
1.			1.
Relinquished by: (signature)	Date:	Time:	Received by: (signature)
2.			2.
Relinquished by: (signature)	Date:	Time:	Received by: (signature)
3.			3.
Relinquished by: (signature)	Date:	Time:	Received by: (signature)
4.			4.

Turnaround Time (Business days):

Normal _____ Other _____

10-Day _____ 7-Day _____

5-Day _____ 3-Day _____

Before submitting rush turnaround samples, you **must** notify the laboratory to confirm that the TAT can be achieved.

Additional Requirements/Comments/Remarks:

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

PACKING SAMPLES FOR SHIPMENT BACK TO THE LABORATORY

A. Line cooler with bubble wrap and large plastic bag. Use absorbent pad inside the bag if bottles contain preservatives.

B. Wipe outside of bottles and put glass in individual bubble bags & seal. Place bottles & the temperature blank into cooler. Leave room for ice in between bottles & on top.

C. Place double bagged or loose ice randomly around bottles throughout the cooler.

D. Place large bag of ice or loose ice on top of the bottles. In warm weather, the cooler should be packed with as much ice as possible.

E. Close outer bag, compress excess air out of bag, twist top and knot. If necessary, use more bubble wrap to fill the dead air spaces. Place chain of custody (COC) and other paperwork in plastic bag and seal. Place on top of cooler.

F. Close cooler, place signed and dated Custody Seals over opening. Tape over the Custody Seal and seal cooler securely. Fill out overnight shipping waybill and attach to the top or handle of the cooler. Attach Saturday delivery stickers if needed. Ship according to DOT regulations.

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

PACKING SAMPLES FOR SHIPMENT BACK TO THE LABORATORY

A. Line cooler with bubble wrap and large plastic bag. Use absorbent pad inside the bag if bottles contain preservatives.

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C. Place double bagged or loose ice randomly around bottles throughout the cooler.

D. Place large bag of ice or loose ice on top of the bottles. In warm weather, the cooler should be packed with as much ice as possible.

E. Close outer bag, compress excess air out of bag, twist top and knot. If necessary, use more bubble wrap to fill the dead air spaces. Place chain of custody (COC) and other paperwork in plastic bag and seal. Place on top of cooler.

F. Close cooler, place signed and dated Custody Seals over opening. Tape over the Custody Seal and seal cooler securely. Fill out overnight shipping waybill and attach to the top or handle of the cooler. Attach Saturday delivery stickers if needed. Ship according to DOT regulations.

STANDARD OPERATING PROCEDURE

FD-004 Photo Documentation

1. Objective

Describe methods to document and retain photographic records.

Keeping a record of photographs taken is crucial to their validity as a representation of existing conditions.

2. Execution

- Photographs of a site, individual samples, or other observations should be taken using a digital camera.
- Set the camera to record the time and date for each photograph.
- All photographic records, along with the following information, should be recorded in the field notebook (SOP FD-001).
    - If applicable, the compass direction describing the direction the photograph was taken (e.g. looking southeast). This may not apply to photographs of individual samples.
    - Brief description of what the photograph is intended to show.
- The field notebook should note who took the photographs.
- The photographs should be electronically backed up on a computer or other data storage device.
- If photographs will be used in a report, memo, or letter, they should be placed on a photograph record template and the relevant information describing the photograph should be inserted into the caption section for each photograph.

3. Limitations

- Some clients and regulatory agencies require photographs of every subsurface soil sample collected. These photographs typically include a “whiteboard” which indicates the site, the boring ID, and the depth of the sample, while logging details are recorded in the field notebook. Under these circumstances, it is not necessary to include compass directions or descriptions.

4. References

New Jersey Department of Environmental Protection, Field Sampling Procedures Manual, August 2005.

5. Attachments

Attachment A – Example of Photo Documentation Template

6. Contact

Melissa Felter
Leslie Lombardo

Attachment A – Example of Photo Documentation Template
GEI Consultants, Inc.

Project: Project Name

Location: Project Location

Photographer: K. Barber
Date: 10/25/07
Photo No.: 1
Direction: N

Comments:
Entrance of site with tree mulching operations.

Photographer: K.Barber
Date: 10/25/07
Photo No.: 2
Direction: W

Comments:
On-site building built in 1936.

STANDARD OPERATING PROCEDURE

FD-006 Handheld Global Positioning Receiver Operation

1. Objective

Use handheld global positioning system (GPS) receivers to locate sample points and site features with "Mapping-Grade" accuracy.

Use handheld GPS receivers to "stake out" proposed sample point locations within the limits of "Mapping Grade" accuracy.

2. Execution

- Handheld GPS receivers provide a low-cost and user-friendly method for locating sample points and site features with a fair degree of horizontal accuracy.
- In simplistic terms, GPS works by measuring the distance from numerous orbiting satellites to a point on the earth surface. Individual satellites broadcast their real-time location in terms of x,y and z coordinates, and the distance from each satellite is measured as a function of the length of time that a time-stamped signal takes to reach the receiver. Built-in GPS software derives new points by intersecting the distances from known orbital locations – in much the same way that points are located by intersecting tape-measured distances from building corners or other pre-existing site features.
- Late-model handheld GPS receivers utilize a real-time differential correction technique called WAAS (Wide Area Augmentation System). This system was designed to provide greater confidence and reliability in using GPS data for commercial aircraft landing approaches, and the additional correction improves all GPS operations.
- Handheld GPS receivers display navigational information on a variety of standard pages. Although each manufacturer uses slightly different formats, all receivers toggle back and forth between the following visual presentations:
- A "satellite" page displays the relative orbital location of all GPS satellites that are currently being tracked by the receiver. The display may include information on the real-time geometrical strength of the solution: satellite intercepts that cross at right angles provide more accurate solutions than intercepts that cross at acute or obtuse angles.
- A "track" page that displays the travel path of the receiver while it is turned on, along with the relative location of recorded points. Many GPS models have a "track-back" function that will guide the user on the same path back to the starting point
- A "navigation" page that displays instantaneous location and the real-time direction and velocity of travel. Some units provide two pages to display this information in different formats. Most units will report the overall "course

made good" (straight-line bearing and distance from the starting point) at any point.

- A "waypoint" page that allows users to "Go To" a created point or previously recorded point by providing a straight-line bearing and distance to the point. The information is instantaneously updated as the user moves along; some units display a pointing arrow that directs the user to the direction of travel. Be careful of go-to lines that lead through swamps or over cliffs – if you will be travelling in difficult terrain have a paper copy of the USGS quadrangle and a compass on hand for navigation.
- Signal strength degrades significantly next to buildings and underneath tree canopy. Most GPS receivers have an "averaging" function to improve the accuracy of shielded locations. GPS users can also improve precision by locating points three times, at different times of the day. Two of the solutions will generally be closer to each other than to the third and can be averaged for a more reliable fix.
- Most GPS receivers default to latitude and longitude, but data is more accurate and easier to input and when expressed in UTM coordinates to the nearest meter. The handheld GPS setup will have a function somewhere to change to UTM. Most of Connecticut is in UTM Zone 18 but the easternmost parts are in Zone 19.
- Consult "Corpscon" the datum translator available from the National Geodetic Survey website. Corpscon translates instantly from latitude/longitude to UTM coordinates to state plane coordinates and provides tools to identify UTM Zones. Also consult the Trimble, Garmin and Magellan websites for technological improvements and discussion of advanced techniques.

3. Limitations

- Handheld GPS receivers operating in unobstructed locations are currently reckoned to provide 2-5 meter accuracy, meaning that the true location of measured points lie within an "error ellipse" with axes of 2-5 meters centered on the measured location. In other words, even under the best of conditions a real-time GPS solution may be as much as 20 feet off the true horizontal location of a point.
- Due to geodetic restrictions, vertical locations (elevations) have less than half the accuracy of horizontal locations, meaning that even under the best of conditions, a surface elevation displayed on a handheld GPS receiver may be off by more than 50 feet.
- Horizontal and vertical data derived from handheld GPS receivers should never be considered more than relatively accurate, and this level of uncertainty should be identified in any discussion of positional tolerance.

4. References

Trimble Website: .trimble.com
Garmin Website: .garmin.com

Magellan Website: .**magellan**gps.com
National Geodetic Survey: ://www.ngs.noaa.gov/

5. Contact

Doug Bonoff, PLS

STANDARD OPERATING PROCEDURE

GW-001 Water Level and NAPL Measurement

1. Objective

Describe procedures to measure the depth to water and non-aqueous phase liquid (NAPL) thickness in an open borehole, cased borehole, monitoring well or piezometer.

2. Equipment and Materials

Field forms and/or field notebook.

- Decontamination fluids
- Bailer
- Weighted cotton string
- Oil/Water interface probe
- Water level meter (if oil/water interface probe is not available)

Water level and NAPL measurements can be collected by a variety of methods. A water level meter is used to collect depth to water measurements however an oil/water interface probe or other methods must be used to gauge NAPL depths. An electronic oil/water interface meter, consists of a cable divided into incremental measurements of 0.01 feet, and probe that consists of an infra-red circuit that detects the presence of a liquid, and a conductivity circuit that differentiates between conductive liquid (water) and non-conductive liquid (LNAPL or dense non-aqueous phase liquid [DNAPL] product). Typically, a steady tone and light indicate a non-conductive liquid (e.g. product) and an intermittent tone and light indicate a conductive liquid (e.g. water). Refer to the manufacturer's instructions for details. Alternately, water level and NAPL measurements can be collected using a water level meter, clear bailer and weighted cotton string. Each method of data collection is described below.

3. General Information

- The water level in a monitoring well or piezometer should be allowed to stabilize for a minimum of 24 hours after development or construction before groundwater elevation and/or NAPL measurements are collected. The water level in a borehole can be measured during drilling; however, this should be noted in the field notebook.
- Water levels in multiple wells should be collected within the shortest timeframe practicable.
- Water and NAPL levels should be measured from the designated survey point as specified by the surveyor or highest point (or "V" notch) on the PVC. If the well is new, mark the datum point with an indelible marker and note reference location in

field book. Discuss with the project manager what reference point should be used to collect water measurements for specific sites.

- Water level and/or NAPL measurements should be made before any water is removed from wells because doing so may influence groundwater levels in the area of the investigation.
- Measurements should be made approximately three times to confirm the measurement. Each time a measurement is made it should be determined to the nearest one-hundredth of a foot (0.01).
- Water level and/or NAPL measurements should first be collected at the wells that are least contaminated and proceed towards the wells that are most contaminated. Decontaminate the water level meter or oil/water interface probe prior to initial use and after use at each location. If NAPL is encountered at a well where it was previously not observed, contact your project manager before continuing.
- Refer to the oil/water interface probe or water level meter instruction manual for guidance on indicator signals, as these may differ by manufacturer.

4. Execution

4.1 Water Level and NAPL Measurements Using Interface Probe

- Open wells to the atmosphere and allow them to equilibrate prior to collecting LNAPL depth measurements.
- LNAPL Depth (if present): Measure the LNAPL/air interface by slowly lowering the interface probe to the LNAPL surface. Be ready to stop as soon as the probe signals the LNAPL surface.
- Record the depth to LNAPL.
- Groundwater Depth: Continue slowly lowering the probe until it signals the presence of water.
- Record the depth to water.
- The LNAPL thickness is determined by subtracting the water depth from the LNAPL depth.

The depth and thickness of DNAPL can sometimes be determined by slowly lowering the interface probe past the LNAPL (if present) and water layers. Record the depth to the DNAPL layer. Finally, measure the depth to the well bottom.

The DNAPL thickness is determined by subtracting the DNAPL depth from the depth to well bottom.

- Decontaminate the interface probe and tape according to SOP QA-001.
- Dispose of any NAPL-impacted debris properly.
- Check with the Project Manager if you are uncertain of the appropriate disposal method.

4.2 LNAPL Measurements Using Clear Bailer

If LNAPL is suspected at a site, an oil/water interface probe should be used when gauging water level and NAPL measurements. However, a water level meter and a clear bailer may be used instead to estimate approximate LNAPL thickness if an oil/water interface probe is not available.

- Open wells to the atmosphere and allow them to equilibrate prior to collecting LNAPL depth measurements.
- Slowly lower the water level meter until contact with fluid is indicated by the meter.
- Record the depth to fluid measurement.
- Lower a clear bailer into the well and slowly into the LNAPL. Do not submerge the bailer.
- Slowly raise the bailer out of the well and measure LNAPL thickness in the bailer using a ruler or tape measure.

Calculating Depth to Groundwater

The depth to water can be calculated as follows:

DTW = DTF + PT
DTW = Depth to Groundwater
DTF = Depth to Fluid
PT = Measured Product Thickness

Calculating Corrected Depth to Groundwater

Once the LNAPL thickness is known and the depth to groundwater is known, the corrected depth to groundwater can be calculated.

Corrected DTW = Static DTW – (PT x G)
DTW = Depth to Ground Water
PT = Measured Product Thickness
G = Specific Gravity (density of free product / density of water)

4.3 DNAPL Measurements Using Weighted Cotton String

A weighted cotton string may be used to estimate approximate DNAPL thickness.

- Secure cotton string.
- Secure clean steel nuts and/or washers.

- Tie the string to the nuts/washers, so that there is adequate weight.
- Lower the weighted string into the well slowly, until a firm bottom is sensed.
- Remove the weighed string and measure the DNAPL coated portion of the string.
- Record the thickness.
- Dispose of any NAPL-impacted debris properly. Check with the Project Manager if you are uncertain of the appropriate disposal method.

5. Health and Safety Considerations

The health and safety considerations for the work associated with this SOP, including both potential physical and chemical hazards, will be addressed in the site specific Health and Safety Plan (HASP). The collection and accumulation of NAPL presents the potential for significant hazards that need to be managed. A detailed job safety analysis (JSA) should be completed prior to the start of work.

6. Considerations

- Weak batteries in water level and oil/water interface meters frequently produce weak or gradual auditory and/or visual responses, making it difficult to accurately determine when the probe of the unit has come in contact with ground water or NAPL. As such, it is recommended that electronic ground water-level indicators be tested before they are brought out into the field.
- Electronic oil/water interface meters do not respond to distilled water. Do not use de-ionized water to test these units.
- Wells that are not vertical may result in probe contact with the side of the well casing providing a false measurement. Once the probe has come in contact with ground water in the well, water may be trapped by capillary action between the probe and the well casing. If this happens, the unit may continue to signal even after the probe has been raised above the ground water surface. The deeper the well, the more likely this problem may occur. To correct this, the cable should be raised several feet above the water and shaken to remove water from the probe. A new ground water-level measurement should then be collected. If the signals from the unit are not abrupt or reproducible, the probe and tape may need to be retrieved and dried off before trying again.
- Accumulation of sediment, organic material, or floating debris in the probe may also result in gradual or non-reproducible readings. Wells that are constructed with metal inner casings may lead to difficulties in collecting reproducible ground water-level measurements because the inner sides of the well casing are conductive.

- In some cases, a rubber grommet or metal centralizer may need to be placed on the probe so that it cannot contact the inner casing.
- Well gauging equipment should be properly decontaminated between wells and piezometers to avoid cross contamination.
- Water levels in wells may be influenced by changes in river stages, pumping of nearby wells, precipitation, tides, etc.
- Using a bailer to estimate LNAPL thickness can result in inaccuracies because successful use of the bailer is dependent upon the expertise of the operator and assumes the check valve does not leak upon retrieval.
- The optical sensor on interface probes may become damaged if solvents are used to clean NAPL from the probes.
- The optical sensor may become smeared when used to measure NAPL, rendering pinpoint accuracy to an estimate at best.
- Close attention to decontamination procedures will improve accuracy, operational life, and reduce the risk of cross contamination with other wells.
- LNAPL thickness can be affected by fluctuations in the water table. In some cases, an LNAPL's thickness may decrease when the water table rises, while its thickness increases as the water table drops. In other cases, fluctuating water tables may cause sudden appearances and disappearances of LNAPL layers.
- Monitoring points with LNAPL can pose a problem when measuring the level of groundwater. Floating LNAPL can depress the groundwater level in a monitoring well or piezometer and distort the measurement. Therefore, the Corrected Depth (CD) formula shown above should be applied to groundwater level measurements in monitoring points where LNAPL are present:
- Some interface probes are factory-calibrated based on an assumed conductivity of NAPL and water, both of which may vary. An interface probe that is functioning properly may not be able to discern different NAPLs at all sites.
- An interface probe may not successfully provide both LNAPL and DNAPL measurements in the same well because the probe is coated by LNAPL and loses its ability to detect DNAPL.
- DNAPL, in particular, may be only slightly heavier than water, or may be neutrally buoyant. As a result, it can be easily disturbed. Once it is disturbed, meaningful measurements can be difficult or impossible to obtain. As such, all tapes or probes used for measurements should be used slowly.

7. References

U.S. EPA Environmental Response Team Standard Operating Procedures SOP: 2043,"Water Level Measurement" REV: 0.0, 2/11/00

U.S. EPA Environmental Response Team Standard Operating Procedures SOP: 2044," Monitor Well Development" REV: 0.1, 10/23/01.

8. Contacts

Brian Conte – (860) 368-5412 Glastonbury
Mark Ensign – (781) 721-4010 Boston
Ryan Hoffman – (781) 721-4091 Boston

STANDARD OPERATING PROCEDURE

GW-003 Low Flow (Low Stress) Groundwater Sampling

1. Objective

Describe methods to collect groundwater samples most likely to produce results that represent aquifer conditions.

Low-flow purging is limited to wells that, with sustained pumping, exhibit no continuous drawdown.

2. Execution

- Prior to groundwater sampling consult with the project manager to confirm that the type of pump is appropriate and consistent with the approved work plan.
- Record activities in the field notebook (see SOP FD-001 Field Notebook) and on a Monitoring Well Sampling Record such as the examples in Attachment A. Use a separate form for each sampling location and event. You may forego the forms and record all information in the field notebook if the Project Manager approves.
- Calibrate pH, temperature, Specific Conductance (SC), turbidity, Dissolved Oxygen (DO), and Oxidation-Reduction Potential (ORP) on the meter(s). Use calibration methods provided by the manufacturer of the equipment. Note that appropriate calibration for dissolved oxygen requires a water saturated air environment, along with measured temperature and barometric pressure.
- Begin with the monitoring well believed to have the least contaminated groundwater and proceed systematically to the well with the most contaminated groundwater. Check the well, the lock, and the locking cap for damage or evidence of tampering.
- Slowly and gently measure the depth to water with a water level probe and/or oil-water interface probe. Do not measure depth to well bottom at this time (wait until sampling has been completed). Measure water level in accordance with SOP GW-001 Water Level Measurement.
- Attach new polyethylene or Teflon lined tubing to the sampling pump and the flow-through cell that contains the meter probes.
- Slowly and gently insert new polyethylene or Teflon lined tubing to the pump intake (or use dedicated tubing that remains in the well) and to the middle of the saturated screened interval or to the pre-determined sampling depth.
- The tubing intake should be kept at least two (2) feet above the bottom of the well to prevent disturbance or suspension of any sediment or Non-Aqueous Phase Liquid (NAPL) present in the bottom of the well. Record the depth of the pump intake.

- If possible, position your sampling equipment and tubing so that it is in the shade. The goal is to minimize the effect of sunlight raising the temperature of water being collected.
- Start the pump on the lowest setting and increase slowly until flow begins. Adjust the pumping rate so that drawdown in the well is minimal (0.3 feet or less, is desirable but not mandatory). Use a pumping rate between 100 to 1,000 milliliters per minute (mL/min) (or approximately 0.1 to 1 quarts per minute). Measure flow rate on the pump or using a graduated container every 3 to 5 minutes and record. The minimum purge volume will be twice the combined volumes of the sampling string (i.e. pump, tubing, and flow-through cell).
- While purging, record water levels every 3 to 5 minutes and monitor and record the water quality indicator parameters: pH, temperature, specific conductance (SC), dissolved oxygen (DO), and turbidity. If specified in the field sampling plan also include ORP.
- Purging is complete when, after three consecutive measurements, the water quality parameters have stabilized as follows:

    - pH (+/- 0.1 standard units)
    - temperature (+/- 3%)
    - SC (+/- 3%)
    - turbidity (+/- 10% if >5 NTU; if 3 values are <5 NTU, consider the values as stabilized)
    - DO (+/-10% if >0.5 mg/L; if 3 values are <0.5 mg/L, consider the values as stabilized)
    - ORP (+/- 10 mV)

- Dispose of purge water according to the field plan.

Sample Collection:

- Following purge, remove the discharge tubing from the flow-through cell. Do not disturb pump and tubing between stabilization and sample collection.
- Fill sample containers directly from the sampling device in order of decreasing volatility (i.e., Volatile Organic Compounds (VOC) samples are collected first; see SOP SC-002 Sampling Handling). Fill all containers from the discharge end of the tubing. Collect samples at a flow rate equal to the steady state purge rate.
- If not using a dedicated pump, remove sampling device and decontaminate (see SOP QA-001 Equipment Decontamination). Discard used tubing.
- Store samples in a cooler on ice for transport to the laboratory.
- Measure depth to bottom of well.

- Secure the well cap.

3. Limitations

- Prior to departure for the field, obtain available information on well construction for use in field investigation (i.e., screen and riser material, well diameter and depth, screened interval, optimum sampling depth, etc.).
- If possible, when using dedicated equipment, install equipment into well at least 24 hours before sample collection to minimize disturbance of the water column and/or suspension of sediments or NAPL on bottom.
- If water quality indicator parameters do not stabilize after removing 3 to 5 well volumes or 2 hours, contact the Project Manager. Three options will be available: 1) continue purging until stabilization; 2) discontinue purging and do not sample; or 3) discontinue purging and sample.
- The key indicator parameter for VOCs is DO. The key indicator parameter for all other samples is turbidity.
- Fill all sample containers with minimal turbulence by allowing the groundwater to flow from the tubing gently down the inside of the container.
- Consult with the project manager before field filtering samples for metals if using low-flow sampling.
- Be aware of any preservatives in the sample bottles and handle with care, in accordance with the Health and Safety Plan.

4. References

Standard Reference for Monitoring Wells (April 19, 1991), Massachusetts DEP, DEP Publication No. WSC-310-91.

Reproducible Well-Purging Procedures and VOC Stabilization Criteria for Ground Water Sampling (1994), M.J. Barcelona, H. A. Wehram, and M.D. Varljen, Ground Water, Vol. 32, No. 1, 12-22.

Low-Flow Purging and Sampling of Ground Water Monitoring Wells with Dedicated Systems (1995), R.W. Puls, and C.J. Paul, Groundwater Monitoring and Review, Summer 1995 116-123.

Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells (2010), EQASOP-GW 001 Low Stress (Low Flow) SOP, Revision 3, U.S. Environmental Protection Agency, Region I, January 19, 2010.

Ground Water Sampling Procedure Low Stress (Low Flow) Purging and Sampling, (1998), Ground-Water Sampling SOP, Final, U.S. Environmental Protection Agency, Region II, March 16, 1998.

RCRA Ground-Water Monitoring: Draft Technical Guidance, (1993), U.S. Environmental Protection Agency, EPA/530-R-93-001.

To Filter, or Not to Filter, That is the Question, (1997), Special Topics Subcommittee Letter Report EPA-SAF-EEC-LTR-97-011, April 29, 1997, Meeting, U.S. Environmental Protection Agency, Science Advisory Board Environmental Engineering Committee, September 5, 1997.

Should Filtered or Unfiltered Groundwater and Surface Water Samples be Collected for the Risk Assessment?, (1995), MCP Q&A: Subparts I and J, Special #4, Bureau of Waste Site Cleanup, Massachusetts Department of Environmental Protection (DEP), February, 1995.

5. Attachments

Attachment A - Monitoring Well Sampling Record

6. Contacts

Brian Conte
Saskia Oosting

Monitoring Well Development / Sampling Log

GEI Consultants

Site Name:	Well ID:

Date_____

Field Personnel_____

Method of Purging/ Sampling_____

Well Depth (ft btoc)_____

Depth to Water (ft btoc)_____

Casing type/dia._____

Well Volume (g)_____

Time	Total Volume (g)	Temp (°F)	pH	Cond (us/cm)	ORP (mV)	D.O. (mg/L)	Turbidity (NTU)	Comments

STANDARD OPERATING PROCEDURE

GW-004 pH and Temperature Measurement

1. Objective

Describe methods for measuring the pH and temperature of liquids using a combination pH/temperature meter.

2. Execution

Calibration

- Calibrate the meter according to the equipment manufacturer's instructions at the beginning of each day of use. Calibration for pH shall be performed using at least two buffer solutions. Solutions chosen should be similar to the expected pH of the liquids tested (pH 7 and 4 buffer solutions are preferred in most cases for groundwater or surface water measurements).
- Check calibration at the end of the day by reading the two solutions used in calibration. Also perform additional field checks as needed based on observed readings (i.e., inconsistent readings). Record measurements and time of measurement in the field book or sample sheet. If the readings are outside +/– 0.2 pH units, recalibrate the meter.

Sample Measurement

- Immediately prior to testing a sample, decontaminate testing container and probe assembly with one rinse of distilled water. Do not use methanol to rinse the probe. Methanol rinses could damage the probe.
- Gently dry the probe with a paper towel and shake beaker to remove excess solution. Visually inspect the bottom of the probe to ensure that liquid or sediment is not trapped between outer casing and probe.
- Pour the sample into the testing container and insert both temperature and pH probe. Stir sample for 30 seconds using both probes. Let the probes equilibrate in the sample solution for another 30 seconds. Measure and record the temperature. Measure and record pH reading after stabilization or 60 seconds, whichever is sooner. A reading has stabilized if pH units have not changed +/– 0.1 pH units during a 30 second period.
- Record pH to the nearest 0.1 unit and temperature to the nearest whole number.

3. Limitations

- Coatings and particulates may affect the response of the probe; more thorough cleaning using a weak alconox solution and distilled water rinse

and gently wiping the probe surface with a paper towel may be required to clean the surface of the probe.

- Temperature affects both the response of the instrument to pH and the actual pH of the sample. The Automatic Temperature Compensation (ATC) function compensates for the variation in the response of the meter only. Therefore, the pH must always be reported with temperature.
- The probe is a fragile thin glass bulb surrounded on three sides by a plastic casing. Care must be taken in handling the probe to avoid breakage.
- Do not use buffer solutions past their expiration date.

4. References

Standard Methods for the Examination of Water and Wastewater, 18th Edition, Method 4500-H. American Public Health Association (1992).

5. Contacts

Brian Conte
Saskia Oosting

STANDARD OPERATING PROCEDURE

GW-005 Turbidity Measurement

1. Objective

Describe calibration and use of a Hach nephelometer/turbidimeter.

The meter is used to measure turbidity of liquids by quantifying how much light passes through them. Turbidiity readings are required to be read using a portable (e.g Hach) instrument directly from the tubing before going through the flow-through cell.

This SOP is specific to a Hach turbidimeter. Follow manufacturer's recommendations for other meters.

2. Execution

i. Turn the meter "ON".
ii. Rinse the sample cell 3 times with distilled water.
iii. Fill the cell to the fill line with distilled water and then cap the cell.
iv. Wipe off excess water and streaks with a non-abrasive lint-free paper or cloth (preferably lens paper).
v. Open the cover and insert the cell (arrow to the front) into the unit and close the cover.
vi. Press "READ" and wait for the 'light bulb' icon to go off. Record the reading.
vii. Using the Gelex standards, repeat steps above. Record all measurements (note anomalies).
viii. Fill the cell with sample liquid to the fill line (about 15 mL) and replace the cap on the cell.
ix. Wipe off excess water and any streaks with a non-abrasive lint-free paper or cloth (lens paper).
x. Press "I/O" and the instrument will turn on. Place the meter on a flat, sturdy surface. Do not hold the instrument while making measurements.
xi. Insert the sample cell, arrow to the front, in the instrument. Close the lid.
xii. Select manual or automatic range selection by pressing the range key.
xiii. Use signal average mode if the sample causes a noisy signal (display changes constantly). Select signal averaging mode by pressing the "Signal Average" key.
xiv. Press Read. The display will show "---- NTU" and then the turbidity in NTU. Record the result after the lamp symbol turns off.
xv. Rinse the cell with distilled water.
xvi. Confirm the validity of the sample measurement by double-checking with one of the Gelex standards.

xvii. Periodically check the turbidity meter during the day by using the Gelex secondary standards provided.
xviii. Perform a post calibration at the end of the day and record all measurements.

3. Limitations

If the turbidity measurements are for National Pollutant Discharge Elimination System (NPDES) reporting purposes, all samples with values above 40 NTU must be diluted with turbidity free water (e.g. distilled water) and sample turbidity is calculated by multiplying the reading of the diluted sample by the dilution factor.

4. References

Standard Methods for the Examination of Water and Wastewater, 18th Edition, Method 4500-H. American Public Health Association (1992).

5. Contacts

Brian Conte
Saskia Oosting

STANDARD OPERATING PROCEDURE

GW-006 Specific Conductance Measurement

1. Objective

Describe standard methods to measure conductivity of water using a field conductivity meter.

2. Execution

- Calibrate the meter according to equipment manufacturer's instructions at the beginning of each day of use. Calibration shall be performed using a standard KCl or other solution recommended by the manufacturer.
- Record the make, model, and serial or identification number of the instrument and calibration information in the field notebook.
- Check calibration at the end of the day by measuring the standard used in calibration and record in field book. Also perform additional field checks as needed based on observed readings (i.e., inconsistent readings). If the readings are outside +/- 0.02 mS/cm, the meter must be recalibrated. Initial calibration should be conducted under the same conditions (i.e., temperature, and location) of field testing.
- Immediately prior to testing a sample, decontaminate testing container and probe assembly with distilled water.
- Gently dry the probe with a paper towel and shake container to remove excess solution.
- Pour sample into the container and insert probe. Stir sample with the probe for approximately 10 seconds. Let the probe equilibrate in the sample solution for another 30 seconds. Measure conductivity and record in the field notebook.
- Record conductivity to the nearest whole number.

3. Limitations

- Oily coatings and particulates may affect the probe's response; more thorough cleaning using a weak alconox solution and distilled water rinse and gently wiping the probe surface may be required to clean the surface of the probe.
- If sample liquid is contaminated, (e.g. stained, conductance >0.75 mS/cm), rinse probe with distilled water immediately after measuring sample to minimize fouling of probe.
- Do not use calibration solutions past their expiration date.

4. References

Standard Methods for the Examination of Water and Wastewater, 18th Edition, Method 4500-H. American Public Health Association (1992).

5. Contact

Brian Conte
Saskia Oosting

STANDARD OPERATING PROCEDURE

GW-007 Dissolved Oxygen Measurement

1. Objective

Describe calibration and field use of dissolved oxygen meter.

2. Execution

- Place instrument in the intended operating position (vertical, tilted, or horizontal) before it is prepared for use and calibrated.
- Recalibration may be necessary when the instrument operating position is changed.
- Attach the prepared probe to the Probe connector of the instrument and adjust the retaining ring finger tight. Check that membrane is intact and check for presence of air bubbles under membrane. If bubbles are present or membrane is damaged, prepare probe again according to manufacturer's instruction.
- Place approximately 1/8 inch of water into the bottom of the calibration cup. Place the probe into the cup and engage only one thread of the calibration cup onto the probe to ensure that the DO probe is readily vented to the atmosphere. Make sure the DO and temperature probes are not in contact with the water. Wait approximately 10 minutes for the air in the calibration cup to become water saturated and for the temperature to equilibrate.
- Calibrate meter according to the procedures outline in the instrument manual. Calibrate probe to a zero oxygen solution provided by manufacturer, and water saturated air.
- The calibration procedure may require correction factors or input of site-specific barometric pressure and temperature. Correction factors can be found at:

  http://water.usgs.gov/owq/FieldManual/Chapter6/6.2_v2.1.pdf

- Otherwise, use appropriate instruments at the site to determine temperature and pressure.
- Perform Dissolved Oxygen Measurement using the following procedure:
  i. Submerge probe in flow-through chamber or water body.
  ii. Gently raise and lower probe in sample.
  iii. Allow sufficient time for probe to stabilize to sample temperature and dissolved oxygen.
  iv. Read and record the temperature and the value of the dissolved oxygen in mg/L.
  v. Document field analysis data and general observations in the field log book or groundwater sampling sheet.

3. Limitations

- Collect DO measurements in the field during sampling. Storing samples in containers will alter the DO concentration of the sample.
- Detection Limit (DL) = 0.1 mg/L for 0-10 mg/L range; do not record values less than Detection Limit: a zero reading is recorded < 0.1 mg/L.

4. References

Standard Methods for the Examination of Water and Wastewater, 18th Edition, Method 4500-H. American Public Health Association (1992).

5. Contacts

Brian Conte
Saskia Oosting

STANDARD OPERATING PROCEDURE

GW-008 Temporary Groundwater Sampling Points

1. Objective

To define the procedures for installation of temporary groundwater sampling points (hereafter referred to as well point) for measuring depth to groundwater and collecting groundwater samples. Well points may aid in the placement of permanent monitoring wells.

A well point is a small diameter (1-2 inch) probe constructed of continuously wrapped stainless steel or wrapped stainless steel gauze screen over perforated carbon steel pipe. No filter or gravel pack is used in the installation.

Well point installations are not the only type of temporary monitoring wells. Alternative temporarily well constructions should be discussed with the project manager and may be more appropriate based on-site conditions.

2. Execution

2.1. Installation

- The well point can be placed with the use of a conventional hollow-stem auger rig, Geoprobe®, slide hammer, jack hammer, rotary hammer, or by hand.

- The well point may be driven through the unsaturated zone only in known "clean" soils. Driving the well point through contaminated soil may carry contamination downward with the point resulting in analytical sample results which are biased high. In areas with contamination above the desired screening zone, the well points should be installed with the aid of either hollow-stem augers or Geoprobe®, to “case off” contamination from the upper layer.

- If the well point is to be installed in an oversized (20% larger than the well point) pre-drilled hole, the hollow-stem augers or bull drive point must be advanced to a point which is just above the targeted sample zone. The well point is then placed in the hole and advanced beyond the bottom of the hole by hammering or pushing into place. The use of pre-drilled holes will reduce clogging of well point screens when driving.

- If the well point is used for piezometeric data, make a survey mark on top of the casing as a reference point for water level measurements.

- Caution must be used when using well points in areas of contaminated soil. Possible cross contamination may be introduced to the screen as it passes through the zone of contamination.

2.2. Sampling Procedures

Development of a well point is not required prior to sampling. Sampling of groundwater or collecting piezometric data must be performed by one of several recommended methods described in this manual.

After sample collection, (See Groundwater Sampling SOP) the well point is removed by back hammering or pulling the tool out with the rig hydraulics.

3. References

ASTM D6001 - 05 Standard Guide for Direct-Push Water Sampling for Geoenvironmental Investigations

Standard Methods for the Examination of Water and Wastewater, 18th Edition, Method 4500-H. American Public Health Association (1992).

Ground Water and Wells. Johnson Division, UOP Inc.; St. Paul, Minn. 1982. p277-294.

Ground Water Manual - A Water Resources Technical Publication; U.S. Dept. of Interior, Bureau of Reclamation. Government Printing Office, Washington DC 1977.

Standard References for Monitoring Wells (April 1991), Commonwealth of Massachusetts Department of Environmental Protection, WSC-310-91.

4. Contacts

Brian Conte
Saskia Oosting

STANDARD OPERATING PROCEDURE

PM-001 Utility Markout and Clearance

1. Objective

Describe typical utility markout/clearance procedures prior to and during excavation. All markout procedures should be performed in accordance with local and state regulations.

Many states, by law, require that utility companies are notified before excavation begins. Actual procedures and requirements differ by state. City/state government may have additional requirements for utility markout procedures. Check the requirements in the locality before beginning. Also check with the GEI project manager to determine whether it is most appropriate for GEI or the drilling/excavation subcontractor to handle mark out and clearance.

2. Execution

Public Utility Markouts

- The drilling/excavation locations should be marked with white paint, stakes, or flags.
- The contractor should call the appropriate one call communication network for the state the work is being conducted in. Refer to the Reference section in this SOP for contact information. Contractors shall provide all necessary information to the one call system. Sample location maps may be provided to clarify sampling locations. The contractor shall provide GEI with the utility clearance ticket number.
- If necessary, contact the municipalities or other utility owners to mark their water, sewer, or other lines if they do not belong to the one call system. The contractor should keep a record of these calls.
- Utility plans, if available, should be obtained from the property owner or municipal offices.
- Prior to excavation, a visual check should be made that all utilities companies and municipalities have marked their locations. This includes looking for signs that a utility exists, and verifying that the markings agree with a visual check. If they do not, contact the appropriate utility to remark their locations.
- Utilities will generally only markout and clear utilities in roadways or other public property up to the property boundary. Owners of fiber optic cables, natural gas pipelines, and other high hazard utilities will often markout on private property if requested. The contractor should request this. See section on private utility clearance below.
- Public agencies, utilities, contractors, other associations, manufacturers and all others involved in excavation should adopt the American Public Works Association (APWA) Uniform Color Code using ANSI standard Z535.1 Safety Colors for temporary marking and utility identification, as follow:

The APWA Uniform Color Code

1. **White** – Proposed Excavation
2. **Pink** – Temporary Survey Markings
3. **Red** – Electric Power Lines, Cables, Conduit and Lighting Cables
4. **Yellow** – Gas, Oil, Steam, Petroleum & Gaseous Material
5. **Orange** – Communications, Alarm or Signal Lines, Cables or Conduit
6. **Blue** – Potable Water
7. **Purple** – Reclaimed Water, Irrigation and Slurry Lines
8. **Green** – Sewers and Drain Lines

However, not all organization do use these colors. Make sure the color scheme is understood.

- The excavator/contractor and consultant begins work on the scheduled work date and time (if all the facility operators have responded) taking care to find and preserve any markings that have been made.
- If markings may be disturbed during work, establish offset marks to create reference points for the underground utilities. Take photographs of the markings before starting work.
- When digging near a buried utility, the excavator/contractor and consultant should be aware of their proximity to the utility and use caution.
- If there is uncertainty about the accuracy of the markings or there are too many utilities in a given location to excavate or drill safely, consideration should be given to hand-digging the first few feet, vacuum excavation, or use of a utility location company as detailed below in Private Utility Markouts.
- Some clients may require hand or vacuum clearance to a minimum depth. Check with the GEI project manager.
- If exposing a utility, the excavator/contractor should provide proper support and protection for the utility to prevent damage. Contact the utility operator for support, guidance, or assistance.
- When the excavation is complete, the excavator/contractor should provide proper backfill for any utilities that have been exposed.

Private Utility Markouts

- Utility markouts on private property should follow the steps outlined above in public utility markouts with the additions below.
- If work is conducted on private property where public utilities may not provide markouts and the property owner cannot provide accurate utility plans, it is

recommended to use a company to determine the utility locations using one or more of the following technologies:

i. Electro-Magnetic (EM) device: This technology uses an electro-magnetic field in the subsurface to accurately locate metallic lines or non-metallic lines incorporating a metallic trace wire along their surface. The field is created either by direct contact to the pipe or tracewire, or by an induced current via radio waves.
ii. Sewer Sonde: For non-metallic lines where internal access is possible (such as clean-out ports in a sewer), a beacon or 'sonde' that emits a signal to the surface receiver as it is snaked through the pipe provides the same accuracy as the EM detector. If the internal condition of the pipe is desired, a camera can be deployed instead of a simple sonde.
iii. Ground Penetrating Radar (GPR): This technology involves radar waves reflecting to a surface receiver which provides a visual real-time map of the subsurface by which anomalies (such as pipes or tanks) may be detected. It has limitations in clay or wet soils and requires a skilled operator for interpretation. GPR should be considered for high risk utilities (e.g. PVC natural gas lines without trace wire) where line-of-sight project from site entry point to a kiosk or other building is uncertain.

- Utility markout on private property should include clearance for other types of underground structures such as underground storage tanks, septic systems, utility or access tunnels, and in-ground irrigation systems.

3. Limitations

- Markout notification time usually does not include holidays. Make sure holidays are considered and markout time is scheduled accordingly. Do not conduct excavation or drilling prior to the required wait time. Do not mark excavation locations using spray paint if it is raining or snowing enough so that the paint markings will be washed away. Consider using long stakes instead of paint if snow is predicted. Excavations within the tolerance zone should be performed with non-powered hand tools until the marked utility is exposed. The tolerance zone may be determined by the utilities, law or codes.
- When excavating close to an underground utility, it is good practice for the contractor/excavator to have a spotter assist and guide the machine operator.
- Take care not to damage the conduit or protective coating of a utility. If the excavator/contractor damages this, leave the damaged utility exposed and immediately call the utility owner.
- If contact to a gas utility occurs, notify police, fire, and emergency personnel, and evacuate employees and general public. No attempt should be made to tamper with or correct the damaged utility.

4. References

Connecticut
Name: Call-Before-You-Dig (CBYD)
Telephone: 1-800-922-4455
Website: www.cbyd.com
Wait time after notification: 2 business days (excluding holidays)
Expiration of markout: 30 days

Massachusetts, Maine, New Hampshire, Rhode Island and Vermont
Name: Dig Safe
Telephone: 1-888-DIG-SAFE or 811
Website: www.digsafe.com
Wait time after notification:
MA, ME, NH, and RI: 3 business days (excluding holidays)
VT: 2 business days (excluding holidays)
Expiration of markout: 30 days

New York State
Name: Dig Safely New York
Telephone: 1-800-962-7962
Website: www.digsafelynewyork.com
Wait time after notification: 2 business days (excluding holidays)
Expiration of markout: 30 days

New York City/Long Island
Name: New York City One Call Center
Telephone: 1-800-272-4480
Website: www.nycli1calldsi.com
Wait time after notification: 2 to 10 days (excluding holidays)
Expiration of markout: 30 days

New Jersey
Name: New Jersey One Call
Telephone: 1-800-272-1000
Website: www.nj1-call.org
Wait time after notification: 2 business days
Expiration of markout: 45 days

5. Attachment

Attachment A – Standard Utility Color Codes

6. Contact

Brian Conte
Anne Leifer

SOP PM-001

Attachment A – Standard Utility Color Codes

Color Code	Utility Description
Red	Electric
Yellow	Gas-Oil
Orange	Communications
Blue	Water
Green	Sewer
White	Proposed Excavation

STANDARD OPERATING PROCEDURE

QA-001 Equipment Decontamination

1. Objective

This SOP describes methods used to decontaminate reusable sampling equipment for projects that require collection of organic and inorganic analytical samples. The goal is to minimize cross-contamination between samples. This maximizes confidence that field samples will be representative of specific locations and conditions.

Refer to the work plan or project manager to determine if different decontamination methods are acceptable.

2. Execution

- All contractor-provided equipment (augers, rods, spoons, backhoe buckets) should be decontaminated by steam cleaning or pressure washing prior to coming on site. If there is doubt about cleanliness of drilling tools, they should be decontaminated before use at the site.
- Sampling equipment decontamination is a sequential procedure consisting of the following steps:

    - Alconox-solution wash (or equivalent non-phosphate detergent)
    - Potable water rinse
    - A ten percent reagent grade nitric acid wash should be used to strip potential inorganic contaminants from sampling devices.
    - Laboratory grade 100 percent methanol, should be used to strip potential organic contaminants from sampling devices.
    - Three distilled/deionized water rinses.

- Alconox solution is a mixture of approximately 1 cup of Alconox per 1 gallon of potable water. Alconox solution wash requires scrubbing the equipment with a brush soaked in Alconox solution to remove visible contamination or dirt from sampling devices.
- Split-spoon samplers must be decontaminated prior to collecting each sample. The procedure follows:

    - Overall wash and scrub in a bucket of Alconox solution
    - Potable water rinse.
    - 10% nitric rinse
    - 100% laboratory grade methanol rinse
    - Three distilled-water rinses.

    The same procedure is applied to all devices that may contact soil or groundwater slated for analytical samples - spoons and knifes used to inspect or sample soils; water level indicators; oil/water interface probes.

Equipment used for well development of multiple wells must be decontaminated between wells.

Pumps and tubing should be flushed using a minimum of one gallon of Alconox-solution followed by a gallon of potable water. Some projects may require methanol (in much lower quantities) and distilled water instead of or in addition to the Alconox-solution and potable water.

For pumps and tubing, a final rinse of the sampling equipment may be performed with the water being sampled.

Equipment blanks measure the effectiveness of the decontamination procedures. Blanks should be collected per guidance provided in QA-002, Field Quality Control Samples.

3. Limitations

- Do not store the deionized/distilled water in polyethylene bottles, use Nalgene, glass, or Teflon. Polyethylene may leach phthalates.
- Do not attempt to decontaminate string or rope - replace it.
- Due to eye and skin absorption hazards, safety glasses and gloves must be worn when handling decontamination solvents.
- Decontamination procedures may also require modification based on state or federal requirements.
- Steam cleaning or pressure washing with potable water is generally an acceptable decontamination method for drilling equipment (i.e., augers). Check with the work plan.
- Dedicated equipment need not be decontaminated beyond initial decontamination prior to field use.

4. References

Environmental Response Team (ERT), US EPA. Sampling Equipment Decontamination, SOP No. 2006, Revision 0.0. August 11, 1994.

US EPA Region 9. Sampling Equipment Decontamination, SOP No. 1230, Revision 1.September 1999.

5. Contacts

Brian Conte
Bill Simons

STANDARD OPERATING PROCEDURE

QA-002 Field Quality Control Samples

1. Objective

Field Quality Control (QC) samples are used to monitor the reproducibility and representativeness of field sampling. The QC samples are handled, transported, and analyzed in the same manner as the associated field samples. QC samples may include trip blanks, equipment blanks, and field duplicates.

2. Execution

2.1. Trip blanks

- Used to monitor possible sources of contamination from transport, storage, inadequate bottle cleaning, or laboratory methodologies.
- Sample containers filled at the laboratory with analyte-free water are transported to and from the site, and are not opened until time of analysis.
- Trip blanks are stored with the sample containers prior to and after field activities and remain with the collected samples until analyzed.
- Generally, one trip blank per volatiles analysis (e.g. volatile organic compounds) shipment.
- Consider submitting a trip blank when sample shipment is by Fed Ex or other large carrier, or laboratory courier.
- Trip blanks should be recorded in the field notebook and on the chain-of-custody that same as all other samples.

2.2. Equipment blanks

- Equipment blanks (also known as equipment rinsate blanks) are used to monitor possible sources of contamination associated with sample collection. Monitors on-site sampling environment, sampling equipment decontamination, sample container cleaning, the suitability of sample preservatives and analyte-free water, and sample transport and storage conditions
- Equipment blanks are collected by pouring laboratory supplied or distilled or deionized water over sampling tools that have been decontaminated per the work plan, into sample containers.
- Equipment blanks are stored with the associated field samples until submitted for analysis.
- Generally collected when site conditions indicate site related contamination is a concern. Check project-specific work plan and/or quality assurance project plan for required frequency.
- Prepare equipment blanks immediately after the equipment is cleaned in the field and before leaving the sampling site.
- Prepare equipment blanks by rinsing the decontaminated sampling equipment set with the appropriate type of analyte-free water and collecting the rinse water in appropriate sample containers.

- If a potable water rinse is the typical final step, collect the equipment blank with analyte-free water after the potable water rinse.
- Equipment blanks should be recorded in the field notebook and on the chain-of-custody that same as all other samples.

2.3. Field Duplicates

- Used to evaluate the precision and representativeness of the sampling procedures.
- Field duplicates are two samples collected from the same location using the same procedures. Both samples are submitted to the laboratory as individual samples with different sample identification.
- Field duplicates from groundwater sampling for all analyses except volatiles analysis are collected by alternating filling sample containers from the same sampling device. Field duplicates for volatiles analysis are filled sequentially.
- Soil or sediment field duplicates are collected by homogenizing the sample for all analyses except volatiles. The homogenized sample is then divided into two equal portions and placed in separate sample containers. Field duplicates for volatile analysis are collected at two adjacent sampling locations.
- Each sample is assigned different sample identifications.
- Field duplicates are generally collected at frequency of 1/20 samples. Check project-specific work plan and/or quality assurance project plan for required frequency.
- All field QC samples should be labeled in the field and submitted “blind” to the laboratory – as if they are separate, primary samples.
- Field duplicates should be recorded in the field notebook and on the chain-of-custody that same as all other samples.
-

2.4. Matrix-Spike samples (MS/MSD)

- Matrix spike and matrix spike duplicate samples (MS/MSDs) are environmental samples that are spiked in the laboratory or in the field with a known concentration of a target analyte(s) to verify percent recoveries.
- Matrix spike and matrix spike duplicate samples are primarily used to check sample matrix interferences. They can also be used to monitor error due to laboratory bias and poor precision. However, a data set of at least three or more results is necessary to statistically distinguish between laboratory performance and matrix interference.
- Generally, the laboratory is required to extract and analyze MS or MS / MSDs at a minimum frequency of 5% of samples being analyzed for the target analyte(s). If the project or client criteria require an MS or MS/MSD, collect sufficient volume in the appropriate containers, and designate the sample to be used as the MS or MS/MSD on the chain of custody.
- Calculate the percent recovery for all spiked analytes for both the MS and MSD. For MS/MSDs also calculate the relative percent difference (RPD). The

RPD for each spiked analyte is calculated using the amount detected not percent recovery. If your data will be subjected to validation, the % recovery and the RPD will generally be determined by the validator.

2.5. Typical QA/QC Frequency

- QA/QC frequency is determined by project, client or regulatory criteria and should be verified prior to sample collection. Generally, QA/QC samples are collected according to the frequency described below:

Duplicate Samples	One per sampling event, one per 10 samples collected, or one every two weeks, whichever comes first.
Equipment Blanks	For each equipment type that is not dedicated or disposable - one per sampling event, one per 20 samples collected, or one every two weeks, whichever comes first.
Trip Blanks	One per sample delivery group, or in each cooler containing VOC soil or aqueous samples, depending on project.
MS or MS / MSDs	One MS or MS/MSD per sampling event, one per 20 samples collected, or one every two weeks, whichever comes first.

3. Limitations

- Trip blanks must never be opened in the field.
- Trip blanks are usually for VOCs only because less volatile compounds are not likely to cross-contaminate other samples by simply being in close proximity.
- Laboratory-grade water must be used during the collection of equipment blanks.
- Field duplicates must have different sample identifications.

4. References

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (November 1986), U.S. Environmental Protection Agency Department of Solid Waste, Washington, D.C.

U.S. Environmental Protection Agency Office of Emergency and Remedial Response, 1990, Quality assurance/quality control guidance for removal activities: EPA/540/G-90/004, Sampling QA/QC Plan and Data Validation Procedures Interim Final, April, 1990.

5. Contact

Brian Conte
Pat King

STANDARD OPERATING PROCEDURE

SC-001 Environmental Sample Types and Sampling Strategies

1. Objective

Describe types of samples and strategic approaches to sample locations.

Refer to Attachment A for guidance on compatible sampling materials.

2. Sample Types

Grab Samples

A grab (or discrete) sample is a single aliquot (part of the sampled media) collected from a single location at a specific time.

Surface soil samples are typically “grab” samples. Volatile organic samples are always grab samples because the least amount of sample disturbance is necessary.

Composite Samples

Composite samples are non-discrete samples composed of more than one aliquot collected from different sampling locations and/or at different points in time. Analysis of composite samples produces an average value.

Composite samples are frequently collected to characterize waste soil that has been stockpiled for eventual disposal. Several grab samples are collected from the stockpile and are blended together into a single sample.

Screening Samples

Screening samples may be grab or composite in nature. However, they offer potential advantages such as rapid results and low cost. The trade-off is that they may only provide results within a range and/or they may have elevated detection limits. Screening samples are most often used to evaluate presence/absence and/or indications of the potential magnitude of impacts.

3. Sampling Strategies

Generally, there are three sampling strategies: random, systematic, and judgmental sampling.

- Random sampling involves collection of samples in a non-systematic fashion from the entire site or a specific portion of a site.
- Systematic sampling involves collection of samples based on a grid or a pattern which has been previously established.
- Judgmental sampling is the collection of all other samples. This sampling might be from areas most likely to be contaminated, areas most likely to be clean, or areas where information is lacking.

Often, a combination of these strategies is the best approach depending on the type of the suspected/known contamination, the uniformity and size of the site, the level/type of information desired, etc.

4. Attachments

Attachment A - General Guidelines for selecting equipment

5. Contacts

Jerry Zak
Ryan Hoffman

General Guidelines for selecting equipment on the basis of construction material and target analyte(s)

[✔, generally appropriate for use shown; Si, silica; Cr, chromium; Ni, nickel; Fe, iron; Mn, manganese; Mo, molybdenum; CFC, chlorofluorocarbon; B, boron]

Construction material for sampling equipment		Target analyte(s)	
Material	**Description**	**Inorganic**	**Organic**
		Plastics[1]	
Fluorocarbon ploymers[2] (other varies available for differing applications)	Chemically inert for most analytes	✔ (potential source of fluoride)	✔ (Sorption of some organics)
Polypropylene	Relatively inert for inorganic analytes	✔ (not appropriate for Hg)	**Do not use**
Polypropylene (linear)	Relatively inert for inorganic analytes	✔ (not appropriate for Hg)	**Do not use**
Polyvinyl chloride (PVC)	Relatively inert for inorganic analytes	✔ (not appropriate for Hg)	**Do not use**
Silicone	Very porous. Relatively inert for most inorganic analytes	✔ (potential source of Si)	**Do not use**
		Metals	
Stainless steel 316 (SS 316)	SS-316-metal having the greatest corrosion resistance. Comes in various grades. Used for submersible pump casing.	✔ (Potential source of Cr, Ni, Fe, and possible Mn and Mo) **Do not use** for surface water unless encasted in plastic.	✔ Do not use if corroded[3]
Stainless steel 304	Similar to SS-316, but less corrosion resistant	**Do not use**	✔ Do not use if corroded[3]
Other metals: brass, iron, copper, aluminum, galvanized and carbon steels	Refrigeration-grade copper or aluminum tubing are used routinely for collection of CFC samples	**Do not use**	✔ Routinely used for CFCs Do not use if corroded[3]
		Glass	
Glass, borosilicate (laboratory grade)	Relatively inert. Potential sorption of analytes	✔ **Do not use** for trace element analyses. Potential source of B and Si	✔

[1]Plastic used in connection with inorganic trace-element sampling should be uncolored or white. Tubing used for trace metal sampling should be cleaned by soaking in 5-10 percent HCl solution for 8-24 hours, rinsing with reagent water (metals free) and allowed to air dry in mercury-free environment. After drying, the tubing is doubled-bagged in clear polyethylene bags, serialized with a unique number, and stored until used.

[2] Fluorocarbon polymers include materials such as Teflon™, Kynar™, and Tefzel™ that are relatively inert for sampling inorganic or organic analytes. Only fluoropolymer should be used for samples that will analyzed for mercury because mercury vapors can diffuse in or out of other materials, resulting in either contaminated or biased results.

[3] Corroded/weathered surfaces are active sorption sites for organic compounds.

STANDARD OPERATING PROCEDURE

SC-002 Environmental Sample Handling

1. Objective

Describe appropriate environmental sample handling procedures.

The procedures include collection and transport of environmental samples to a laboratory for chemical analysis. Appropriate sample handling should ensure that samples are properly:

- labeled and documented;
- preserved;
- packaged; and
- transported

2. Execution

- Prior to mobilizing to the field, select a shipper or arrange for a courier for sample delivery to the laboratory. If using a shipper (i.e., FedEx or UPS) determine the time constraints for pickup requests, the location and hours of the nearest shipping office, and any size/weight restrictions.
- A waterproof or permanent ink pen should be used for all labels. The label should have an adhesive backing and be placed on the jar or bottle, not on the cap. In addition, clear packing tape can be placed over the sample label to secure it to the bottle as moisture from the samples can loosen the label adhesive.
- Record the following information on the label and in the field notebook (See SOPs FD-001 and FD-003):
    - Project number
    - Sample identification (i.e. MW-201 or SS-2)
    - Date and time (military time) of collection
    - Sampler's initials
    - Analysis methods
    - Preservative, if present
- Pre-preserved laboratory jars are preferable and should be used whenever practicable. If sample jars are not pre-preserved, add preservative as appropriate.
- At each sampling location, samples should be collected in order of volatility, most volatile first. Samples collected for volatile analysis should be placed in sample containers immediately upon retrieval of the sample.
- Aqueous samples for volatile analysis should be collected without air bubbles.
- The collection and preservation method of soil samples for volatile analysis may depend on project, client, or state regulatory requirements. Check with your Project Manager and/or SOPs SM-001 and SM-002 where appropriate.

- Care must be taken to avoid getting soils on the threads of sample jars, which can cause a faulty seal.
- If compositing samples in the field, specify the basis for composite (i.e. volume, weight, spoon recovery, etc.) and record in the field book the procedure for compositing the sample.
- Once samples have been collected and labeled, place samples in a cooler with sufficient bagged ice or freezer packs (blue ice) (if allowed) to chill samples to 4°C. If using ice, use double-bagged ice.
- Complete the chain-of-custody (COC) (SOP FD-003).
- If transporting the samples by way of a shipper:
    i. The sample cooler should have water drains securely sealed with duct tape, both on the inside and outside of the cooler.
    ii. Place a layer of packing material on the bottom of the cooler as a cushion.
    iii. Individually wrap each sample bottle with bubble packing or suitable packing material and place the wrapped bottles upright in the cooler with sufficient packing material between samples to avoid breakage.
    iv. Methanol preserved samples for volatiles analysis should be packed so they remain upright with the soil completely covered by the methanol during transport.
    v. Place a layer of packing material on top of the sample bottles.
    vi. Place bagged ice or freezer packs on top of the packing material. Fill the remaining space in the cooler with packing material to eliminate the possibility of vertical movement of samples.
    vii. Place the completed and signed chain-of-custody form in a sealable plastic bag and place on top of the packing material in the cooler, or tape it to the inside lid of the cooler.
    viii. Fill out the appropriate shipping or courier forms and attach to the top or handle of the cooler. If necessary, place the proper shipping labels on the cooler. Have the courier sign the COC form (or write pickup by FEDEX, UPS, etc. with date and time). Place a signed and dated custody seal on the cooler.
- All samples should be submitted to the laboratory as soon as possible. In many cases, same day shipping will be required by the client or the project manager. Be clear on this before beginning the field work.
- A copy of the waybills should be kept by the field supervisor to track shipments if necessary.

3. Limitations

- If samples are shipped on a Friday, call the laboratory ahead of time to confirm that personnel will be at the laboratory to receive and log-in the samples.
- During warm weather, make sure to use plenty of ice in the shipping container.

- Field personnel should be aware of analyses which have short hold times and schedule sampling events and shipping accordingly. Shipment of samples for analyses with short hold times must be arranged for in advance. Refer to the project work plan, quality assurance project plan, or state/federal regulations for holding time and preservative information. Contact the laboratory ahead of time when shipping samples with short hold time to ensure the lab is prepared for these analyses.
- For glassware containing preservatives (e.g., HCl, HNO_3), take care not to overfill the container, thus flushing the preservative out of the bottle.
- Never composite samples for VOCs in the field. Collect individual aliquots and direct the laboratory to perform compositing, if needed.
- Collection of aqueous samples should not be performed over the opening of a monitoring well. Preservatives from overfilling, a marker pen or other objects could fall into the well.
- If the recharge volume for a monitoring well is low, completely fill all volatile vials and then collect the minimum sample volume required for each remaining analysis.
- During subsurface soil sampling, if the recovery from the split-spoon sample is inadequate, if appropriate, resample the bottom of the borehole to obtain proper sample volume.
- Laboratories will homogenize and test the contents of the sample container, unless directed otherwise. Samples should not contain rocks, twigs, leaves, etc… unless these materials are of interest.

4. References

New Jersey Department of Environmental Protection, Field Sampling Procedures Manual, August 2005.

Connecticut Department of Environmental Protection, Guidance for Collecting and Preserving Soil and Sediment Samples for Laboratory

Preservation Techniques for Volatile Organic Compound (VOC) Soil Sample Analyses, WSC#99-415. Massachusetts Department of Environmental Protection.

5. Contacts

Jennifer Belonsoff
Leslie Lombardo

STANDARD OPERATING PROCEDURE

SC-003 Investigation Derived Waste

1. Objective

Describe characterization and management of Investigation Derived Waste (IDW) resulting from site investigation activities.

IDW is solid and/or aqueous waste generated during environmental site investigations.

2. Execution

- Determine the suspected contamination type and impacted media based on previous investigations, available analytical data, and/or site history.
- Consider the following when selecting IDW management option(s):
    - Anticipated volume of IDW to be generated during on-site activities
    - Potential contaminants and their concentrations
    - Proximity to population centers and the potential for unauthorized site access
    - Potential exposures to workers
    - Potential for environmental impacts
    - Community concerns
    - Potential storage areas
    - Regulatory constraints
    - Potential on-site treatment options
    - Duration of storage
    - Client concerns or requirements
- Review IDW Management Options summarized in Attachment A for each media suspected of contamination.
- Select IDW Management Option(s) prior to the commencement of field activities that will generate waste materials.
- Include the selected IDW Management Option(s) in the Field Plan or other project documents.

Considerations and guidelines for IDW management for specific field tasks are provided below.

2.1. Test Pit Excavation

- Segregate contaminated soil from uncontaminated soil using visual and/or field screening methods.
- Use appropriate barrier (such as two layers of 6-ml plastic sheeting) for temporary stockpiling of contaminated soil adjacent to test pit.

- Backfill test pits with uncontaminated soil, unless otherwise directed by project manager.
- If directed by the Project Manager to return contaminated soil to the test pit, backfill soil in the same order as the soil was excavated from the test pit.

2.2. Boring/Monitoring Well Installation

- For auger borings, segregate contaminated soil (determined by visual and/or field screening methods) from uncontaminated soil during drilling. Segregate residual contaminated soil from split-spoon sampling.
- Auger cuttings or sediment generated by drive and wash may be spread around the ground surface at the boring location if it is acceptable to the client and the governing regulatory agency. If not, IDW may be placed in an appropriate area or container pending characterization and appropriate disposal. (A useful rule of thumb is to assume generation of one 55-gallon drum of cuttings for each 20 feet drilled with 7-¼-inch-I.D. augers).
- Segregate contaminated drilling fluid from uncontaminated fluid for rotary wash borings.
- Drilling fluid management options include pouring the drilling fluid on the ground near the boring location, if acceptable to the client and governing regulatory agency, or containerizing the fluid in drums or tanks.

2.3. Well Development/Sampling

Contaminated groundwater removed from wells by pumping or bailing for the purpose of well development and sampling may be poured on the ground near the well, if it is acceptable to the client and the governing regulatory agency. Otherwise, it should be containerized in drums or tanks.

2.4. Decontamination Fluids

Decontamination fluids may be poured on the ground in the vicinity of the well if approved by the project manager. Alternatively, the fluids may be containerized in drums or tanks.

2.5. Disposable Personal Protective Equipment

Disposable personal protective equipment (PPE) should be managed like any other IDW. However, with the clients' and project manager's approval, it may be removed from the site and disposed of as ordinary rubbish if it has not come into contact with contaminated materials.

3. Limitations

- The simplest IDW management option is to return the IDW to its source location.
- However, the selected IDW management options must meet state/federal regulations and have the client's approval. Consult with state/federal policies for IDW-related matters.

- The client is responsible for the disposal of IDW, should disposal be necessary.

4. References

Guide to Management of Investigation - Derived Wastes (April 1992), United States Environmental Protection Agency, Publication 9345.3-03FS.

Standard References for Monitoring Wells, Massachusetts Department of Environmental Protection, Publication No. WSC-310-91.

5. Attachments

Attachment A - Summary of Investigation Derived Waste Management Options
Attachment B - CTDEP Waste Guidance

6. Contacts

David Terry
Leslie Lombardo

Attachment A: - SUMMARY OF IDW MANAGEMENT OPTIONS
GEI Consultants, Inc. Standard Operating Procedures
Management of Investigation - Derived Waste

Type of IDW	Generation Processes	Management Options	Remarks
Soil	Boring/monitoring well installation Test pit excavation Soil sampling	Return to source location immediately after generation	Acceptable, if authorized by the client, the governing regulatory agency, and the project manager.
		Spread around boring, test pit, or original source location	Acceptable, if authorized by the client, the governing regulatory agency, and the project manager.
		Containerize and temporarily store on site	Can temporarily store in stockpiles or covered containers (i.e. drums, roll-off containers). Stockpiles must be underlain by plastic sheeting and covered with plastic sheeting. Plastic sheeting must be secure. Storage consistent with state/federal regulations.
		Send to off-site, treatment or disposal facility within appropriate timeframes	Requires proper shipping documents (i.e. manifest, Bill of Lading, etc.), analytical characterization
		Store for future treatment and/or disposal.	Storage consistent with state/federal regulations. If a RCRA hazardous waste, must meet RCRA Container/Waste Pile/Tank requirements (see notes)
		Store temporarily awaiting laboratory analysis.	Storage consistent with state/federal regulations. Can temporarily store in stockpiles or covered containers (i.e. drums, roll-off containers). Stockpiles must be underlain by plastic sheeting and covered with plastic sheeting. Plastic sheeting must be secure.
Sediment/Sludge	Sludge pit sampling Sediment sampling	Return to source immediately after generation	Acceptable, if authorized by the client, the governing regulatory agency, and the project manager.
		Store temporarily on site.	Storage consistent with state/federal regulations.
		Send to off-site facility within 90 days	Requires manifests, analytical characterization
		Store for future treatment and/or disposal.	Storage consistent with state/federal regulations. If a RCRA hazardous waste, must meet RCRA Container/Waste Pile/Tank requirements (see notes)

Attachment A: - SUMMARY OF IDW MANAGEMENT OPTIONS
GEI Consultants, Inc. Standard Operating Procedures
Management of Investigation - Derived Waste

Type of IDW	Generation Processes	Management Options	Remarks
Aqueous liquids (groundwater, surface water, drilling fluids, other wastewater)	Well installation/development Well purging during sampling Ground water discharge - pump tests Surface water sampling	Pour onto ground close to well	Non-hazardous liquids only. Should not exhibit a sheen or separate phase product. Do not discharge to the ground up-gradient of the source location. Ensure that it is permissible by local, state, and Federal regulations Is acceptable to the client, the governing regulatory agency, and the project manager.
		Store temporarily on site	If a RCRA hazardous waste, must meet RCRA Container/Waste Pile/Tank requirements (see notes)
		Send to off-site commercial treatment unit within appropriate timeframes	Refer to State regulations for appropriate timeframe. Requires appropriate shipping documents (i.e., manifest, Bill of Lading), analytical characterization
		Send to POTW	Obtain appropriate discharge permit(s)
		Store for future treatment and/or disposal.	Storage consistent with state/federal regulations. Consistent with final remedial action
		Discharge to surface water	OK if it complies with state and federal regulations. Obtain appropriate discharge permit(s).
Decontamination fluids	Decontamination of PPE and equipment	Store temporarily on site	If a RCRA hazardous waste, must meet RCRA Container/Waste Pile/Tank requirements (see notes)
		Send to off-site facility within appropriate timeframes	Requires manifests, analytical characterization
		Store for future treatment and/or disposal. Storage consistent with state/federal regulations.	Consistent with final remedial action
Disposable PPE	Sampling, drilling, and test pit excavation observation, other on-site activities	Store temporarily on site	Dispose of appropriately after characterization
		Place in on-site industrial dumpster	Project-specific determination required – must be acceptable to client and project manager
		Send to off-site facility within 90 days	Project-specific determination required
		Store for future treatment and disposal.	Storage consistent with state/federal regulations. Project-specific determination required

Notes:

1) PPE - personal protective equipment
2) POTW - publicly owned treatment works
3) Generation processes listed here are provided as examples.
   IDW may also be generated as a result of other site activities.
4) RCRA Container/Waste Pile/Tank requirements:
   Containers; 40 CFR 264 Subpart I and 265 Subpart I
   Waste Piles; 40 CFR 264 Subpart L and 265 Subpart L
   Tanks; 40 CFR 264 Subpart J and 265 Subpart J

STANDARD OPERATING PROCEDURE

SC-004 Headspace VOC Screening

1. Objective

Describe methods to obtain site-specific measurement of the total volatile organic compound (VOC) concentrations present in the headspace of a jar containing soil.

This information can be used for several purposes:

- Segregate soil based on degree of contamination.
- Identify samples for quantitative analysis of VOCs.
- Evaluate the presence or absence of VOCs in soil.

2. Execution

- A photoionization detector (PID) or flame ionization detector (FID) instrument is used to measure VOCs in jar headspace (JHS) screening.
- Select the appropriate instrument, lamp, and calibration gas for the site-specific contaminants. Calibrate the instrument in accordance with the manufacturer's instructions before JHS screening begins. Record the type of calibration gas, detector, lamp, and results of calibration in the field notebook.
- Note the highest VOC concentration that the instrument measures in air in the work area before performing JHS screening. Record this as the initial background concentration.
- Half-fill a clean, glass jar with the soil. Quickly cover the open top with one or two sheets of clean, aluminum foil and screw on the cap to tightly seal the jar. Label the jar with the sample location and sample depth.
- Allow headspace development for at least 10 minutes at an ambient temperature of 50°F or greater. Vigorously shake the jar for 15 seconds at the beginning and end of the headspace development period. When ambient temperatures are below 50°F, place the jar in a heated vehicle or building during the headspace development period.
- After headspace development, remove the screw cap to expose the foil seal. Quickly puncture the foil seal with the instrument's sampling probe and insert it to a point at about one-half of the headspace depth.
- Record the highest VOC concentration that the instrument displays as the JHS concentration. The highest concentration should occur between 2 and 5 seconds after probe insertion.

3. Limitations

- The instruments may work poorly in the rain and in freezing temperatures. Under such conditions, operate the instrument in a heated vehicle or building if possible.

- Prevent water and soil particles from entering the tip of the instrument probe. Use a filter on the instrument's probe.
- Measure background VOC conditions and perform JHS screening away from non-site-related VOC sources, such as vehicle and heavy equipment exhaust.
- The VOC concentration on the instrument's display may vary when the air contains high VOC concentrations or high moisture.
- JHS screening is a guide that helps the screener to segregate soils into broadly defined categories. JHS screening results may differ by orders of magnitude from laboratory testing results.
- Note that states may have specific procedures for field monitoring. In Massachusetts, the Massachusetts Department of Environmental Protection (DEP) requires that screening of gasoline-contaminated soil be performed in accordance with Attachment II of the DEP's policy #WSC-94-400 Interim Remediation Waste Management Policy for Petroleum Contaminated Soils. Consult this procedure or any relevant guidance documents for assistance.

4. References

Interim Remediation Waste Management Policy for Petroleum Contaminated Soils. (April 1994), Massachusetts Department of Environmental Protection, Policy #WSC-94-400.

5. Contacts

Lynn Willey
Leslie Lombardo

STANDARD OPERATING PROCEDURE

SG-001 General Guidance on Soil Vapor Intrusion Evaluations

1. Objective

The goal of a soil vapor intrusion evaluation is to assess whether complete exposure pathways of soil vapor to indoor air exist. A complete exposure pathway exists if vapors from constituents are migrating through various pathways into residential or commercial buildings at concentrations that may result in an unacceptable human health risk. If a complete exposure pathway does not exist, then further assessment of soil vapor intrusion is not required.

Depending on the status of investigation performed at the site it may be appropriate to approach an evaluation of soil vapor intrusion at different tiers. If little work has been performed relative to the potential for contaminants to affect soil vapor near a structure, then a screening level assessment is an appropriate first step. However, if a plume is well delineated and the potential for groundwater impacts, or nearby source material, to affect soil vapor near a potential receptor structure is well understood, then it may be more appropriate to directly develop and implement a soil vapor and/or indoor air sampling plan. To accommodate the potential varied states of knowledge when a vapor intrusion evaluation is required, a flexible approach is needed that incorporates the following elements.

- SOP SG-002 Soil Vapor Sample Collection
- SOP SG-003 Sub-Slab Soil Vapor Collection
- Indoor Air Sampling
- SOP SG-004 Ambient Air Sample Collection

Soil vapor intrusion evaluations should be approached on a site-specific basis and depending on the site-specific setting and proximity to impacted groundwater or source material, it may be appropriate to proceed in a hierarchical fashion through each tier of evaluation or a variety of tiers may be combined and implemented simultaneously. The SOPs presented in this SOP address each of these sampling procedures.

2. Execution

2.1. Implementation Triggers

Soil vapor intrusion evaluations may be implemented at various times based on event triggers throughout the Site Characterization (SC), Remedial Investigation (RI), and site remedial action plan. The following event triggers would require the implementation of this soil vapor intrusion investigation.

- Identification of a potential complete exposure pathway
- Private property owner request for sampling

- State or Federal administrative order

2.2. Factors Affecting Soil Vapor Intrusion

Prior to conducting a soil vapor intrusion assessment at a private property, an analysis of the factors contributing to the migration of soil vapor to indoor air should be conducted. The completion of this analysis should take into account the two types of factors: environmental and building factors.

2.2.1. Environmental Factors

Environmental factors include site specific conditions in the subsurface and above the ground surface that may affect the rate and direction at which soil vapor may migrate.

The soil and groundwater conditions between the contamination and the residential/commercial building should be evaluated and recorded in any soil vapor intrusion investigation. If the SC/RI has been completed, then the data are available for this review. If the SC/RI has not been completed, then at a minimum the nature and extent of impacted soil and/or groundwater between the site and the residential/commercial building should be defined.

After compiling the necessary site-specific data, that information should be reviewed to determine groundwater conditions at the site. The potential for man-made or natural preferential pathways for vapor migration in the vadose zone and/or for groundwater migration in the saturated zone should also be determined at this time.

- The depth to groundwater below the residential or commercial building will be determined. For example, in cases where groundwater intersects the foundation there is no vadose zone to collect a sub-slab sample. In cases where the groundwater is close to the foundation, there is a risk of causing/exacerbating groundwater intrusion through the foundation during periods of high groundwater.

Additional Site Observations

- Direction of groundwater flow from the contaminant source to the residential or commercial building;
- The location, depth, extent, and concentration of potential constituents in unsaturated soil and groundwater on the property; and,
- Presence of an overlying water bearing zone that does not have impacts beneath the residential or commercial building. An un-impacted shallow water zone will significantly retard or completely prohibit the potential for deeper impacted groundwater to affect soil vapor.
- Potential “smear zones” (residual non-aqueous phase liquid (NAPL) present at depths over which the water table fluctuates) should also be identified as they may also affect the rate of soil vapor migration.
- Location, depth, extent of NAPL, if present.

Soils which are highly organic, wet, and/or of low permeability should be identified. If these soils are present beneath a structure and above impacted groundwater or soil, they may effectively shield the building from potential vapor intrusion. Conversely, dry and porous soils underlying a building may provide a less inhibited soil vapor intrusion pathway. The limits of backfill surrounding residential or commercial building should be also noted.

2.2.2. Building Factors

Building Factors include the physical characteristics, such as structure, floor layout, air flow, and physical conditions. These conditions will be documented during the evaluation. The New York State Department of Health (NYSDOH) Center for Environmental Health's Indoor Air Quality Questionnaire and Building Inventory form is presented in Attachment A. At a minimum, the following information should be recorded.

- Building foundation construction characteristics (basement, footers, crawl spaces, etc), including potential preferential vapor intrusion pathways such as foundations cracks and utility penetrations.
- Basement wall materials (hollow block, stone, or poured concrete, etc.)
- Presence of an attached garage.
- Recent renovations to the building such as new paint or new carpet.
- Mechanical heating/cooling equipment that may affect air flow.
- Use and storage of petroleum products such as home heating oil storage tanks, underground storage tanks (USTs), or kerosene heaters.
- Recent use of petroleum-based finish or other products containing volatile organic compounds (VOCs).
- Areas of pavement on the property should also be identified in the event sub slab vapor sampling is not feasible or appropriate due to a high groundwater table. Paved areas could serve as surrogate locations in lieu of sub slab soil vapor sampling if high water table conditions exist.

The construction materials and integrity of the floor of the structure closest to the potential point of entry for soil vapor (basement level or first floor for slab-on-grade constructions) should be identified. In addition to the foundation type and integrity, this survey should note any preferential pathways (utility lines/pipes, sumps, etc.) that may exist within the bottom-most level of the structure.

The operation and presence of heating systems, including fireplaces and clothes dryers, may create a pressure differential between the structure and the outside environment, causing an increase of migration of soil vapor into the building. The NYSDOH guidance document suggests limiting indoor air sampling to the heating season (with the exception of immediate inhalation hazard situations), which is roughly defined as November 15th to March 31st. However, sampling may be completed at any time during the year for any sampling completed in response to a request by a community member. In situations where non-heating season sampling

has taken place, consideration should be given to re-sampling the property within the heating season. The operation of HVAC systems should be noted on the building inventory form (Attachment A).

During the initial building assessment and visit, and again when sub-slab soil vapor and/or indoor air sampling are performed, differential pressure measurements between indoor air, ambient air, and soil vapor should be collected and recorded to document the potential effect building conditions have on soil vapor migration.

2.2.3. Property Visit

A property visit will be conducted prior to sampling. During the site visit, technical representatives will complete site visit observations, inventories and occupant questionnaire forms (Appendix A). During the course of the interview, observations will be made to identify any potential areas or issues of concern or the presence of any odors, and if sampling appears necessary, identify potential sampling points and general building characteristics. The questionnaire is also used to identify potential sources and activities that may interfere with sampling results. The questionnaire will specifically address the activities of the occupant's (e.g., smoking, work place activities) that may contribute to indoor air concentrations of volatile chemicals.

The responses to the questionnaire will be evaluated and a determination will be made as to whether additional investigation is required.

2.2.4. Chemical Inventory

The chemical inventory complements the identification of the building factors affecting soil vapor intrusion. The chemical inventory will identify the occurrence and use of chemicals and products throughout the building. These products can be used to develop an indoor environmental profile. A separate inventory should be prepared for each room on the floor being tested as well as any other indoor areas physically connected to the areas being tested. Inventories will include product names, chemical ingredients, or both. If possible, photographs of the products should be taken of the location and condition of the inventoried products and the photographic records should be indexed with the inventory records. The products inventory can also be used to document odors and if possible portable vapor monitoring equipment measurements should be taken and recorded. A product inventory will be repeated prior to each round of testing at the building. If available, the volatile ingredients should be recorded for each product. If the ingredients are not listed on the label, record the manufacturer's name and address or phone number if available. The product inventory form is presented in Attachment A.

2.2.5. Water Table Conditions and Vapor Intrusion Assessment Approach

Sub-slab soil vapor sampling is intended to evaluate the potential for vapor intrusion. However, there are circumstances where collection of sub-slab soil vapor samples may not be feasible if the water table is near, at, or above the elevation of a buildings foundation slab. An evaluation of the water table elevation relative to the

building slab should be made before attempting to install a sub-slab vapor sampling point.

If the water table is found to be sufficiently below the building slab and sub-slab vapor sampling can be performed, then the following Low Water Table Scenario should be followed.

2.2.5.1. Low Water Table Scenario

If the water table elevation is lower than the basement slab, then the following samples should be collected.

- Sub-slab soil vapor samples
- Indoor air samples from basement level
- Indoor air samples from main living space (First floor)
- Outdoor ambient air sample

If the water table is deemed to be at too high of an elevation to allow sub-slab vapor sampling, then alternate means of evaluating the potential for vapor intrusion must be employed. If a building has a groundwater sump, the sump should be evaluated to determine if there is water present in the sump and if that water is representative of groundwater or if the water is stagnant. If water in the sump represents groundwater, then a sample from the sump should be collected. The High Water Scenario below summarizes the methods to evaluate potential vapor intrusion if sub-slab vapor sampling cannot be conducted due to high groundwater conditions.

2.2.5.2. High Water Table Scenario

If the water table elevation is higher than the basement slab, then the following tasks should be performed.

- Determine if a sump pump is present and actively pumping water.
- If sump is actively pumping, collect a sample of groundwater from the sump.
- Collect an indoor air sample from basement level.
- Collect an indoor air sample from main living space (first floor).
- Identify exterior soil vapor sample location near foundation (outside of foundation backfill) and preferably beneath a surrogate vapor cap (e.g. paved driveway, patio).
- Collect soil vapor samples from exterior soil vapor location
- Collect an outdoor ambient air sample.

3. References

USEPA modified Method TO-15 and helium via ASTM D-1945.

Section 2.7.1 of the New York State Department of Health (NYSDOH) Final Guidance for Evaluating Soil Vapor Intrusion in the State of New York*, dated October 2006.*

4. Attachments

Attachment A - NYSDOH Center for Environmental Health's Indoor Air Quality Questionnaire and Building Inventory Form

5. Contact

Chris Berotti

ATTACHMENT A

Off-Site Property Sampling Documentation Form

Property Location/Address: _____

Property: _____

Sampling Date: _____

Property Location/Address: _____
Property: _____
Sampling Date: _____

Preparer's Name: _____ Date/Time Prepared: _____

Preparer's Affiliation: _____ Phone No.: _____

Purpose of Investigation:_____

1. OCCUPANT Interviewed: Yes □ No □

Last Name: _____ First Name: _____

Address: _____

County: _____

Home Phone: _____ Office Phone: _____

Number of Occupants/persons at this location _____ Age of Occupants_____

2. OWNER OR LANDLORD (Check if same as occupant __) Interviewed: Yes □ No □

Last Name: _____ First Name: _____

Address: _____

County: _____

Home Phone: _____ Office Phone: _____

3. CONTACT NAME (Check if same as Occupant ____, Owner ____)

Last Name: _____ First Name: _____

Address: _____

County: _____

Home Phone: _____ Office Phone: _____

4. PROPERTY LOCATION: _____

Relative to Site:

Direction _____ Direction to Nearest Cross Street: _____

Distance _____ Distance to Nearest Cross Street: _____

Surrounding Land Use:

North: _____ East: _____

South: _____ West: _____

Property Location/Address: _____
Property: _____
Sampling Date: _____

5. PROPERTY BOUNDARIES

Delineate the boundaries of the property (on a separate project map, outline property location, private well location, septic/leachfield location, groundwater flow, compass direction, windrose.)

6. BUILDING CONSTRUCTION

Type of Building (Circle appropriate response)

Residential	School	Commercial/Multi-use
Industrial	Church	Other: _____

If the property is residential, type? (Circle appropriate response)

Ranch	2-Family	3-Family
Raised Ranch	Split Level	Colonial
Cape Cod	Contemporary	Mobile Home
Duplex	Apartment House	Townhouses/Condos
Modular	Log Home	Other:_____

If multiple units, how many? _____

If the property is commercial, type?

Business Type(s) _____

Does it include residences (i.e., multi-use)? Yes □ No □

If yes, how many? _____

Other characteristics:

Number of floors_____ Building age_____

Is the building insulated? Yes □ No □ How air tight? Tight / Average / Not Tight

Construction Material _____

7. BASEMENT AND CONSTRUCTION CHARACTERISTICS

Does the building have a basement and/or crawl space, or is it slab-on-grade construction?

_____

Describe the construction of the basement/crawl space (Circle all that apply)

a. Above grade construction:	wood frame	concrete	stone	brick
b. Basement type:	full	crawlspace	slab	other _____

Property Location/Address: _____
Property: _____
Sampling Date: _____

c. Basement floor:	concrete	dirt	stone	other _____
d. Basement floor surface:	uncovered	covered	covered with _____	
e. Concrete floor:	unsealed	sealed	sealed with _____	
	unpainted	painted	painted with _____	
f. Foundation walls:	poured	block	stone	other _____
g. Foundation walls:	unsealed	sealed	sealed with _____	
h. The basement is:	wet	damp	dry	moldy
i. The basement is:	finished	unfinished	partially finished	

Does your basement have a sump? Yes □ No □

Is, is there water in the sump? Yes □ No □

Describe sump conditions:_____

_____

Have you observed standing water in your basement? Yes □ No □

If so, what is the frequency of this observation? _____During rain events? □

Have you observed sheen atop the standing water? Yes □ No □

Basement/Lowest level depth below grade: _____(feet)

Are there any cracks in the floor of your basement? Yes □ No □

Description: _____

_____

Identify potential soil vapor entry points and approximate size (e.g., cracks, utility ports, drains)

Description: _____

_____

What activities occur in the finished basement?

Description: _____

_____

Approximately how many hours per day (or week) do you spend in your basement? _____

8. HEATING, VENTING AND AIR CONDITIONING

Type of heating system(s) used in building: (Circle all that apply – note primary)

Property Location/Address: _____
Property: _____
Sampling Date: _____

Hot Air Circulation	Hot Water Baseboard	Steam Radiation
Electric Baseboard	Heat Pump	Wood Stove
Space Heaters	Radiant Floor	Outdoor wood boiler

Unvented Kerosene Heater Other _____

The primary type of fuel used is:

Fuel Oil	Natural Gas	Electric
Kerosene	Propane	Solar
Wood	Coal	Other? _____

Time of use of each type of heating? _____

Domestic hot water tank fueled by: _____

Boiler/furnace located in: Basement Outdoors Main Floor Other_____

Air conditioning: Central Air Window units Open Windows None

Are there air distribution ducts present? Yes □ No □

Describe the supply and cold air return ductwork, and its condition where visible, including whether there is a cold air return and the tightness of duct joints. Indicate the locations on the floor plan diagram.

_____

_____

_____

_____

Type of insulation (e.g. blown, fiber, etc.)? _____

Does building have energy efficient windows (e.g. double paned) Yes □ No □

Was weather-stripping recently added/upgraded? Yes □ No □

Particleboard used in construction? Yes □ No □

9. OCCUPANCY

Property Location/Address: _____
Property: _____
Sampling Date: _____

Level General Use of Each Floor (e.g., family room, bedroom, laundry, workshop, storage)

Basement _____

1st Floor _____

2nd Floor _____

3rd Floor _____

4th Floor _____

10. BULK PETROLEUM STORAGE

Aboveground storage tank on the property Yes ☐ No ☐

If yes, how old is tank? _____ Condition? _____

Last inspected? _____ Location: _____

Describe conduits to building (type, location, and entry portal condition): _____

_____

Underground storage tank on the property. Yes ☐ No ☐

If yes, how old is tank? _____ Condition? _____

Last inspected? _____ Location: _____

Describe conduits to building (type, location, and entry portal condition): _____

_____

11. WATER AND SEWAGE

Water Supply:

Public Water Drilled Well Driven Well Dug Well Other _____

Is there use of groundwater water for irrigation purposes? Yes ☐ No ☐

Sewage Disposal:

Public Sewer Septic Tank Leach Field Dry Well Other _____

12. FACTORS THAT MAY INFLUENCE INDOOR AIR QUALITY

a. Is there an attached garage? Yes ☐ No ☐

If not, is there a separate garage or carport? Yes ☐ No ☐

b. Does the garage have a separate heating unit? Yes ☐ No ☐ NA ☐

Property Location/Address: _____
Property: _____
Sampling Date: _____

c. Are petroleum-powered machines or vehicles stored in the garage (e.g., lawnmower, ATV, car)
Yes □ No □ NA □ Please specify_____

Is gasoline stored in the garage? Yes □ No □

Quantity? _____

d. Has the building ever had a fire? Yes □ No □

When?_____

e. Is a kerosene or unvented gas space heater present? Yes □ No □

Where? _____

f. Is there a workshop or hobby/craft area? Yes □ No □

Where & Type? _____

g. Is there smoking in the building? Yes □ No □

How frequently? _____

h. Have cleaning products been used recently? Yes □ No □

When & Type? _____

i. Have cosmetic products been used recently? Yes □ No □

When & Type? _____

j. Has painting/staining been done in the last 6 months? Yes □ No □

Where & When? _____

Is house paint stored inside? Yes □ No □

Where? _____

k. Is there new carpet, drapes or other textiles? Yes □ No □

Where & When? _____

l. Have air fresheners been used recently? Yes □ No □

When & Type? _____

m. Is there a kitchen exhaust fan? Yes □ No □

If yes, where vented?_____

n. Is there a bathroom exhaust fan? Yes □ No □

If yes, where vented?_____

o. Is there a clothes dryer? Yes □ No □

If yes, is it vented outside? Yes □ No □

p. Has there been a pesticide/chemical fertilizer application? Yes □ No □

Property Location/Address: _____
Property: _____
Sampling Date: _____

When & Type?_____

Conducted by Owner or Private Yard Service _____

Is yard waste/trash burned on-site? Yes □ No □

Do any of the building occupants use solvents at work? Yes □ No □

(e.g., chemical manufacturing or laboratory, auto mechanic or auto body shop, painting, fuel oil delivery, boiler mechanic, pesticide application, cosmetologist

If yes, what types of solvents are used? _____

If yes, are their clothes washed at work? Yes □ No □

Do any of the building occupants regularly use or work at a dry-cleaning service? (Circle appropriate response)

Yes, Use dry-cleaning regularly (weekly) No

Use dry-cleaning infrequently (monthly or less) Unknown

Yes, work at a dry-cleaning service

Is there a radon mitigation system for the building/structure? Yes □ No □

Date of Installation: _____

Is the system active or passive? Active □ Passive □

Are there any recent/past improvements to building? Yes □ No □

Interior painting? _____

Any landscaping improvements that involved bringing fill on site? Yes □ No □

Other _____

Approximately when (how long ago) did these improvements occur? _____

Does anyone living here engage in any of the following activities or hobbies?

a. Art projects (e.g. oil painting, ceramics, pottery, stained glass, metal sculpture)

Yes □ No □

Name: _____ Age: _____ Sex: _____

Name: _____ Age: _____ Sex: _____

Property Location/Address: _____
Property: _____
Sampling Date: _____

b. Furniture refinishing Yes □ No □

Name: _____ Age: _____ Sex: _____

Name: _____ Age: _____ Sex: _____

c. Model building(e.g. planes,boats,cars) Yes □ No □

Name: _____ Age: _____ Sex: _____

Name: _____ Age: _____ Sex: _____

d. Gardening Yes □ No □

Name: _____ Age: _____ Sex: _____

Name: _____ Age: _____ Sex: _____

e. Automotive work Yes □ No □

Name: _____ Age: _____ Sex: _____

Name: _____ Age: _____ Sex: _____

f. Ammunition reloading Yes □ No □

Name: _____ Age: _____ Sex: _____

Name: _____ Age: _____ Sex: _____

Is there a wood burning stove? Yes □ No □

If so, how frequently is it used?

_____

_____

Is there a barbeque grill? Yes □ No □

If so, how frequently is it used? What is the type of fuel?

_____

_____

Has the building ever had fumigation? Yes □ No □

Property Location/Address: _____
Property: _____
Sampling Date: _____

If so, when and how frequently? Type?

_____

_____

13. ODOR SUMMARY

Have the occupants observed any unusual odors? _____

_____

_____

_____

_____

History of odor observation – date of onset, duration, severity, etc.

_____

_____

_____

_____

_____

_____

_____

14. PRODUCT INVENTORY

Record the specific products found in building that have the potential to affect indoor air quality on the attached product inventory form.

15. INDOOR SKETCH

Draw a plan view sketch (on grid paper) of the basement, first floor, and any other floor where sampling was conducted in the building as well as any outdoor sample locations. Indicate air sampling locations, possible indoor air pollution sources and PID meter readings. If the building does not have a basement, please note.

Property Location/Address: _____
Property: _____
Sampling Date: _____

Product Inventory
Off-Site Property Sampling Documentation
Soil Vapor Intrusion Investigation

Property Address:	**Performed by:**
Date of Inventory:	**Field Instrument Make & Model:**

Location	**Product Description**	**Size (units)**	**Condition ***	**Chemical Ingredients**	**Field Instrument Reading (units)**	**Photo ** Y/N**

Notes

* Describe the condition of the product containers as Unopened (UO), Used (U), or Deteriorated (D)

** Photographs of the front and back of product containers can replace the handwritten list of chemical ingredients. However, the photographs must be of good quality and ingredient labels must be legible.

STANDARD OPERATING PROCEDURE

SG-002 Soil Vapor Sample Collection

1. Objective

This procedure outlines the general steps to collect soil vapor samples. The site-specific Sampling and Analysis Work Plan should be consulted for proposed sample locations, sample depths, and sampling duration.

2. Execution

Permanent and temporary soil vapor probes should be installed using the procedures outlined below. All soil vapor probes should be installed using a direct-push drill rig (e.g., Geoprobe® or similar), hand auger, or manually using a slide hammer.

2.1. Document Field Conditions

Document pertinent field conditions prior to installation of any probe points.

- Record weather information (precipitation, temperature, barometric pressure, relative humidity, wind speed, and wind direction) at the beginning of the sampling event. Record substantial changes to these conditions that may occur during the course of sampling. The information may be measured with on-site equipment or obtained from a reliable source of local measurements (e.g., a local airport). Data should be obtained for the past 24 to 48 hours.
- If sampling near a commercial or industrial building, uses of volatile chemicals during normal operations of the facility should be identified.
- Outdoor plot sketches should be drawn that include the site, area streets, neighboring commercial or industrial facilities (with estimated distance to the site), outdoor air sampling locations (if applicable), and compass orientation (North);
- Any pertinent observations should be recorded, such as odors and readings from field instrumentation.

2.2. Soil Vapor Point Installation Specifications

Each soil vapor point should be constructed as follows:

- Six-inch stainless steel Geoprobe® AT86 series Permanent Implants (soil vapor screens) or equivalent and threaded to an (expendable) stainless steel anchor point.
- The implants should be fitted with inert Teflon or stainless steel tubing of laboratory or food grade quality.
- The annular space surrounding the vapor screen interval and a minimum of 6-inches above the top of the screen should be filled with a porous backfill

material (e.g., glass beads or coarse silica sand) to create a sampling zone 1 foot in length.

For temporary points, a hydrated bentonite surface seal should be created at the surface to minimize infiltration. For permanent points, the additional measures described below should be included.

- The soil vapor points should be sealed above the sampling zone with a bentonite slurry for a minimum distance of 3 feet (or to grade, whichever is smaller) to prevent ambient air infiltration.
- If needed, the remainder of the borehole should be backfilled with clean material.
- A protective casing should be set around the top of the point tubing and grouted in place to the top of the bentonite to minimize infiltration of water or ambient air, as well as to prevent accidental damage to the soil vapor point.
- The tubing top should be fitted with a Swagelok® and cap to prevent moisture and foreign material from infiltrating the tubing.

2.3. Soil Vapor Sample Collection

Soil vapor samples should be collected as indicated in the work plan and in accordance with applicable state or federal guidance documents. Specifically, samples from the points should be collected as follows:

- Permanent soil vapor points should not be sampled or purged for a minimum of 24 hours after installation. Temporary points may be purged and sampled immediately following installation.
- Document pertinent field conditions prior to sampling as described above.
- A suction pump should be used to remove a minimum of three implant volumes from the soil vapor points prior to sampling. Include the volume of any additional tubing added to affix sampling equipment and the annular space between the probe and the native material if sand or glass beads were used.
- The purge rate shall not exceed 0.2 liters per minute.
- Samples should be collected for volatile organic compounds (VOCs) in an individually laboratory certified clean 1-liter SUMMA® canister (or equivalent) using a certified flow controller calibrated for the anticipated sample duration (4 minutes). The regulator flow rate should not exceed 0.2 liters per minute.
- A helium tracer gas should be used to identify any potential migration or short circuiting of ambient air during sampling as described below.
- Remove the protective brass plug from the canister. Connect the pre-calibrated flow controller to the canister.
- Record the identification numbers for the canister and flow controller.
- Record the initial canister pressure on the vacuum gauge (check equipment-specific instructions for taking this measurement). A canister with a significantly different pressure than originally recorded by the testing

laboratory should not be used for sampling. Record these numbers and values on the chain-of-custody form for each sample.

- Connect the tubing from the soil vapor probe to the flow controller.
- Open the valve on the canister. Record the time that the valve was opened (beginning of sampling) and the canister pressure on the vacuum gauge.
- Photograph the canister and the area surrounding the canister.
- Monitor the vacuum pressure in the canister routinely during sampling.
- Stop sample collection when the canister still has a minimum amount of vacuum remaining. Check with the laboratory supplying the canister and flow controller for the ideal final vacuum pressure. Typically, the minimum vacuum is between 2 and 5 inches of mercury, but not zero. If there is no vacuum remaining, the sample should be rejected and collected again in a new canister.
- Record the final vacuum pressure and close the canister valve. Record the date and time that sample collection was stopped.
- Remove the flow controller from the canister and replace the protective brass plug.
- Attach labels/tags (sample name, time/date of sampling, etc.) to the canister as directed by the laboratory.
- Place the canister and other laboratory-supplied equipment in the packaging provided by the laboratory.
- Enter the information required for each sample on the chain-of-custody form, making sure to include the identification numbers for the canister and flow controller, and the initial and final canister pressures on the vacuum gauge.
- Samples should be analyzed for VOCs and naphthalene via modified USEPA modified Method TO-15 and helium via ASTM D-1945.
- Include the required copies of the chain-of-custody form in the shipping packaging, as directed by the laboratory. Maintain a copy of the chain-of-custody for the project file.
- Deliver or ship the samples to the laboratory as soon as practical.
- All laboratory analytical data should be validated by a data validation professional in accordance with the USEPA Contract Laboratory Program National Functional Guidelines for Organic Data Review, January 2005 and the USEPA Region II Standard Operating Procedure (SOP) for the Validation of Organic Data modified to accommodate the USEPA Method TO-15 and natural gas analysis by ASTM D-1945.

2.4. Tracer Gas Evaluation

The tracer gas evaluation provides a means to evaluate the integrity of the soil vapor probe seal and assess the potential for introduction of ambient air into the soil vapor sample.

A tracer gas evaluation should be conducted on the each temporary soil vapor probe to be sampled in a sampling event. A tracer gas evaluation should be conducted on

the each permanent soil vapor probe during the initial sampling event and a minimum of 10% of the soil vapor probes during subsequent sampling events.

The following tracer gas evaluation procedure uses helium as a tracer gases which can be measured through laboratory analysis or by a portable detector.

Retain the tracer gas around the sample probe by filling an air-tight chamber (such as a plastic bucket) positioned over the sample location.

- Make sure the chamber is suitably sealed to the ground surface.
- Introduce the tracer gas into the chamber. The chamber should have tubing at the top of the chamber to introduce the tracer gas into the chamber and a valved fitting at the bottom to let the ambient air out while introducing tracer gas. Close the valve after the chamber has been enriched with tracer gas at concentrations >10%.
- The chamber should have a gas-tight fitting or sealable penetration to allow the soil vapor sample probe tubing to pass through and exit the chamber.
- After the chamber has been filled with tracer gas, attach the sample probe tubing to a pump that should be pre-calibrated to extract soil vapor at a rate of no more than 0.2 liters per minute. Purge the tubing using the pump. Calculate the volume of air in the tubing and probe and purge one to three tubing/probe volumes prior collecting an analytical sample or using a portable device to measuring the tracer gas concentration.
- Samples collected from vapor points during a tracer gas evaluation should be analyzed for VOCs and naphthalene via modified USEPA modified Method TO-15 and helium via ASTM D-1945.
- Alternately, a tracer gas detector may be used to verify the presence of the tracer gas in the chamber by affixing it to the valve fitting at the bottom of the chamber. The tracer gas detector may also be used to measure the tracer gas concentration in the pump exhaust during purging. If used, then record the tracer gas concentrations in the chamber and in the soil vapor sample.
- Based on the concentrations of the tracer gas detected during analysis or direct measurement, determine whether additional gas tracer evaluations are necessary.

If the evaluation on a probe indicates a high concentration of tracer gas in the sample (>10% of the concentration of the tracer gas in the chamber), then the surface seal is not sufficient and requires improvement via repair or replacement prior to commencement subsequent sample collection.

A non-detectable level of tracer gas is preferred, however, if the evaluation on a probe indicates a low potential for introduction of ambient air into the sample (<10% of the concentration of the tracer gas in the chamber), then proceed with the soil

vapor sampling. While lower concentrations of tracer gas are acceptable, the impact of the detectable leak on sample results should be evaluated in the sampling report.

3. References

USEPA modified Method TO-15 and helium via ASTM D-1945

Section 2.7.1 of the New York State Department of Health (NYSDOH) Final Guidance for Evaluating Soil Vapor Intrusion in the State of New York*, dated October 2006.*

4. Contact

Chris Berotti

Appendix B Lab SOPs and Project Limits

Date Created: 01/24/25
Created By: Melissa Deyo
File: PM18460-1
Page: 1

NY PFAAs via EPA 1633 (WATER)

Holding Time: 28 days
Container/Sample Preservation: 2 - Plastic 500ml unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Perfluorobutanoic Acid (PFBA)	375-22-4	6.4	0.528	ng/l	70-140	30	70-140	30	30			
Perfluoropentanoic Acid (PFPeA)	2706-90-3	3.2	0.36	ng/l	65-135	30	65-135	30	30			
Perfluorobutanesulfonic Acid (PFBS)	375-73-5	1.6	0.4	ng/l	60-145	30	60-145	30	30			
1H,1H,2H,2H-Perfluorohexanesulfonic Acid (4:2FTS)	757124-72-4	6.4	0.912	ng/l	70-145	30	70-145	30	30			
Perfluorohexanoic Acid (PFHxA)	307-24-4	1.6	0.248	ng/l	70-145	30	70-145	30	30			
Perfluoropentanesulfonic Acid (PFPeS)	2706-91-4	1.6	0.208	ng/l	65-140	30	65-140	30	30			
Perfluoroheptanoic Acid (PFHpA)	375-85-9	1.6	0.24	ng/l	70-150	30	70-150	30	30			
Perfluorohexanesulfonic Acid (PFHxS)	355-46-4	1.6	0.136	ng/l	65-145	30	65-145	30	30			
Perfluorooctanoic Acid (PFOA)	335-67-1	1.6	0.264	ng/l	70-150	30	70-150	30	30			
1H,1H,2H,2H-Perfluorooctanesulfonic Acid (6:2FTS)	27619-97-2	6.4	4.816	ng/l	65-155	30	65-155	30	30			
Perfluoroheptanesulfonic Acid (PFHpS)	375-92-8	1.6	0.2	ng/l	70-150	30	70-150	30	30			
Perfluorononanoic Acid (PFNA)	375-95-1	1.6	0.264	ng/l	70-150	30	70-150	30	30			
Perfluorooctanesulfonic Acid (PFOS)	1763-23-1	1.6	0.264	ng/l	55-150	30	55-150	30	30			
Perfluorodecanoic Acid (PFDA)	335-76-2	1.6	0.208	ng/l	70-140	30	70-140	30	30			
1H,1H,2H,2H-Perfluorodecanesulfonic Acid (8:2FTS)	39108-34-4	6.4	1.224	ng/l	60-150	30	60-150	30	30			
Perfluorononanesulfonic Acid (PFNS)	68259-12-1	1.6	0.2	ng/l	65-145	30	65-145	30	30			
N-Methyl Perfluorooctanesulfonamidoacetic Acid (NMeFOSA	2355-31-9	1.6	0.48	ng/l	50-140	30	50-140	30	30			
Perfluoroundecanoic Acid (PFUnA)	2058-94-8	1.6	0.176	ng/l	70-145	30	70-145	30	30			
Perfluorodecanesulfonic Acid (PFDS)	335-77-3	1.6	0.136	ng/l	60-145	30	60-145	30	30			
Perfluorooctanesulfonamide (FOSA)	754-91-6	1.6	0.096	ng/l	70-145	30	70-145	30	30			
N-Ethyl Perfluorooctanesulfonamidoacetic Acid (NEtFOSAA)	2991-50-6	1.6	0.48	ng/l	70-145	30	70-145	30	30			
Perfluorododecanoic Acid (PFDoA)	307-55-1	1.6	0.216	ng/l	70-140	30	70-140	30	30			
Perfluorotridecanoic Acid (PFTrDA)	72629-94-8	1.6	0.184	ng/l	65-140	30	65-140	30	30			
Perfluorotetradecanoic Acid (PFTA)	376-06-7	1.6	0.16	ng/l	60-140	30	60-140	30	30			
2,3,3,3-Tetrafluoro-2-[1,1,2,2,3,3,3-Heptafluoropropoxy]-Pr	13252-13-6	6.4	1.6	ng/l	70-140	30	70-140	30	30			
4,8-Dioxa-3h-Perfluorononanoic Acid (ADONA)	919005-14-4	6.4	0.376	ng/l	65-145	30	65-145	30	30			
Perfluorododecane Sulfonic Acid (PFDoDS)	79780-39-5	1.6	0.24	ng/l	50-145	30	50-145	30	30			
9-Chlorohexadecafluoro-3-Oxanone-1-Sulfonic Acid (9Cl-PF	756426-58-1	6.4	0.44	ng/l	70-155	30	70-155	30	30			
11-Chloroeicosafluoro-3-Oxaundecane-1-Sulfonic Acid (11Cl	763051-92-9	6.4	0.448	ng/l	55-160	30	55-160	30	30			
N-Methyl Perfluorooctane Sulfonamide (NMeFOSA)	31506-32-8	1.6	0.224	ng/l	60-150	30	60-150	30	30			
N-Ethyl Perfluorooctane Sulfonamide (NEtFOSA)	4151-50-2	1.6	0.352	ng/l	65-145	30	65-145	30	30			
N-Methyl Perfluorooctanesulfonamido Ethanol (NMeFOSE)	24448-09-7	16	1.304	ng/l	70-145	30	70-145	30	30			
N-Ethyl Perfluorooctanesulfonamido Ethanol (NEtFOSE)	1691-99-2	16	1.104	ng/l	70-135	30	70-135	30	30			
Perfluoro-3-Methoxypropanoic Acid (PFMPA)	377-73-1	3.2	0.248	ng/l	55-140	30	55-140	30	30			
Perfluoro-4-Methoxybutanoic Acid (PFMBA)	863090-89-5	3.2	0.36	ng/l	60-150	30	60-150	30	30			
Perfluoro(2-Ethoxyethane)Sulfonic Acid (PFEESA)	113507-82-7	3.2	0.328	ng/l	70-140	30	70-140	30	30			
Nonafluoro-3,6-Dioxaheptanoic Acid (NFDHA)	151772-58-6	3.2	0.544	ng/l	50-150	30	50-150	30	30			
3-Perfluoropropyl Propanoic Acid (3:3FTCA)	356-02-5	8	0.536	ng/l	65-130	30	65-130	30	30			
2H,2H,3H,3H-Perfluorooctanoic Acid (5:3FTCA)	914637-49-3	40	4.256	ng/l	70-135	30	70-135	30	30			
3-Perfluoroheptyl Propanoic Acid (7:3FTCA)	812-70-4	40	3.184	ng/l	50-145	30	50-145	30	30			
Perfluoro[13C4]Butanoic Acid (MPFBA)	*NONE*									*5-130*		
Perfluoro[13C5]Pentanoic Acid (M5PFPEA)	*NONE*									*40-130*		

Please Note that the RL information provided in this table is calculated using a 100% Solids factor (Soil/Solids only)
Please Note that the information provided in this table is subject to change at anytime at the discretion of Pace Analytical Services

Date Created: 01/24/25
Created By: Melissa Deyo
File: PM18460-1

NY PFAAs via EPA 1633 (WATER)

Holding Time: 28 days
Container/Sample Preservation: 2 - Plastic 500ml unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Perfluoro[2,3,4-13C3]Butanesulfonic Acid (M3PFBS)	*NONE*									*40-135*		
1H,1H,2H,2H-Perfluoro[1,2-13C2]Hexanesulfonic Acid (M2-	*NONE*									*40-200*		
Perfluoro[1,2,3,4,6-13C5]Hexanoic Acid (M5PFHxA)	*NONE*									*40-130*		
Perfluoro[1,2,3,4-13C4]Heptanoic Acid (M4PFHpA)	*NONE*									*40-130*		
Perfluoro[1,2,3-13C3]Hexanesulfonic Acid (M3PFHxS)	*NONE*									*40-130*		
Perfluoro[13C8]Octanoic Acid (M8PFOA)	*NONE*									*40-130*		
1H,1H,2H,2H-Perfluoro[1,2-13C2]Octanesulfonic Acid (M2-	*NONE*									*40-200*		
Perfluoro[13C9]Nonanoic Acid (M9PFNA)	*NONE*									*40-130*		
Perfluoro[13C8]Octanesulfonic Acid (M8PFOS)	*NONE*									*40-130*		
Perfluoro[1,2,3,4,5,6-13C6]Decanoic Acid (M6PFDA)	*NONE*									*40-130*		
1H,1H,2H,2H-Perfluoro[1,2-13C2]Decanesulfonic Acid (M2-	*NONE*									*40-300*		
N-Deuteriomethylperfluoro-1-octanesulfonamidoacetic Acid	*NONE*									*40-170*		
Perfluoro[1,2,3,4,5,6,7-13C7]Undecanoic Acid (M7-PFUDA)	*NONE*									*30-130*		
Perfluoro[13C8]Octanesulfonamide (M8FOSA)	*NONE*									*40-130*		
N-Deuterioethylperfluoro-1-octanesulfonamidoacetic Acid (	*NONE*									*25-135*		
Perfluoro[1,2-13C2]Dodecanoic Acid (MPFDOA)	*NONE*									*10-130*		
Perfluoro[1,2-13C2]Tetradecanoic Acid (M2PFTEDA)	*NONE*									*10-130*		
2,3,3,3-Tetrafluoro-2-[1,1,2,2,3,3,3-Heptafluoropropoxy]-1	*NONE*									*40-130*		
N-Methyl-d3-Perfluoro-1-Octanesulfonamide (d3-NMeFOSA)	*NONE*									*10-130*		
N-Ethyl-d5-Perfluoro-1-Octanesulfonamide (d5-NEtFOSA)	*NONE*									*10-130*		
2-(N-Methyl-d3-Perfluoro-1-Octanesulfonamido)ethan-d4-o	*1265205-95-5*									*10-130*		
2-(N-Ethyl-d5-Perfluoro-1-Octanesulfonamido)ethan-d4-ol (	*NONE*									*10-130*		

Please Note that the RL information provided in this table is calculated using a 100% Solids factor ***(Soil/Solids only)***
Please Note that the information provided in this table is subject to change at anytime at the discretion of Pace Analytical Services

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File: PM18460-1
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1,4 Dioxane via EPA 8270E-SIM (WATER)

Holding Time: 7 days
Container/Sample Preservation: 2 - Amber 250ml unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
1,4-Dioxane	123-91-1	150	33.9	ng/l	40-140	30	40-140	30	30			
1,4-Dioxane-d8	*17647-74-4*									*15-110*		
1,4-Dioxane-d8 (IS)	17647-74-4			ng/l								

Please Note that the RL information provided in this table is calculated using a 100% Solids factor (Soil/Solids only)
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NYTCL Semivolatiles - EPA 8270E (RVT) (WATER)

Holding Time: 7 days
Container/Sample Preservation: 2 - Amber 100ml unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Acenaphthene	83-32-9	2	0.403	ug/l	37-111	30	37-111	30	30			
1,2,4-Trichlorobenzene	120-82-1	5	0.977	ug/l	39-98	30	39-98	30	30			
Hexachlorobenzene	118-74-1	2	0.452	ug/l	40-140	30	40-140	30	30			
Bis(2-chloroethyl)ether	111-44-4	2	0.392	ug/l	40-140	30	40-140	30	30			
2-Chloronaphthalene	91-58-7	2	0.35	ug/l	40-140	30	40-140	30	30			
1,2-Dichlorobenzene	95-50-1	2	0.329	ug/l	40-140	30	40-140	30	30			
1,3-Dichlorobenzene	541-73-1	2	0.315	ug/l	40-140	30	40-140	30	30			
1,4-Dichlorobenzene	106-46-7	2	0.391	ug/l	36-97	30	36-97	30	30			
3,3'-Dichlorobenzidine	91-94-1	5	1.85	ug/l	40-140	30	40-140	30	30			
2,4-Dinitrotoluene	121-14-2	5	0.541	ug/l	48-143	30	48-143	30	30			
2,6-Dinitrotoluene	606-20-2	5	0.845	ug/l	40-140	30	40-140	30	30			
Fluoranthene	206-44-0	2	0.411	ug/l	40-140	30	40-140	30	30			
4-Chlorophenyl phenyl ether	7005-72-3	2	0.386	ug/l	40-140	30	40-140	30	30			
4-Bromophenyl phenyl ether	101-55-3	2	0.244	ug/l	40-140	30	40-140	30	30			
Bis(2-chloroisopropyl)ether	108-60-1	2	0.403	ug/l	40-140	30	40-140	30	30			
Bis(2-chloroethoxy)methane	111-91-1	5	0.842	ug/l	40-140	30	40-140	30	30			
Hexachlorobutadiene	87-68-3	2	0.355	ug/l	40-140	30	40-140	30	30			
Hexachlorocyclopentadiene	77-47-4	20	1.23	ug/l	40-140	30	40-140	30	30			
Hexachloroethane	67-72-1	2	0.203	ug/l	40-140	30	40-140	30	30			
Isophorone	78-59-1	5	0.862	ug/l	40-140	30	40-140	30	30			
Naphthalene	91-20-3	2	0.542	ug/l	40-140	30	40-140	30	30			
Nitrobenzene	98-95-3	2	0.205	ug/l	40-140	30	40-140	30	30			
NitrosoDiPhenylAmine(NDPA)/DPA	86-30-6	2	0.924	ug/l	40-140	30	40-140	30	30			
n-Nitrosodi-n-propylamine	621-64-7	5	0.906	ug/l	29-132	30	29-132	30	30			
Bis(2-Ethylhexyl)phthalate	117-81-7	3	1.42	ug/l	40-140	30	40-140	30	30			
Butyl benzyl phthalate	85-68-7	5	2.61	ug/l	40-140	30	40-140	30	30			
Di-n-butylphthalate	84-74-2	5	0.957	ug/l	40-140	30	40-140	30	30			
Di-n-octylphthalate	117-84-0	5	2.26	ug/l	40-140	30	40-140	30	30			
Diethyl phthalate	84-66-2	5	0.765	ug/l	40-140	30	40-140	30	30			
Dimethyl phthalate	131-11-3	5	0.916	ug/l	40-140	30	40-140	30	30			
Benzo(a)anthracene	56-55-3	2	0.323	ug/l	40-140	30	40-140	30	30			
Benzo(a)pyrene	50-32-8	2	0.368	ug/l	40-140	30	40-140	30	30			
Benzo(b)fluoranthene	205-99-2	2	0.533	ug/l	40-140	30	40-140	30	30			
Benzo(k)fluoranthene	207-08-9	2	0.621	ug/l	40-140	30	40-140	30	30			
Chrysene	218-01-9	2	0.222	ug/l	40-140	30	40-140	30	30			
Acenaphthylene	208-96-8	2	0.315	ug/l	45-123	30	45-123	30	30			
Anthracene	120-12-7	2	0.467	ug/l	40-140	30	40-140	30	30			
Benzo(ghi)perylene	191-24-2	2	0.369	ug/l	40-140	30	40-140	30	30			
Fluorene	86-73-7	2	0.439	ug/l	40-140	30	40-140	30	30			
Phenanthrene	85-01-8	2	0.419	ug/l	40-140	30	40-140	30	30			
Dibenzo(a,h)anthracene	53-70-3	2	0.286	ug/l	40-140	30	40-140	30	30			
Indeno(1,2,3-cd)Pyrene	193-39-5	2	0.484	ug/l	40-140	30	40-140	30	30			

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NYTCL Semivolatiles - EPA 8270E (RVT) (WATER)

Holding Time: 7 days
Container/Sample Preservation: 2 - Amber 100ml unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Pyrene	129-00-0	2	0.407	ug/l	26-127	30	26-127	30	30			
Biphenyl	92-52-4	2	0.196	ug/l	40-140	30	40-140	30	30			
4-Chloroaniline	106-47-8	5	0.468	ug/l	40-140	30	40-140	30	30			
2-Nitroaniline	88-74-4	5	1.03	ug/l	52-143	30	52-143	30	30			
3-Nitroaniline	99-09-2	5	1.16	ug/l	25-145	30	25-145	30	30			
4-Nitroaniline	100-01-6	5	1.45	ug/l	51-143	30	51-143	30	30			
Dibenzofuran	132-64-9	2	0.401	ug/l	40-140	30	40-140	30	30			
2-Methylnaphthalene	91-57-6	2	0.372	ug/l	40-140	30	40-140	30	30			
Acetophenone	98-86-2	5	0.917	ug/l	39-129	30	39-129	30	30			
2,4,6-Trichlorophenol	88-06-2	5	2.09	ug/l	30-130	30	30-130	30	30			
P-Chloro-M-Cresol	59-50-7	2	0.606	ug/l	23-97	30	23-97	30	30			
2-Chlorophenol	95-57-8	2	0.653	ug/l	27-123	30	27-123	30	30			
2,4-Dichlorophenol	120-83-2	5	1.7	ug/l	30-130	30	30-130	30	30			
2,4-Dimethylphenol	105-67-9	5	2.04	ug/l	30-130	30	30-130	30	30			
2-Nitrophenol	88-75-5	10	1.95	ug/l	30-130	30	30-130	30	30			
4-Nitrophenol	100-02-7	10	1.42	ug/l	10-80	30	10-80	30	30			
2,4-Dinitrophenol	51-28-5	20	5.42	ug/l	20-130	30	20-130	30	30			
4,6-Dinitro-o-cresol	534-52-1	10	2.31	ug/l	20-164	30	20-164	30	30			
Pentachlorophenol	87-86-5	10	2.51	ug/l	9-103	30	9-103	30	30			
Phenol	108-95-2	5	0.35	ug/l	12-110	30	12-110	30	30			
2-Methylphenol	95-48-7	5	2.31	ug/l	30-130	30	30-130	30	30			
3-Methylphenol/4-Methylphenol	106-44-5	5	1.39	ug/l	30-130	30	30-130	30	30			
2,4,5-Trichlorophenol	95-95-4	5	2.07	ug/l	30-130	30	30-130	30	30			
Benzoic Acid	65-85-0	50	2.62	ug/l	10-164	30	10-164	30	30			
Benzyl Alcohol	100-51-6	2	0.381	ug/l	26-116	30	26-116	30	30			
Carbazole	86-74-8	2	0.309	ug/l	55-144	30	55-144	30	30			
2-Fluorophenol	*367-12-4*									*21-120*		
Phenol-d6	*13127-88-3*									*10-120*		
Nitrobenzene-d5	*4165-60-0*									*23-120*		
2-Fluorobiphenyl	*321-60-8*									*15-120*		
2,4,6-Tribromophenol	*118-79-6*									*10-120*		
4-Terphenyl-d14	*1718-51-0*									*41-149*		

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METALS by 6020B (WATER)

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	Holding Time	Container
Aluminum, Total	7429-90-5	0.01	0.00327	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Antimony, Total	7440-36-0	0.004	0.000429	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Arsenic, Total	7440-38-2	0.0005	0.000165	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Barium, Total	7440-39-3	0.0005	0.000173	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Beryllium, Total	7440-41-7	0.0005	0.000107	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Cadmium, Total	7440-43-9	0.0002	0.0000599	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Calcium, Total	7440-70-2	0.1	0.0394	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Chromium, Total	7440-47-3	0.001	0.000178	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Cobalt, Total	7440-48-4	0.0005	0.000163	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Copper, Total	7440-50-8	0.001	0.000384	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Iron, Total	7439-89-6	0.05	0.0191	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Lead, Total	7439-92-1	0.001	0.000343	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Magnesium, Total	7439-95-4	0.07	0.0242	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Manganese, Total	7439-96-5	0.001	0.00044	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Nickel, Total	7440-02-0	0.002	0.000556	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Potassium, Total	7440-09-7	0.1	0.0309	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Selenium, Total	7782-49-2	0.005	0.00173	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Silver, Total	7440-22-4	0.0004	0.000163	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Sodium, Total	7440-23-5	0.5	0.0293	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Thallium, Total	7440-28-0	0.001	0.000143	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Vanadium, Total	7440-62-2	0.005	0.00157	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved
Zinc, Total	7440-66-6	0.01	0.00341	mg/l	80-120		75-125	20	20		180 days	1 - Plastic 500ml HNO3 preserved

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METALS by 7470A (WATER)

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	Holding Time	Container
Mercury, Total	7439-97-6	0.0002	0.0000915	mg/l	80-120		75-125	20	20		28 days	1 - Plastic 500ml HNO3 preserved

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TCL PCBs - EPA 8082A (RVT) (WATER)

Holding Time: 365 days
Container/Sample Preservation: 2 - Amber 100ml unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Aroclor 1016	12674-11-2	0.071	0.0128	ug/l	40-140	50	40-140	50	50			
Aroclor 1221	11104-28-2	0.071	0.0153	ug/l	40-140	50	40-140	50	50			
Aroclor 1232	11141-16-5	0.071	0.0153	ug/l	40-140	50	40-140	50	50			
Aroclor 1242	53469-21-9	0.071	0.0153	ug/l	40-140	50	40-140	50	50			
Aroclor 1248	12672-29-6	0.071	0.0153	ug/l	40-140	50	40-140	50	50			
Aroclor 1254	11097-69-1	0.071	0.0153	ug/l	40-140	50	40-140	50	50			
Aroclor 1260	11096-82-5	0.071	0.0153	ug/l	40-140	50	40-140	50	50			
Aroclor 1262	37324-23-5	0.071	0.0153	ug/l	40-140	50	40-140	50	50			
Aroclor 1268	11100-14-4	0.071	0.0153	ug/l	40-140	50	40-140	50	50			
PCBs, Total	1336-36-3	0.071	0.0153	ug/l				50	50			
2,4,5,6-Tetrachloro-m-xylene	*877-09-8*									*30-150*		
Decachlorobiphenyl	*2051-24-3*									*30-150*		

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NYTCL Semivolatiles -EPA 8270E-SIM (RVT) (WATER)

Holding Time: 7 days
Container/Sample Preservation: 2 - Amber 100ml unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Acenaphthene	83-32-9	0.1	0.0235	ug/l	40-140	40	40-140	40	40			
2-Chloronaphthalene	91-58-7	0.2	0.0227	ug/l	40-140	40	40-140	40	40			
Fluoranthene	206-44-0	0.1	0.0272	ug/l	40-140	40	40-140	40	40			
Hexachlorobutadiene	87-68-3	0.5	0.02	ug/l	40-140	40	40-140	40	40			
Naphthalene	91-20-3	0.1	0.0245	ug/l	40-140	40	40-140	40	40			
Benzo(a)anthracene	56-55-3	0.1	0.0295	ug/l	40-140	40	40-140	40	40			
Benzo(a)pyrene	50-32-8	0.1	0.024	ug/l	40-140	40	40-140	40	40			
Benzo(b)fluoranthene	205-99-2	0.1	0.0272	ug/l	40-140	40	40-140	40	40			
Benzo(k)fluoranthene	207-08-9	0.1	0.0338	ug/l	40-140	40	40-140	40	40			
Chrysene	218-01-9	0.1	0.031	ug/l	40-140	40	40-140	40	40			
Acenaphthylene	208-96-8	0.1	0.0205	ug/l	40-140	40	40-140	40	40			
Anthracene	120-12-7	0.1	0.0239	ug/l	40-140	40	40-140	40	40			
Benzo(ghi)perylene	191-24-2	0.1	0.0237	ug/l	40-140	40	40-140	40	40			
Fluorene	86-73-7	0.1	0.0255	ug/l	40-140	40	40-140	40	40			
Phenanthrene	85-01-8	0.1	0.0392	ug/l	40-140	40	40-140	40	40			
Dibenzo(a,h)anthracene	53-70-3	0.1	0.0235	ug/l	40-140	40	40-140	40	40			
Indeno(1,2,3-cd)Pyrene	193-39-5	0.1	0.022	ug/l	40-140	40	40-140	40	40			
Pyrene	129-00-0	0.1	0.0425	ug/l	40-140	40	40-140	40	40			
2-Methylnaphthalene	91-57-6	0.1	0.0277	ug/l	40-140	40	40-140	40	40			
Pentachlorophenol	87-86-5	0.8	0.0565	ug/l	40-140	40	40-140	40	40			
Hexachlorobenzene	118-74-1	0.8	0.0134	ug/l	40-140	40	40-140	40	40			
Hexachloroethane	67-72-1	0.8	0.0224	ug/l	40-140	40	40-140	40	40			
2-Fluorophenol	*367-12-4*									*21-120*		
Phenol-d6	*13127-88-3*									*10-120*		
Nitrobenzene-d5	*4165-60-0*									*23-120*		
2-Fluorobiphenyl	*321-60-8*									*15-120*		
2,4,6-Tribromophenol	*118-79-6*									*10-120*		
4-Terphenyl-d14	*1718-51-0*									*41-149*		

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TCL Volatiles - EPA 8260D (WATER)

Holding Time: 14 days
Container/Sample Preservation: 3 - Vial HCl preserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Methylene chloride	75-09-2	2.5	0.7	ug/l	70-130	20	70-130	20	20			
1,1-Dichloroethane	75-34-3	2.5	0.7	ug/l	70-130	20	70-130	20	20			
Chloroform	67-66-3	2.5	0.7	ug/l	70-130	20	70-130	20	20			
Carbon tetrachloride	56-23-5	0.5	0.134	ug/l	63-132	20	63-132	20	20			
1,2-Dichloropropane	78-87-5	1	0.137	ug/l	70-130	20	70-130	20	20			
Dibromochloromethane	124-48-1	0.5	0.149	ug/l	63-130	20	63-130	20	20			
1,1,2-Trichloroethane	79-00-5	1.5	0.5	ug/l	70-130	20	70-130	20	20			
Tetrachloroethene	127-18-4	0.5	0.181	ug/l	70-130	20	70-130	20	20			
Chlorobenzene	108-90-7	2.5	0.7	ug/l	75-130	20	75-130	20	20			
Trichlorofluoromethane	75-69-4	2.5	0.7	ug/l	62-150	20	62-150	20	20			
1,2-Dichloroethane	107-06-2	0.5	0.132	ug/l	70-130	20	70-130	20	20			
1,1,1-Trichloroethane	71-55-6	2.5	0.7	ug/l	67-130	20	67-130	20	20			
Bromodichloromethane	75-27-4	0.5	0.192	ug/l	67-130	20	67-130	20	20			
trans-1,3-Dichloropropene	10061-02-6	0.5	0.164	ug/l	70-130	20	70-130	20	20			
cis-1,3-Dichloropropene	10061-01-5	0.5	0.144	ug/l	70-130	20	70-130	20	20			
1,3-Dichloropropene, Total	542-75-6	0.5	0.144	ug/l				20	20			
Bromoform	75-25-2	2	0.65	ug/l	54-136	20	54-136	20	20			
1,1,2,2-Tetrachloroethane	79-34-5	0.5	0.167	ug/l	67-130	20	67-130	20	20			
Benzene	71-43-2	0.5	0.159	ug/l	70-130	20	70-130	20	20			
Toluene	108-88-3	2.5	0.7	ug/l	70-130	20	70-130	20	20			
Ethylbenzene	100-41-4	2.5	0.7	ug/l	70-130	20	70-130	20	20			
Chloromethane	74-87-3	2.5	0.7	ug/l	64-130	20	64-130	20	20			
Bromomethane	74-83-9	2.5	0.7	ug/l	39-139	20	39-139	20	20			
Vinyl chloride	75-01-4	1	0.0714	ug/l	55-140	20	55-140	20	20			
Chloroethane	75-00-3	2.5	0.7	ug/l	55-138	20	55-138	20	20			
1,1-Dichloroethene	75-35-4	0.5	0.169	ug/l	61-145	20	61-145	20	20			
trans-1,2-Dichloroethene	156-60-5	2.5	0.7	ug/l	70-130	20	70-130	20	20			
Trichloroethene	79-01-6	0.5	0.175	ug/l	70-130	20	70-130	20	20			
1,2-Dichlorobenzene	95-50-1	2.5	0.7	ug/l	70-130	20	70-130	20	20			
1,3-Dichlorobenzene	541-73-1	2.5	0.7	ug/l	70-130	20	70-130	20	20			
1,4-Dichlorobenzene	106-46-7	2.5	0.7	ug/l	70-130	20	70-130	20	20			
Methyl tert butyl ether	1634-04-4	2.5	0.166	ug/l	63-130	20	63-130	20	20			
p/m-Xylene	179601-23-1	2.5	0.7	ug/l	70-130	20	70-130	20	20			
o-Xylene	95-47-6	2.5	0.7	ug/l	70-130	20	70-130	20	20			
Xylene (Total)	1330-20-7	2.5	0.7	ug/l				20	20			
cis-1,2-Dichloroethene	156-59-2	2.5	0.7	ug/l	70-130	20	70-130	20	20			
1,2-Dichloroethene (total)	540-59-0	2.5	0.7	ug/l				20	20			
Styrene	100-42-5	2.5	0.7	ug/l	70-130	20	70-130	20	20			
Dichlorodifluoromethane	75-71-8	5	1	ug/l	36-147	20	36-147	20	20			
Acetone	67-64-1	5	1.46	ug/l	58-148	20	58-148	20	20			
Carbon disulfide	75-15-0	5	1	ug/l	51-130	20	51-130	20	20			
2-Butanone	78-93-3	5	1.94	ug/l	63-138	20	63-138	20	20			

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TCL Volatiles - EPA 8260D (WATER)

Holding Time: 14 days
Container/Sample Preservation: 3 - Vial HCl preserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
4-Methyl-2-pentanone	108-10-1	5	1	ug/l	59-130	20	59-130	20	20			
2-Hexanone	591-78-6	5	1	ug/l	57-130	20	57-130	20	20			
Bromochloromethane	74-97-5	2.5	0.7	ug/l	70-130	20	70-130	20	20			
1,2-Dibromoethane	106-93-4	2	0.65	ug/l	70-130	20	70-130	20	20			
n-Butylbenzene	104-51-8	2.5	0.7	ug/l	53-136	20	53-136	20	20			
sec-Butylbenzene	135-98-8	2.5	0.7	ug/l	70-130	20	70-130	20	20			
tert-Butylbenzene	98-06-6	2.5	0.7	ug/l	70-130	20	70-130	20	20			
1,2-Dibromo-3-chloropropane	96-12-8	2.5	0.7	ug/l	41-144	20	41-144	20	20			
Isopropylbenzene	98-82-8	2.5	0.7	ug/l	70-130	20	70-130	20	20			
p-Isopropyltoluene	99-87-6	2.5	0.7	ug/l	70-130	20	70-130	20	20			
n-Propylbenzene	103-65-1	2.5	0.7	ug/l	69-130	20	69-130	20	20			
1,2,3-Trichlorobenzene	87-61-6	2.5	0.7	ug/l	70-130	20	70-130	20	20			
1,2,4-Trichlorobenzene	120-82-1	2.5	0.7	ug/l	70-130	20	70-130	20	20			
1,3,5-Trimethylbenzene	108-67-8	2.5	0.7	ug/l	64-130	20	64-130	20	20			
1,2,4-Trimethylbenzene	95-63-6	2.5	0.7	ug/l	70-130	20	70-130	20	20			
1,4-Dioxane	123-91-1	250	60.8	ug/l	56-162	20	56-162	20	20			
1,2-Dichloroethane-d4	*17060-07-0*									*70-130*		
Toluene-d8	*2037-26-5*									*70-130*		
4-Bromofluorobenzene	*460-00-4*									*70-130*		
Dibromofluoromethane	*1868-53-7*									*70-130*		

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WETCHEM (WATER)

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Method	Holding Time	Container
Cyanide, Total	57-12-5	0.005	0.0018	mg/l	85-115	20	80-120	20	20	9010C/9012B	14 days	1 - Plastic 250ml NaOH preserved

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TCLP Volatile Organics - EPA 8260D (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Vial Large Septa unpreserved (4oz)

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Methylene chloride	75-09-2	30	6.78	ug/l	70-130	20	70-130	20	20			
1,1-Dichloroethane	75-34-3	7.5	2.1	ug/l	70-130	20	70-130	20	20			
Chloroform	67-66-3	7.5	2.22	ug/l	70-130	20	70-130	20	20			
Carbon tetrachloride	56-23-5	5	1.34	ug/l	63-132	20	63-132	20	20			
1,2-Dichloropropane	78-87-5	17.5	1.37	ug/l	70-130	20	70-130	20	20			
Dibromochloromethane	124-48-1	5	1.49	ug/l	63-130	20	63-130	20	20			
1,1,2-Trichloroethane	79-00-5	7.5	1.44	ug/l	70-130	20	70-130	20	20			
Tetrachloroethene	127-18-4	5	1.81	ug/l	70-130	20	70-130	20	20			
Chlorobenzene	108-90-7	5	1.78	ug/l	75-130	25	75-130	25	25			
Trichlorofluoromethane	75-69-4	25	1.61	ug/l	62-150	20	62-150	20	20			
1,2-Dichloroethane	107-06-2	5	1.32	ug/l	70-130	20	70-130	20	20			
1,1,1-Trichloroethane	71-55-6	5	1.58	ug/l	67-130	20	67-130	20	20			
Bromodichloromethane	75-27-4	5	1.92	ug/l	67-130	20	67-130	20	20			
trans-1,3-Dichloropropene	10061-02-6	5	1.64	ug/l	70-130	20	70-130	20	20			
cis-1,3-Dichloropropene	10061-01-5	5	1.44	ug/l	70-130	20	70-130	20	20			
1,1-Dichloropropene	563-58-6	25	2.4	ug/l	70-130	20	70-130	20	20			
Bromoform	75-25-2	20	2.48	ug/l	54-136	20	54-136	20	20			
1,1,2,2-Tetrachloroethane	79-34-5	5	1.67	ug/l	67-130	20	67-130	20	20			
Benzene	71-43-2	5	1.59	ug/l	70-130	25	70-130	25	25			
Toluene	108-88-3	7.5	2.03	ug/l	70-130	25	70-130	25	25			
Ethylbenzene	100-41-4	5	1.67	ug/l	70-130	20	70-130	20	20			
Chloromethane	74-87-3	25	2	ug/l	64-130	20	64-130	20	20			
Bromomethane	74-83-9	10	2.56	ug/l	39-139	20	39-139	20	20			
Vinyl chloride	75-01-4	10	0.714	ug/l	55-140	20	55-140	20	20			
Chloroethane	75-00-3	10	1.34	ug/l	55-138	20	55-138	20	20			
1,1-Dichloroethene	75-35-4	5	1.69	ug/l	61-145	25	61-145	25	25			
trans-1,2-Dichloroethene	156-60-5	7.5	1.63	ug/l	70-130	20	70-130	20	20			
Trichloroethene	79-01-6	5	1.75	ug/l	70-130	25	70-130	25	25			
1,2-Dichlorobenzene	95-50-1	25	1.84	ug/l	70-130	20	70-130	20	20			
1,3-Dichlorobenzene	541-73-1	25	1.86	ug/l	70-130	20	70-130	20	20			
1,4-Dichlorobenzene	106-46-7	25	1.87	ug/l	70-130	20	70-130	20	20			
Methyl tert butyl ether	1634-04-4	10	1.66	ug/l	63-130	20	63-130	20	20			
p/m-Xylene	179601-23-1	10	3.32	ug/l	70-130	20	70-130	20	20			
o-Xylene	95-47-6	10	3.92	ug/l	70-130	20	70-130	20	20			
cis-1,2-Dichloroethene	156-59-2	5	1.87	ug/l	70-130	20	70-130	20	20			
Dibromomethane	74-95-3	50	3.63	ug/l	70-130	20	70-130	20	20			
1,4-Dichlorobutane	110-56-5	50	4.64	ug/l	70-130	20	70-130	20	20			
1,2,3-Trichloropropane	96-18-4	50	1.76	ug/l	64-130	20	64-130	20	20			
Styrene	100-42-5	10	3.59	ug/l	70-130	20	70-130	20	20			
Dichlorodifluoromethane	75-71-8	50	2.44	ug/l	36-147	20	36-147	20	20			
Acetone	67-64-1	50	14.6	ug/l	58-148	20	58-148	20	20			
Carbon disulfide	75-15-0	50	2.99	ug/l	51-130	20	51-130	20	20			

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TCLP Volatile Organics - EPA 8260D (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Vial Large Septa unpreserved (4oz)

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
2-Butanone	78-93-3	50	19.4	ug/l	63-138	20	63-138	20	20			
Vinyl acetate	108-05-4	50	3.11	ug/l	70-130	20	70-130	20	20			
4-Methyl-2-pentanone	108-10-1	50	4.16	ug/l	59-130	20	59-130	20	20			
2-Hexanone	591-78-6	50	5.15	ug/l	57-130	20	57-130	20	20			
Ethyl methacrylate	97-63-2	50	6.06	ug/l	70-130	20	70-130	20	20			
Acrylonitrile	107-13-1	50	4.3	ug/l	70-130	20	70-130	20	20			
Bromochloromethane	74-97-5	25	1.52	ug/l	70-130	20	70-130	20	20			
Tetrahydrofuran	109-99-9	50	5.25	ug/l	58-130	20	58-130	20	20			
2,2-Dichloropropane	594-20-7	25	2.04	ug/l	63-133	20	63-133	20	20			
1,2-Dibromoethane	106-93-4	20	1.93	ug/l	70-130	20	70-130	20	20			
1,3-Dichloropropane	142-28-9	25	2.12	ug/l	70-130	20	70-130	20	20			
1,1,1,2-Tetrachloroethane	630-20-6	5	1.64	ug/l	64-130	20	64-130	20	20			
Bromobenzene	108-86-1	25	1.52	ug/l	70-130	20	70-130	20	20			
n-Butylbenzene	104-51-8	5	1.92	ug/l	53-136	20	53-136	20	20			
sec-Butylbenzene	135-98-8	5	1.81	ug/l	70-130	20	70-130	20	20			
tert-Butylbenzene	98-06-6	25	1.96	ug/l	70-130	20	70-130	20	20			
o-Chlorotoluene	95-49-8	25	2.15	ug/l	70-130	20	70-130	20	20			
p-Chlorotoluene	106-43-4	25	1.85	ug/l	70-130	20	70-130	20	20			
1,2-Dibromo-3-chloropropane	96-12-8	25	3.53	ug/l	41-144	20	41-144	20	20			
Hexachlorobutadiene	87-68-3	5	2.17	ug/l	63-130	20	63-130	20	20			
Isopropylbenzene	98-82-8	5	1.87	ug/l	70-130	20	70-130	20	20			
p-Isopropyltoluene	99-87-6	5	1.88	ug/l	70-130	20	70-130	20	20			
Naphthalene	91-20-3	25	2.16	ug/l	70-130	20	70-130	20	20			
n-Propylbenzene	103-65-1	5	1.73	ug/l	69-130	20	69-130	20	20			
1,2,3-Trichlorobenzene	87-61-6	25	2.34	ug/l	70-130	20	70-130	20	20			
1,2,4-Trichlorobenzene	120-82-1	25	2.2	ug/l	70-130	20	70-130	20	20			
1,3,5-Trimethylbenzene	108-67-8	25	2.17	ug/l	64-130	20	64-130	20	20			
1,2,4-Trimethylbenzene	95-63-6	25	1.91	ug/l	70-130	20	70-130	20	20			
trans-1,4-Dichloro-2-butene	110-57-6	25	2.13	ug/l	70-130	20	70-130	20	20			
Ethyl ether	60-29-7	25	1.63	ug/l	59-134	20	59-134	20	20			
1,2-Dichloroethane-d4	*17060-07-0*									*70-130*		
Toluene-d8	*2037-26-5*									*70-130*		
4-Bromofluorobenzene	*460-00-4*									*70-130*		
Dibromofluoromethane	*1868-53-7*									*70-130*		

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TPH - Gasoline Range Organics (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Vial MeOH preserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Gasoline Range Organics	NONE	2500	1450	ug/kg	80-120	20	80-120	20	20			
1,1,1-Trifluorotoluene	*98-08-8*									*70-130*		
4-Bromofluorobenzene	*460-00-4*									*70-130*		

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TCLP ABN Compounds - EPA 8270E/1311 (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Glass 250ml/8oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Acenaphthene	83-32-9	2	0.403	ug/l	37-111	30	37-111	30	30			
Benzidine	92-87-5	20	6.01	ug/l	10-75	30	10-75	30	30			
1,2,4-Trichlorobenzene	120-82-1	5	0.977	ug/l	39-98	30	39-98	30	30			
Hexachlorobenzene	118-74-1	2	0.452	ug/l	40-140	30	40-140	30	30			
Bis(2-chloroethyl)ether	111-44-4	2	0.392	ug/l	40-140	30	40-140	30	30			
2-Chloronaphthalene	91-58-7	2	0.35	ug/l	40-140	30	40-140	30	30			
1,2-Dichlorobenzene	95-50-1	2	0.329	ug/l	40-140	30	40-140	30	30			
1,3-Dichlorobenzene	541-73-1	2	0.315	ug/l	40-140	30	40-140	30	30			
1,4-Dichlorobenzene	106-46-7	2	0.391	ug/l	36-97	30	36-97	30	30			
3,3'-Dichlorobenzidine	91-94-1	5	1.85	ug/l	40-140	30	40-140	30	30			
2,4-Dinitrotoluene	121-14-2	5	0.541	ug/l	48-143	30	48-143	30	30			
2,6-Dinitrotoluene	606-20-2	5	0.845	ug/l	40-140	30	40-140	30	30			
Azobenzene	122-66-7	2	0.418	ug/l	40-140	30	40-140	30	30			
Fluoranthene	206-44-0	2	0.411	ug/l	40-140	30	40-140	30	30			
4-Chlorophenyl phenyl ether	7005-72-3	2	0.386	ug/l	40-140	30	40-140	30	30			
4-Bromophenyl phenyl ether	101-55-3	2	0.244	ug/l	40-140	30	40-140	30	30			
Bis(2-chloroisopropyl)ether	108-60-1	2	0.403	ug/l	40-140	30	40-140	30	30			
Bis(2-chloroethoxy)methane	111-91-1	5	0.842	ug/l	40-140	30	40-140	30	30			
Hexachlorobutadiene	87-68-3	2	0.355	ug/l	40-140	30	40-140	30	30			
Hexachlorocyclopentadiene	77-47-4	20	1.23	ug/l	40-140	30	40-140	30	30			
Hexachloroethane	67-72-1	2	0.203	ug/l	40-140	30	40-140	30	30			
Isophorone	78-59-1	5	0.862	ug/l	40-140	30	40-140	30	30			
Naphthalene	91-20-3	2	0.542	ug/l	40-140	30	40-140	30	30			
Nitrobenzene	98-95-3	2	0.205	ug/l	40-140	30	40-140	30	30			
NitrosoDiPhenylAmine(NDPA)/DPA	86-30-6	2	0.924	ug/l	40-140	30	40-140	30	30			
n-Nitrosodi-n-propylamine	621-64-7	5	0.906	ug/l	29-132	30	29-132	30	30			
Bis(2-Ethylhexyl)phthalate	117-81-7	3	1.42	ug/l	40-140	30	40-140	30	30			
Butyl benzyl phthalate	85-68-7	5	2.61	ug/l	40-140	30	40-140	30	30			
Di-n-butylphthalate	84-74-2	5	0.957	ug/l	40-140	30	40-140	30	30			
Di-n-octylphthalate	117-84-0	5	2.26	ug/l	40-140	30	40-140	30	30			
Diethyl phthalate	84-66-2	5	0.765	ug/l	40-140	30	40-140	30	30			
Dimethyl phthalate	131-11-3	5	0.916	ug/l	40-140	30	40-140	30	30			
Benzo(a)anthracene	56-55-3	2	0.323	ug/l	40-140	30	40-140	30	30			
Benzo(a)pyrene	50-32-8	2	0.368	ug/l	40-140	30	40-140	30	30			
Benzo(b)fluoranthene	205-99-2	2	0.533	ug/l	40-140	30	40-140	30	30			
Benzo(k)fluoranthene	207-08-9	2	0.621	ug/l	40-140	30	40-140	30	30			
Chrysene	218-01-9	2	0.222	ug/l	40-140	30	40-140	30	30			
Acenaphthylene	208-96-8	2	0.315	ug/l	45-123	30	45-123	30	30			
Anthracene	120-12-7	2	0.467	ug/l	40-140	30	40-140	30	30			
Benzo(ghi)perylene	191-24-2	2	0.369	ug/l	40-140	30	40-140	30	30			
Fluorene	86-73-7	2	0.439	ug/l	40-140	30	40-140	30	30			
Phenanthrene	85-01-8	2	0.419	ug/l	40-140	30	40-140	30	30			

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TCLP ABN Compounds - EPA 8270E/1311 (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Glass 250ml/8oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Dibenzo(a,h)anthracene	53-70-3	2	0.286	ug/l	40-140	30	40-140	30	30			
Indeno(1,2,3-cd)Pyrene	193-39-5	2	0.484	ug/l	40-140	30	40-140	30	30			
Pyrene	129-00-0	2	0.407	ug/l	26-127	30	26-127	30	30			
Biphenyl	92-52-4	2	0.196	ug/l	40-140	30	40-140	30	30			
Aniline	62-53-3	2	0.67	ug/l	40-140	30	40-140	30	30			
4-Chloroaniline	106-47-8	5	0.468	ug/l	40-140	30	40-140	30	30			
1-Methylnaphthalene	90-12-0	2	0.415	ug/l	41-103	30	41-103	30	30			
2-Nitroaniline	88-74-4	5	1.03	ug/l	52-143	30	52-143	30	30			
3-Nitroaniline	99-09-2	5	1.16	ug/l	25-145	30	25-145	30	30			
4-Nitroaniline	100-01-6	5	1.45	ug/l	51-143	30	51-143	30	30			
Dibenzofuran	132-64-9	2	0.401	ug/l	40-140	30	40-140	30	30			
2-Methylnaphthalene	91-57-6	2	0.372	ug/l	40-140	30	40-140	30	30			
Acetophenone	98-86-2	5	0.917	ug/l	39-129	30	39-129	30	30			
n-Nitrosodimethylamine	62-75-9	2	0.44	ug/l	22-74	30	22-74	30	30			
2,4,6-Trichlorophenol	88-06-2	5	2.09	ug/l	30-130	30	30-130	30	30			
P-Chloro-M-Cresol	59-50-7	2	0.606	ug/l	23-97	30	23-97	30	30			
2-Chlorophenol	95-57-8	2	0.653	ug/l	27-123	30	27-123	30	30			
2,4-Dichlorophenol	120-83-2	5	1.7	ug/l	30-130	30	30-130	30	30			
2,4-Dimethylphenol	105-67-9	5	2.04	ug/l	30-130	30	30-130	30	30			
2-Nitrophenol	88-75-5	10	1.95	ug/l	30-130	30	30-130	30	30			
4-Nitrophenol	100-02-7	10	1.42	ug/l	10-80	30	10-80	30	30			
2,4-Dinitrophenol	51-28-5	20	5.42	ug/l	20-130	30	20-130	30	30			
4,6-Dinitro-o-cresol	534-52-1	10	2.31	ug/l	20-164	30	20-164	30	30			
Pentachlorophenol	87-86-5	10	2.51	ug/l	9-103	30	9-103	30	30			
Phenol	108-95-2	5	0.35	ug/l	12-110	30	12-110	30	30			
2-Methylphenol	95-48-7	5	2.31	ug/l	30-130	30	30-130	30	30			
3-Methylphenol/4-Methylphenol	106-44-5	5	1.39	ug/l	30-130	30	30-130	30	30			
2,4,5-Trichlorophenol	95-95-4	5	2.07	ug/l	30-130	30	30-130	30	30			
Benzoic Acid	65-85-0	50	2.62	ug/l	10-164	30	10-164	30	30			
Benzyl Alcohol	100-51-6	2	0.381	ug/l	26-116	30	26-116	30	30			
Carbazole	86-74-8	2	0.309	ug/l	55-144	30	55-144	30	30			
Pyridine	110-86-1	3.5	0.308	ug/l	10-66	30	10-66	30	30			
2,6-Dichlorophenol	87-65-0	10	2.57	ug/l	40-140	30	40-140	30	30			
2-Fluorophenol	*367-12-4*									*21-120*		
Phenol-d6	*13127-88-3*									*10-120*		
Nitrobenzene-d5	*4165-60-0*									*23-120*		
2-Fluorobiphenyl	*321-60-8*									*15-120*		
2,4,6-Tribromophenol	*118-79-6*									*10-120*		
4-Terphenyl-d14	*1718-51-0*									*41-149*		

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TPH - Diesel Range Organics (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Glass 250ml/8oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
DRO (C10-C28)	NONE	33350	1840.92	ug/kg	60-140		60-140	20	20			
o-Terphenyl	*84-15-1*									*40-140*		

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METALS by 6020B (SOIL)

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	Holding Time	Container
Cadmium, Total	7440-43-9	0.00089	0.00089	umoles/gm	80-120	20	75-125	20	20		14 days	1 - Vial Large Septa unpreserved (4oz)
Copper, Total	7440-50-8	0.0157	0.0157	umoles/gm	80-120	20	75-125	20	20		14 days	1 - Vial Large Septa unpreserved (4oz)
Lead, Total	7439-92-1	0.00966	0.00966	umoles/gm	80-120	20	75-125	20	20		14 days	1 - Vial Large Septa unpreserved (4oz)
Nickel, Total	7440-02-0	0.0342	0.0342	umoles/gm	80-120	20	75-125	20	20		14 days	1 - Vial Large Septa unpreserved (4oz)
Zinc, Total	7440-66-6	0.0612	0.0612	umoles/gm	80-120	20	75-125	20	20		14 days	1 - Vial Large Septa unpreserved (4oz)

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METALS by 6010D (SOIL)

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	Holding Time	Container
Aluminum, Total	7429-90-5	4	1.08	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Antimony, Total	7440-36-0	2	0.152	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Arsenic, Total	7440-38-2	0.4	0.0832	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Arsenic, TCLP	7440-38-2	1	0.019	mg/l	75-125	20	75-125	20	20		180 days	1 - Glass 250ml/8oz unpreserved
Barium, TCLP	7440-39-3	0.5	0.021	mg/l	75-125	20	75-125	20	20		180 days	1 - Glass 250ml/8oz unpreserved
Barium, Total	7440-39-3	0.4	0.0696	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Beryllium, Total	7440-41-7	0.2	0.0132	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Cadmium, TCLP	7440-43-9	0.1	0.01	mg/l	75-125	20	75-125	20	20		180 days	1 - Glass 250ml/8oz unpreserved
Cadmium, Total	7440-43-9	0.4	0.0392	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Calcium, Total	7440-70-2	4	1.4	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Chromium, Total	7440-47-3	0.4	0.0384	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Chromium, TCLP	7440-47-3	0.2	0.021	mg/l	75-125	20	75-125	20	20		180 days	1 - Glass 250ml/8oz unpreserved
Cobalt, Total	7440-48-4	0.8	0.0664	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Copper, Total	7440-50-8	0.4	0.1032	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Iron, Total	7439-89-6	2	0.3612	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Lead, Total	7439-92-1	2	0.1072	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Lead, TCLP	7439-92-1	0.5	0.027	mg/l	75-125	20	75-125	20	20		180 days	1 - Glass 250ml/8oz unpreserved
Magnesium, Total	7439-95-4	4	0.616	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Manganese, Total	7439-96-5	0.4	0.0636	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Nickel, Total	7440-02-0	1	0.0968	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Potassium, Total	7440-09-7	100	5.76	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Selenium, TCLP	7782-49-2	0.5	0.035	mg/l	75-125	20	75-125	20	20		180 days	1 - Glass 250ml/8oz unpreserved
Selenium, Total	7782-49-2	0.8	0.1032	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Silver, TCLP	7440-22-4	0.1	0.028	mg/l	75-125	20	75-125	20	20		180 days	1 - Glass 250ml/8oz unpreserved
Silver, Total	7440-22-4	0.2	0.1132	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Sodium, Total	7440-23-5	80	1.26	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Thallium, Total	7440-28-0	0.8	0.126	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Vanadium, Total	7440-62-2	0.4	0.0812	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved
Zinc, Total	7440-66-6	2	0.1172	mg/kg	80-120		75-125	20	20		180 days	Metals Only-Glass 60mL/2oz unpreserved

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METALS by 7470A (SOIL)

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	Holding Time	Container
Mercury, TCLP	7439-97-6	0.000001	0.0000004575	mg/l	85-115		80-120	20	20		28 days	1 - Glass 250ml/8oz unpreserved

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METALS by 7471B (SOIL)

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria	Holding Time	Container
Mercury, Total	7439-97-6	0.08	0.05216	mg/kg	80-120		80-120	20	20		28 days	Metals Only-Glass 60mL/2oz unpreserved

Please Note that the RL information provided in this table is calculated using a 100% Solids factor (Soil/Solids only)
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Date Created: 01/24/25
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File: PM18459-1

TCL Pesticides - EPA 8081B (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Glass 250ml/8oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Delta-BHC	319-86-8	1.6008	0.31349	ug/kg	30-150	30	30-150	50	50			
Lindane	58-89-9	0.667	0.298149	ug/kg	30-150	30	30-150	50	50			
Alpha-BHC	319-84-6	0.667	0.189428	ug/kg	30-150	30	30-150	50	50			
Beta-BHC	319-85-7	1.6008	0.60697	ug/kg	30-150	30	30-150	50	50			
Heptachlor	76-44-8	0.8004	0.358846	ug/kg	30-150	30	30-150	50	50			
Aldrin	309-00-2	1.6008	0.563615	ug/kg	30-150	30	30-150	50	50			
Heptachlor epoxide	1024-57-3	3.0015	0.90045	ug/kg	30-150	30	30-150	50	50			
Endrin	72-20-8	0.667	0.27347	ug/kg	30-150	30	30-150	50	50			
Endrin aldehyde	7421-93-4	2.001	0.70035	ug/kg	30-150	30	30-150	50	50			
Endrin ketone	53494-70-5	1.6008	0.412206	ug/kg	30-150	30	30-150	50	50			
Dieldrin	60-57-1	1.0005	0.50025	ug/kg	30-150	30	30-150	50	50			
4,4'-DDE	72-55-9	1.6008	0.370185	ug/kg	30-150	30	30-150	50	50			
4,4'-DDD	72-54-8	1.6008	0.570952	ug/kg	30-150	30	30-150	50	50			
4,4'-DDT	50-29-3	1.6008	1.28731	ug/kg	30-150	30	30-150	50	50			
Endosulfan I	959-98-8	1.6008	0.378189	ug/kg	30-150	30	30-150	50	50			
Endosulfan II	33213-65-9	1.6008	0.534934	ug/kg	30-150	30	30-150	50	50			
Endosulfan sulfate	1031-07-8	0.667	0.317492	ug/kg	30-150	30	30-150	50	50			
Methoxychlor	72-43-5	3.0015	0.9338	ug/kg	30-150	30	30-150	50	50			
Toxaphene	8001-35-2	30.015	8.4042	ug/kg	30-150	30	30-150	50	50			
cis-Chlordane	5103-71-9	2.001	0.557612	ug/kg	30-150	30	30-150	50	50			
trans-Chlordane	5103-74-2	2.001	0.528264	ug/kg	30-150	30	30-150	50	50			
Chlordane	57-74-9	13.34	5.30265	ug/kg	30-150	30	30-150	50	50			
2,4,5,6-Tetrachloro-m-xylene	*877-09-8*									*30-150*		
Decachlorobiphenyl	*2051-24-3*									*30-150*		

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TCL Volatiles - EPA 8260D/5035 High&Low (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - 1 Vial MeOH/2 Vial Water

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Methylene chloride	75-09-2	5	2.29	ug/kg	70-130	30	70-130	30	30			
1,1-Dichloroethane	75-34-3	1	0.145	ug/kg	70-130	30	70-130	30	30			
Chloroform	67-66-3	1.5	0.14	ug/kg	70-130	30	70-130	30	30			
Carbon tetrachloride	56-23-5	1	0.23	ug/kg	70-130	30	70-130	30	30			
1,2-Dichloropropane	78-87-5	1	0.125	ug/kg	70-130	30	70-130	30	30			
Dibromochloromethane	124-48-1	1	0.14	ug/kg	70-130	30	70-130	30	30			
1,1,2-Trichloroethane	79-00-5	1	0.267	ug/kg	70-130	30	70-130	30	30			
Tetrachloroethene	127-18-4	0.5	0.196	ug/kg	70-130	30	70-130	30	30			
Chlorobenzene	108-90-7	0.5	0.127	ug/kg	70-130	30	70-130	30	30			
Trichlorofluoromethane	75-69-4	4	0.695	ug/kg	70-139	30	70-139	30	30			
1,2-Dichloroethane	107-06-2	1	0.257	ug/kg	70-130	30	70-130	30	30			
1,1,1-Trichloroethane	71-55-6	0.5	0.167	ug/kg	70-130	30	70-130	30	30			
Bromodichloromethane	75-27-4	0.5	0.109	ug/kg	70-130	30	70-130	30	30			
trans-1,3-Dichloropropene	10061-02-6	1	0.273	ug/kg	70-130	30	70-130	30	30			
cis-1,3-Dichloropropene	10061-01-5	0.5	0.158	ug/kg	70-130	30	70-130	30	30			
1,3-Dichloropropene, Total	542-75-6	0.5	0.158	ug/kg				30	30			
Bromoform	75-25-2	4	0.246	ug/kg	70-130	30	70-130	30	30			
1,1,2,2-Tetrachloroethane	79-34-5	0.5	0.166	ug/kg	70-130	30	70-130	30	30			
Benzene	71-43-2	0.5	0.166	ug/kg	70-130	30	70-130	30	30			
Toluene	108-88-3	1	0.543	ug/kg	70-130	30	70-130	30	30			
Ethylbenzene	100-41-4	1	0.141	ug/kg	70-130	30	70-130	30	30			
Chloromethane	74-87-3	4	0.932	ug/kg	52-130	30	52-130	30	30			
Bromomethane	74-83-9	2	0.581	ug/kg	57-147	30	57-147	30	30			
Vinyl chloride	75-01-4	1	0.335	ug/kg	67-130	30	67-130	30	30			
Chloroethane	75-00-3	2	0.452	ug/kg	50-151	30	50-151	30	30			
1,1-Dichloroethene	75-35-4	1	0.238	ug/kg	65-135	30	65-135	30	30			
trans-1,2-Dichloroethene	156-60-5	1.5	0.137	ug/kg	70-130	30	70-130	30	30			
Trichloroethene	79-01-6	0.5	0.137	ug/kg	70-130	30	70-130	30	30			
1,2-Dichlorobenzene	95-50-1	2	0.144	ug/kg	70-130	30	70-130	30	30			
1,3-Dichlorobenzene	541-73-1	2	0.148	ug/kg	70-130	30	70-130	30	30			
1,4-Dichlorobenzene	106-46-7	2	0.171	ug/kg	70-130	30	70-130	30	30			
Methyl tert butyl ether	1634-04-4	2	0.201	ug/kg	66-130	30	66-130	30	30			
p/m-Xylene	179601-23-1	2	0.56	ug/kg	70-130	30	70-130	30	30			
o-Xylene	95-47-6	1	0.291	ug/kg	70-130	30	70-130	30	30			
Xylene (Total)	1330-20-7	1	0.291	ug/kg				30	30			
cis-1,2-Dichloroethene	156-59-2	1	0.175	ug/kg	70-130	30	70-130	30	30			
1,2-Dichloroethene (total)	540-59-0	1	0.137	ug/kg				30	30			
Styrene	100-42-5	1	0.196	ug/kg	70-130	30	70-130	30	30			
Dichlorodifluoromethane	75-71-8	10	0.915	ug/kg	30-146	30	30-146	30	30			
Acetone	67-64-1	10	4.811	ug/kg	54-140	30	54-140	30	30			
Carbon disulfide	75-15-0	10	4.55	ug/kg	59-130	30	59-130	30	30			
2-Butanone	78-93-3	10	2.22	ug/kg	70-130	30	70-130	30	30			

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TCL Volatiles - EPA 8260D/5035 High&Low (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - 1 Vial MeOH/2 Vial Water

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
4-Methyl-2-pentanone	108-10-1	10	1.28	ug/kg	70-130	30	70-130	30	30			
2-Hexanone	591-78-6	10	1.18	ug/kg	70-130	30	70-130	30	30			
Bromochloromethane	74-97-5	2	0.205	ug/kg	70-130	30	70-130	30	30			
1,2-Dibromoethane	106-93-4	1	0.279	ug/kg	70-130	30	70-130	30	30			
n-Butylbenzene	104-51-8	1	0.167	ug/kg	70-130	30	70-130	30	30			
sec-Butylbenzene	135-98-8	1	0.146	ug/kg	70-130	30	70-130	30	30			
tert-Butylbenzene	98-06-6	2	0.118	ug/kg	70-130	30	70-130	30	30			
1,2-Dibromo-3-chloropropane	96-12-8	3	0.998	ug/kg	68-130	30	68-130	30	30			
Isopropylbenzene	98-82-8	1	0.109	ug/kg	70-130	30	70-130	30	30			
p-Isopropyltoluene	99-87-6	1	0.109	ug/kg	70-130	30	70-130	30	30			
n-Propylbenzene	103-65-1	1	0.171	ug/kg	70-130	30	70-130	30	30			
1,2,3-Trichlorobenzene	87-61-6	2	0.322	ug/kg	70-130	30	70-130	30	30			
1,2,4-Trichlorobenzene	120-82-1	2	0.272	ug/kg	70-130	30	70-130	30	30			
1,3,5-Trimethylbenzene	108-67-8	2	0.193	ug/kg	70-130	30	70-130	30	30			
1,2,4-Trimethylbenzene	95-63-6	2	0.334	ug/kg	70-130	30	70-130	30	30			
1,4-Dioxane	123-91-1	80	35.1	ug/kg	65-136	30	65-136	30	30			
1,2-Dichloroethane-d4	*17060-07-0*									*70-130*		
Toluene-d8	*2037-26-5*									*70-130*		
4-Bromofluorobenzene	*460-00-4*									*70-130*		
Dibromofluoromethane	*1868-53-7*									*70-130*		

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NYTCL Semivolatiles - EPA 8270E (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Glass 250ml/8oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Acenaphthene	83-32-9	133.6	17.3012	ug/kg	31-137	50	31-137	50	50			
1,2,4-Trichlorobenzene	120-82-1	167	19.1048	ug/kg	38-107	50	38-107	50	50			
Hexachlorobenzene	118-74-1	100.2	18.704	ug/kg	40-140	50	40-140	50	50			
Bis(2-chloroethyl)ether	111-44-4	150.3	22.6452	ug/kg	40-140	50	40-140	50	50			
2-Chloronaphthalene	91-58-7	167	16.5664	ug/kg	40-140	50	40-140	50	50			
1,2-Dichlorobenzene	95-50-1	167	29.9932	ug/kg	40-140	50	40-140	50	50			
1,3-Dichlorobenzene	541-73-1	167	28.724	ug/kg	40-140	50	40-140	50	50			
1,4-Dichlorobenzene	106-46-7	167	29.1582	ug/kg	28-104	50	28-104	50	50			
3,3'-Dichlorobenzidine	91-94-1	167	44.422	ug/kg	40-140	50	40-140	50	50			
2,4-Dinitrotoluene	121-14-2	167	33.4	ug/kg	40-132	50	40-132	50	50			
2,6-Dinitrotoluene	606-20-2	167	28.6572	ug/kg	40-140	50	40-140	50	50			
Fluoranthene	206-44-0	100.2	19.1716	ug/kg	40-140	50	40-140	50	50			
4-Chlorophenyl phenyl ether	7005-72-3	167	17.869	ug/kg	40-140	50	40-140	50	50			
4-Bromophenyl phenyl ether	101-55-3	167	25.4842	ug/kg	40-140	50	40-140	50	50			
Bis(2-chloroisopropyl)ether	108-60-1	200.4	28.5236	ug/kg	40-140	50	40-140	50	50			
Bis(2-chloroethoxy)methane	111-91-1	180.36	16.7334	ug/kg	40-117	50	40-117	50	50			
Hexachlorobutadiene	87-68-3	167	24.4488	ug/kg	40-140	50	40-140	50	50			
Hexachlorocyclopentadiene	77-47-4	477.62	151.302	ug/kg	40-140	50	40-140	50	50			
Hexachloroethane	67-72-1	133.6	27.0206	ug/kg	40-140	50	40-140	50	50			
Isophorone	78-59-1	150.3	21.6766	ug/kg	40-140	50	40-140	50	50			
Naphthalene	91-20-3	167	20.3406	ug/kg	40-140	50	40-140	50	50			
Nitrobenzene	98-95-3	150.3	24.716	ug/kg	40-140	50	40-140	50	50			
NitrosoDiPhenylAmine(NDPA)/DPA	86-30-6	133.6	19.0046	ug/kg	36-157	50	36-157	50	50			
n-Nitrosodi-n-propylamine	621-64-7	167	25.7848	ug/kg	32-121	50	32-121	50	50			
Bis(2-Ethylhexyl)phthalate	117-81-7	167	57.782	ug/kg	40-140	50	40-140	50	50			
Butyl benzyl phthalate	85-68-7	167	42.084	ug/kg	40-140	50	40-140	50	50			
Di-n-butylphthalate	84-74-2	167	31.6632	ug/kg	40-140	50	40-140	50	50			
Di-n-octylphthalate	117-84-0	167	56.78	ug/kg	40-140	50	40-140	50	50			
Diethyl phthalate	84-66-2	167	15.4642	ug/kg	40-140	50	40-140	50	50			
Dimethyl phthalate	131-11-3	167	35.07	ug/kg	40-140	50	40-140	50	50			
Benzo(a)anthracene	56-55-3	100.2	18.8042	ug/kg	40-140	50	40-140	50	50			
Benzo(a)pyrene	50-32-8	133.6	40.748	ug/kg	40-140	50	40-140	50	50			
Benzo(b)fluoranthene	205-99-2	100.2	28.1228	ug/kg	40-140	50	40-140	50	50			
Benzo(k)fluoranthene	207-08-9	100.2	26.72	ug/kg	40-140	50	40-140	50	50			
Chrysene	218-01-9	100.2	17.368	ug/kg	40-140	50	40-140	50	50			
Acenaphthylene	208-96-8	133.6	25.7848	ug/kg	40-140	50	40-140	50	50			
Anthracene	120-12-7	100.2	32.565	ug/kg	40-140	50	40-140	50	50			
Benzo(ghi)perylene	191-24-2	133.6	19.6392	ug/kg	40-140	50	40-140	50	50			
Fluorene	86-73-7	167	16.2324	ug/kg	40-140	50	40-140	50	50			
Phenanthrene	85-01-8	100.2	20.3072	ug/kg	40-140	50	40-140	50	50			
Dibenzo(a,h)anthracene	53-70-3	100.2	19.3052	ug/kg	40-140	50	40-140	50	50			
Indeno(1,2,3-cd)Pyrene	193-39-5	133.6	23.2798	ug/kg	40-140	50	40-140	50	50			

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NYTCL Semivolatiles - EPA 8270E (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Glass 250ml/8oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Pyrene	129-00-0	100.2	16.5998	ug/kg	35-142	50	35-142	50	50			
Biphenyl	92-52-4	380.76	21.71	ug/kg	37-127	50	37-127	50	50			
4-Chloroaniline	106-47-8	167	30.394	ug/kg	40-140	50	40-140	50	50			
2-Nitroaniline	88-74-4	167	32.1976	ug/kg	47-134	50	47-134	50	50			
3-Nitroaniline	99-09-2	167	31.4962	ug/kg	26-129	50	26-129	50	50			
4-Nitroaniline	100-01-6	167	69.138	ug/kg	41-125	50	41-125	50	50			
Dibenzofuran	132-64-9	167	15.7982	ug/kg	40-140	50	40-140	50	50			
2-Methylnaphthalene	91-57-6	200.4	20.1736	ug/kg	40-140	50	40-140	50	50			
Acetophenone	98-86-2	167	20.6746	ug/kg	14-144	50	14-144	50	50			
2,4,6-Trichlorophenol	88-06-2	100.2	31.6632	ug/kg	30-130	50	30-130	50	50			
P-Chloro-M-Cresol	59-50-7	167	24.883	ug/kg	26-103	50	26-103	50	50			
2-Chlorophenol	95-57-8	167	19.7394	ug/kg	25-102	50	25-102	50	50			
2,4-Dichlorophenol	120-83-2	150.3	26.8536	ug/kg	30-130	50	30-130	50	50			
2,4-Dimethylphenol	105-67-9	167	55.11	ug/kg	30-130	50	30-130	50	50			
2-Nitrophenol	88-75-5	360.72	62.792	ug/kg	30-130	50	30-130	50	50			
4-Nitrophenol	100-02-7	233.8	68.136	ug/kg	11-114	50	11-114	50	50			
2,4-Dinitrophenol	51-28-5	801.6	77.822	ug/kg	4-130	50	4-130	50	50			
4,6-Dinitro-o-cresol	534-52-1	434.2	80.16	ug/kg	10-130	50	10-130	50	50			
Pentachlorophenol	87-86-5	133.6	36.74	ug/kg	17-109	50	17-109	50	50			
Phenol	108-95-2	167	25.217	ug/kg	26-90	50	26-90	50	50			
2-Methylphenol	95-48-7	167	25.885	ug/kg	30-130.	50	30-130.	50	50			
3-Methylphenol/4-Methylphenol	106-44-5	240.48	26.1522	ug/kg	30-130	50	30-130	50	50			
2,4,5-Trichlorophenol	95-95-4	167	31.9972	ug/kg	30-130	50	30-130	50	50			
Benzoic Acid	65-85-0	541.08	169.004	ug/kg	10-110	50	10-110	50	50			
Benzyl Alcohol	100-51-6	167	51.102	ug/kg	40-140	50	40-140	50	50			
Carbazole	86-74-8	167	16.2324	ug/kg	54-128	50	54-128	50	50			
1,4-Dioxane	123-91-1	25.05	7.682	ug/kg	40-140	50	40-140	50	50			
2-Fluorophenol	*367-12-4*									*25-120*		
Phenol-d6	*13127-88-3*									*10-120*		
Nitrobenzene-d5	*4165-60-0*									*23-120*		
2-Fluorobiphenyl	*321-60-8*									*30-120*		
2,4,6-Tribromophenol	*118-79-6*									*10-136*		
4-Terphenyl-d14	*1718-51-0*									*18-120*		

Please Note that the RL information provided in this table is calculated using a 100% Solids factor ***(Soil/Solids only)***
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TCL PCBs - EPA 8082A (SOIL)

Holding Time: 365 days
Container/Sample Preservation: 1 - Glass 250ml/8oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Aroclor 1016	12674-11-2	0.05	0.00444	mg/kg	40-140	50	40-140	50	50			
Aroclor 1221	11104-28-2	0.05	0.00501	mg/kg	40-140	50	40-140	50	50			
Aroclor 1232	11141-16-5	0.05	0.0106	mg/kg	40-140	50	40-140	50	50			
Aroclor 1242	53469-21-9	0.05	0.00674	mg/kg	40-140	50	40-140	50	50			
Aroclor 1248	12672-29-6	0.05	0.0075	mg/kg	40-140	50	40-140	50	50			
Aroclor 1254	11097-69-1	0.05	0.00547	mg/kg	40-140	50	40-140	50	50			
Aroclor 1260	11096-82-5	0.05	0.00924	mg/kg	40-140	50	40-140	50	50			
Aroclor 1262	37324-23-5	0.05	0.00635	mg/kg	40-140	50	40-140	50	50			
Aroclor 1268	11100-14-4	0.05	0.00518	mg/kg	40-140	50	40-140	50	50			
PCBs, Total	1336-36-3	0.05	0.00444	mg/kg				50	50			
2,4,5,6-Tetrachloro-m-xylene	*877-09-8*									*30-150*		
Decachlorobiphenyl	*2051-24-3*									*30-150*		

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NY PAHs - EPA 8270E (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Glass 250ml/8oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Acenaphthene	83-32-9	0.1332	0.0172494	mg/kg	31-137	50	31-137	50	50			
2-Chloronaphthalene	91-58-7	0.1665	0.0165168	mg/kg	40-140	50	40-140	50	50			
Fluoranthene	206-44-0	0.0999	0.0191142	mg/kg	40-140	50	40-140	50	50			
Naphthalene	91-20-3	0.1665	0.0202797	mg/kg	40-140	50	40-140	50	50			
Benzo(a)anthracene	56-55-3	0.0999	0.0187479	mg/kg	40-140	50	40-140	50	50			
Benzo(a)pyrene	50-32-8	0.1332	0.040626	mg/kg	40-140	50	40-140	50	50			
Benzo(b)fluoranthene	205-99-2	0.0999	0.0280386	mg/kg	40-140	50	40-140	50	50			
Benzo(k)fluoranthene	207-08-9	0.0999	0.02664	mg/kg	40-140	50	40-140	50	50			
Chrysene	218-01-9	0.0999	0.017316	mg/kg	40-140	50	40-140	50	50			
Acenaphthylene	208-96-8	0.1332	0.0257076	mg/kg	40-140	50	40-140	50	50			
Anthracene	120-12-7	0.0999	0.0324675	mg/kg	40-140	50	40-140	50	50			
Benzo(ghi)perylene	191-24-2	0.1332	0.0195804	mg/kg	40-140	50	40-140	50	50			
Fluorene	86-73-7	0.1665	0.0161838	mg/kg	40-140	50	40-140	50	50			
Phenanthrene	85-01-8	0.0999	0.0202464	mg/kg	40-140	50	40-140	50	50			
Dibenzo(a,h)anthracene	53-70-3	0.0999	0.0192474	mg/kg	40-140	50	40-140	50	50			
Indeno(1,2,3-cd)Pyrene	193-39-5	0.1332	0.0232101	mg/kg	40-140	50	40-140	50	50			
Pyrene	129-00-0	0.0999	0.0165501	mg/kg	35-142	50	35-142	50	50			
2-Methylnaphthalene	91-57-6	0.1998	0.0201132	mg/kg	40-140	50	40-140	50	50			
Nitrobenzene-d5	*4165-60-0*									*23-120*		
2-Fluorobiphenyl	*321-60-8*									*30-120*		
4-Terphenyl-d14	*1718-51-0*									*18-120*		

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WETCHEM (SOIL)

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Method	Holding Time	Container
Cyanide, Reactive	57-12-5	10	10	mg/kg	30-125	40		40	40	7.3	14 days	1 - Glass 250ml/8oz unpreserved
Sulfide, Reactive	NONE	10	10	mg/kg	60-125	40		40	40	7.3	14 days	1 - Glass 250ml/8oz unpreserved
pH	12408-02-5	0		SU	99-101			5	5	9045D	24 hours	1 - Glass 250ml/8oz unpreserved
Cyanide, Total	57-12-5	1	0.212	mg/kg	80-120	35	75-125	35	35	9010C/9012B	14 days	1 - Glass 250ml/8oz unpreserved
Nitrogen, Ammonia	7664-41-7	7.5	2.79	mg/kg	83-115	20	55-144	20	20	4500NH3-BH	28 days	1 - Glass 250ml/8oz unpreserved
Total Organic Carbon	7440-44-0	0.01	0.01	%	75-125	25	75-125	25	25	9060A	28 days	1 - Glass 120ml/4oz unpreserved
Total Organic Carbon	7440-44-0	0.05	0.05	%	75-125	25	75-125	25	25	-	14 days	1 - Glass 120ml/4oz unpreserved
Cobbles	NONE	0.1		%				20	20	D6913/D7928	days	- Plastic 8oz unpreserved for Grain Size
% Coarse Gravel	NONE	0.1		%				20	20	D6913/D7928	days	- Plastic 8oz unpreserved for Grain Size
% Fine Gravel	NONE	0.1		%				20	20	D6913/D7928	days	- Plastic 8oz unpreserved for Grain Size
% Total Gravel	NONE	0.1		%				20	20	D6913/D7928	days	- Plastic 8oz unpreserved for Grain Size
% Coarse Sand	NONE	0.1		%				20	20	D6913/D7928	days	- Plastic 8oz unpreserved for Grain Size
% Medium Sand	NONE	0.1		%				20	20	D6913/D7928	days	- Plastic 8oz unpreserved for Grain Size
% Fine Sand	NONE	0.1		%				20	20	D6913/D7928	days	- Plastic 8oz unpreserved for Grain Size
% Total Sand	NONE	0.1		%				20	20	D6913/D7928	days	- Plastic 8oz unpreserved for Grain Size
% Silt Fine	NONE	0.1		%				20	20	D6913/D7928	days	- Plastic 8oz unpreserved for Grain Size
% Clay Fine	NONE	0.1		%				20	20	D6913/D7928	days	- Plastic 8oz unpreserved for Grain Size
% Total Fines	NONE	0.1		%				20	20	D6913/D7928	days	- Plastic 8oz unpreserved for Grain Size

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Alkylated PAHs - EPA 8270E-SIM(M) (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Glass 120ml/4oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Cis/Trans-Decalin	493-01-6/493-02-7	0.5025	0.099897	ug/kg	50-130	30	50-150	30	30			
C1-Decalins	DECALINSC1	0.5025	0.099897	ug/kg	50-130	30	50-150	30	30			
C2-Decalins	DECALINSC2	0.5025	0.099897	ug/kg	50-130	30	50-150	30	30			
C3-Decalins	DECALINSC3	0.5025	0.099897	ug/kg	50-130	30	50-150	30	30			
C4-Decalins	DECALINSC4	0.5025	0.099897	ug/kg	50-130	30	50-150	30	30			
Naphthalene	91-20-3	1.005	0.288837	ug/kg	50-130	30	50-150	30	30			
C1-Naphthalenes	91-20-3C1	1.005	0.288837	ug/kg	50-130	30	50-150	30	30			
C2-Naphthalenes	91-20-3C2	1.005	0.288837	ug/kg	50-130	30	50-150	30	30			
C3-Naphthalenes	91-20-3C3	1.005	0.288837	ug/kg	50-130	30	50-150	30	30			
C4-Naphthalenes	91-20-3C4	1.005	0.288837	ug/kg	50-130	30	50-150	30	30			
2-Methylnaphthalene	91-57-6	1.005	0.259156	ug/kg	50-130	30	50-150	30	30			
1-Methylnaphthalene	90-12-0	1.005	0.316642	ug/kg	50-130	30	50-150	30	30			
Benzothiophene	95-15-8	1.005	0.314766	ug/kg	50-130	30	50-150	30	30			
C1-Benzo(b)thiophenes	95-15-8C1	1.005	0.314766	ug/kg	50-130	30	50-150	30	30			
C2-Benzo(b)thiophenes	95-15-8C2	1.005	0.314766	ug/kg	50-130	30	50-150	30	30			
C3-Benzo(b)thiophenes	95-15-8C3	1.005	0.314766	ug/kg	50-130	30	50-150	30	30			
C4-Benzo(b)thiophenes	95-15-8C4	1.005	0.314766	ug/kg	50-130	30	50-150	30	30			
Biphenyl	92-52-4	1.005	0.310545	ug/kg	50-130	30	50-150	30	30			
2,6-Dimethylnaphthalene	581-42-0	1.005	0.238855	ug/kg	50-130	30	50-150	30	30			
Dibenzofuran	132-64-9	1.005	0.316508	ug/kg	50-130	30	50-150	30	30			
Acenaphthylene	208-96-8	1.005	0.191754	ug/kg	50-130	30	50-150	30	30			
Acenaphthene	83-32-9	1.005	0.177148	ug/kg	50-130	30	50-150	30	30			
2,3,5-Trimethylnaphthalene	2245-38-7	1.005	0.164351	ug/kg	50-130	30	50-150	30	30			
Fluorene	86-73-7	1.005	0.268067	ug/kg	50-130	30	50-150	30	30			
C1-Fluorenes	86-73-7C1	1.005	0.268067	ug/kg	50-130	30	50-150	30	30			
C2-Fluorenes	86-73-7C2	1.005	0.268067	ug/kg	50-130	30	50-150	30	30			
C3-Fluorenes	86-73-7C3	1.005	0.268067	ug/kg	50-130	30	50-150	30	30			
Dibenzothiophene	132-65-0	1.005	0.277112	ug/kg	50-130	30	50-150	30	30			
4-Methyldibenzothiophene(4MDT)	7372-88-5	1.005	0.277112	ug/kg	50-130	30	50-150	30	30			
2/3-Methyldibenzothiophene(2MDT)	20928-02-3/16587-52-3	1.005	0.277112	ug/kg	50-130	30	50-150	30	30			
1-Methyldibenzothiophene(1MDT)	31317-07-4	1.005	0.277112	ug/kg	50-130	30	50-150	30	30			
C1-Dibenzothiophenes	132-65-0C1	1.005	0.277112	ug/kg	50-130	30	50-150	30	30			
C2-Dibenzothiophenes	132-65-0C2	1.005	0.277112	ug/kg	50-130	30	50-150	30	30			
C3-Dibenzothiophenes	132-65-0C3	1.005	0.277112	ug/kg	50-130	30	50-150	30	30			
C4-Dibenzothiophenes	132-65-0C4	1.005	0.277112	ug/kg	50-130	30	50-150	30	30			
Phenanthrene	85-01-8	1.005	0.33299	ug/kg	50-130	30	50-150	30	30			
3-Methylphenanthrene (3MP)	832-71-3	1.005	0.33299	ug/kg	50-130	30	50-150	30	30			
2-Methylphenanthrene (2MP)	2531-84-2	1.005	0.33299	ug/kg	50-130	30	50-150	30	30			
2-Methylanthracene (2MA)	613-12-7	1.005	0.33299	ug/kg	50-130	30	50-150	30	30			
9/4-Methylphenanthrene (9MP)	883-20-5/832-64-4	1.005	0.33299	ug/kg	50-130	30	50-150	30	30			
1-Methylphenanthrene (1MP)	832-69-9	1.005	0.33299	ug/kg	50-130	30	50-150	30	30			
C1-Phenanthrenes/Anthracenes	PHENANTHC1	1.005	0.33299	ug/kg	50-130	30	50-150	30	30			

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Alkylated PAHs - EPA 8270E-SIM(M) (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Glass 120ml/4oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
C2-Phenanthrenes/Anthracenes	PHENANTHC2	1.005	0.33299	ug/kg	50-130	30	50-150	30	30			
C3-Phenanthrenes/Anthracenes	PHENANTHC3	1.005	0.33299	ug/kg	50-130	30	50-150	30	30			
C4-Phenanthrenes/Anthracenes	PHENANTHC4	1.005	0.33299	ug/kg	50-130	30	50-150	30	30			
Retene	483-65-8	1.005	0.24656	ug/kg	50-130	30	50-150	30	30			
Anthracene	120-12-7	1.005	0.207164	ug/kg	50-130	30	50-150	30	30			
Carbazole	86-74-8	1.005	0.328769	ug/kg	50-130	30	50-150	30	30			
1-Methylphenanthrene	832-69-9	1.005	0.265387	ug/kg	50-130	30	50-150	30	30			
Fluoranthene	206-44-0	1.005	0.319322	ug/kg	50-130	30	50-150	30	30			
Benzo(b)fluorene	243-17-4	1.005	0.291115	ug/kg	50-130	30	50-150	30	30			
7H-Benzo(c)fluorene	205-12-9	1.005	0.291115	ug/kg	50-130	30	50-150	30	30			
2-Methylpyrene	3442-78-2	1.005	0.264315	ug/kg	50-130	30	50-150	30	30			
4-Methylpyrene	3353-12-6	1.005	0.264315	ug/kg	50-130	30	50-150	30	30			
1-Methylpyrene	2381-21-7	1.005	0.264315	ug/kg	50-130	30	50-150	30	30			
Pyrene	129-00-0	1.005	0.264315	ug/kg	50-130	30	50-150	30	30			
C1-Fluoranthenes/Pyrenes	FLUORPYRC1	1.005	0.264315	ug/kg	50-130	30	50-150	30	30			
C2-Fluoranthenes/Pyrenes	FLUORPYRC2	1.005	0.264315	ug/kg	50-130	30	50-150	30	30			
C3-Fluoranthenes/Pyrenes	FLUORPYRC3	1.005	0.264315	ug/kg	50-130	30	50-150	30	30			
C4-Fluoranthenes/Pyrenes	FLUORPYRC4	1.005	0.264315	ug/kg	50-130	30	50-150	30	30			
Naphthobenzothiophene	61523-34-0	1.005	0.281199	ug/kg	50-130	30	50-150	30	30			
Naphthobenzothiophene-2,1-D		1.005	0.281199	ug/kg	50-130	30	50-150	30	30			
Naphthobenzothiophene-1,2-D		1.005	0.281199	ug/kg	50-130	30	50-150	30	30			
Naphthobenzothiophene-2,3-D		1.005	0.281199	ug/kg	50-130	30	50-150	30	30			
C1-Naphthobenzothiophenes	NAPBENZOTHIOPC1	1.005	0.281199	ug/kg	50-130	30	50-150	30	30			
C2-Naphthobenzothiophenes	NAPBENZOTHIOPC2	1.005	0.281199	ug/kg	50-130	30	50-150	30	30			
C3-Naphthobenzothiophenes	NAPBENZOTHIOPC3	1.005	0.281199	ug/kg	50-130	30	50-150	30	30			
C4-Naphthobenzothiophenes	NAPBENZOTHIOPC4	1.005	0.281199	ug/kg	50-130	30	50-150	30	30			
Benz(a)anthracene	56-55-3	1.005	0.204886	ug/kg	50-130	30	50-150	30	30			
Chrysene	218-01-9	1.005	0.203144	ug/kg	50-130	30	50-150	30	30			
Chrysene/Triphenylene	218-01-9/217-59-4	1.005	0.203144	ug/kg	50-130	30	50-150	30	30			
C1-Chrysenes	218-01-9C1	1.005	0.203144	ug/kg	50-130	30	50-150	30	30			
C2-Chrysenes	218-01-9C2	1.005	0.203144	ug/kg	50-130	30	50-150	30	30			
C3-Chrysenes	218-01-9C3	1.005	0.203144	ug/kg	50-130	30	50-150	30	30			
C4-Chrysenes	218-01-9C4	1.005	0.203144	ug/kg	50-130	30	50-150	30	30			
Benzo(b)fluoranthene	205-99-2	1.005	0.261367	ug/kg	50-130	30	50-150	30	30			
Benzo(j)+(k)Fluoranthene	205-82-3/207-08-9	1.005	0.199459	ug/kg	50-130	30	50-150	30	30			
Benzo(a)fluoranthene	203-33-8	1.005	0.199459	ug/kg	50-130	30	50-150	30	30			
Benzo(e)Pyrene	192-97-2	1.005	0.207365	ug/kg	50-130	30	50-150	30	30			
Benzo(a)pyrene	50-32-8	1.005	0.286894	ug/kg	50-130	30	50-150	30	30			
Perylene	198-55-0	1.005	0.193965	ug/kg	50-130	30	50-150	30	30			
Indeno(1,2,3-cd)Pyrene	193-39-5	1.005	0.272757	ug/kg	50-130	30	50-150	30	30			
Dibenz(a,h)+(a,c)anthracene	215-58-7/53-70-3	1.005	0.271484	ug/kg	50-130	30	50-150	30	30			
Benzo(ghi)perylene	191-24-2	1.005	0.266995	ug/kg	50-130	30	50-150	30	30			

Please Note that the RL information provided in this table is calculated using a 100% Solids factor (Soil/Solids only)
Please Note that the information provided in this table is subject to change at anytime at the discretion of Pace Analytical Services

Date Created: 02/25/25
Created By: Melissa Deyo
File: PM18775-1

Alkylated PAHs - EPA 8270E-SIM(M) (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Glass 120ml/4oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
Hopane (T19)	13849-96-2	1.005	0.286023	ug/kg	50-130	30	50-150	30	30			
Naphthalene-d8	*1146-65-2*									*50-130*		
Phenanthrene-d10	*1517-22-2*									*50-130*		
Benzo(a)pyrene-d12	*63466-71-7*									*50-130*		
5B(H)Cholane-Surr	*80373-86-0*									*50-130*		

Please Note that the RL information provided in this table is calculated using a 100% Solids factor (Soil/Solids only)
Please Note that the information provided in this table is subject to change at anytime at the discretion of Pace Analytical Services

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Page: 1

EPH - Carbon ranges only (SOIL)

Holding Time: 14 days
Container/Sample Preservation: 1 - Glass 250ml/8oz unpreserved

Analyte	CAS #	RL	MDL	Units	LCS Criteria	LCS RPD	MS Criteria	MS RPD	Duplicate RPD	Surrogate Criteria		
C9-C18 Aliphatics	C9-C18-ALPHA-UJ	6.65	6.65	mg/kg	40-140	25	40-140	50	50			
C19-C36 Aliphatics	C19-C36-ALPHA-UJ	6.65	6.65	mg/kg	40-140	25	40-140	50	50			
C11-C22 Aromatics	C11-C22-ALPHA-UJ	6.65	6.65	mg/kg	40-140	25	40-140	50	50			
C11-C22 Aromatics, Adjusted	C11-C22-ALPHA-J	6.65	6.65	mg/kg	40-140	25	40-140	50	50			
Chloro-Octadecane	*3386-33-2*									*40-140*		
o-Terphenyl	*84-15-1*									*40-140*		
2-Fluorobiphenyl	*321-60-8*									*40-140*		
2-Bromonaphthalene	*580-13-2*									*40-140*		

Please Note that the RL information provided in this table is calculated using a 100% Solids factor ***(Soil/Solids only)***
Please Note that the information provided in this table is subject to change at anytime at the discretion of Pace Analytical Services

Analysis of Parent and Alkylated Polynuclear Aromatic Hydrocarbons, Selected Heterocyclic Compounds, Steranes, Triterpanes and Triaromatic Steroids by GC / MS – SIM

References: Federal Register 2003, 40 CFR, Chapter I – EPA. Part 300: National Oil and Hazardous Substances Pollution Contingency Plan, Appendix C to Part 300, Chemical Analysis of Oil Composition, May 28, 2003.

EPA 8270E, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision VI (Phase II), June 2018.

ASTM, Method D7363 - 07 Standard Test Method for Determination of Parent and Alkyl Polycyclic Aromatics in Sediment Pore Water Using Solid-Phase Microextraction and Gas Chromatography/Mass Spectrometry in Selected Ion Monitoring Mode.

1. Scope and Application

Matrices: Extracts from water, soil, sediment, tissue, sludges and petroleum products.

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the analysis of sample extracts for parent and alkylated polynuclear aromatic hydrocarbons (PAHs), selected heterocyclic compounds, steranes, triterpanes and triaromatic steroids by gas chromatography / mass spectrometry with selected ion monitoring (GC/MS-SIM). Target analytes listed in Tables IA and IB are determined and measured in the concentration range of 10 to 20,000 parts per trillion (ng/L) for water samples, 1 to 2,000 parts per billion (ug/Kg) for soil and sediment samples, 2 to 4,000 parts per billion (ug/Kg) for tissue samples, and 2 to 4,000 parts per million (mg/Kg) in petroleum product samples. Analytes detected over these ranges will be diluted and re-analyzed for accurate quantitation. Lower detection limits can be achieved if large volume injection (LVI, from 1uL to 50uL volume injections) techniques are employed. This technique requires client and project specific requests.

This method is intended to assist in the identification or “fingerprinting” of source material against a potentially contaminated site and can also provide information to assist in the identification of petrogenic or pyrogenic contamination.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is required by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/MS and in the interpretation of GC/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

An aliquot of a well mixed, homogeneous aqueous, solid, tissue or petroleum sample is accurately measured or weighed for sample preparation (Generally, 1L of water sample, 15-30g of soil, sediment or tissue sample, and 0.1g of petroleum sample). Please refer to the appropriate Alpha Analytical SOPs for extraction methods and sample preparation information:

- Method 3510C – *Extraction of Water Samples by Separatory Funnel* (SOP/2165),
- *Tissue Preparation and Homogenization* (SOP/2166) and *Tissue Extraction* (SOP/2264)
- *Shaker Table Extraction* (SOP/2261)
- *Organic Waste Dilution* (SOP/2265)

Water, soil/sediment, tissue and petroleum samples are spiked with surrogate compounds and extracted using methylene chloride. Sample extracts are concentrated and preliminarily screened for oil content following Alpha Analytical SOP *Gravimetric Determination* (SOP/2263). Gravimetric screening is essential at times to ensure the analytical equipment, as well as the cleanup columns, are not overloaded with oil laden samples. Samples may be cleaned by *Alumina Column Cleanup* (SOP/2260), or they may then be exchanged into hexane for optional cleanup and/or fractionation into saturated (F1) and aromatic (F2) fractions prior to analysis. See the SOP *Silica Fractionation and Cleanup* (SOP/2267) for additional sample cleanup information and details. After cleanup, the extracts are concentrated to an appropriate final volume based on oil content as determined by gravimetric weighing and spiked with internal standards for GC/MS-SIM analysis.

Analytes are introduced into the GC/MS by injecting a known volume of the calibration standards, quality control samples, and sample extracts into the GC equipped with a narrow-bore capillary column. The GC column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer operating in the selective ion mode. Identification of target analytes is accomplished by comparing retention times and mass spectra with the retention times and electron impact spectra of the calibration standards. Concentrations are determined using mean relative response factors from a multi-level calibration curve. Response factors for target analytes and surrogate compounds are determined relative to the internal standards. Multi-component analytes (alkylated PAHs) are assigned the response factors of their unsubstituted, parent compounds. Sterane and Steroid compounds are assigned the response factor of the compound 5B(H)-Cholane. Triterpane compounds are assigned the response factor of the compound 17A(H), 21B(H)-Hopane-C30H52.

2.1 Method Modifications from Reference

The continuing calibration verification %D for each calibrated PAH must be below 25%, with no more than 10% of all compounds greater than 25% but less than 35%. Each CCV must be analyzed within 24 hours of the previous CCV.

The surrogate recovery limits are 50%-130%.

The duplicate RPD limit is 30%.

The PFTBA tuning is done once before each initial calibration.

The internal standard compounds used for this method are Acenaphthene-d10 and Chrysene-d12.

The confirmation ions listed in Table II are recommended and may be included or excluded based on project requirements.

3. Reporting Limits

Reporting limits for individual alkylated PAH and biomarker compounds are 2 mg/Kg (ppm) for petroleum products, 0.667and 1.0 µg/Kg (ppb) for soil/sediment samples per project requirements, 2.67 µg/Kg (ppb) for tissue samples, and 10ng/L (ppt) for water samples.

4. Interferences

Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause interferences that lead to discrete artifacts and/or elevated baselines in the ion current profiles. Demonstrate that all of these materials are free from interferences under the conditions of the preparation and analysis by extracting and analyzing a laboratory method blank with each batch of up to 20 samples.

Contaminants coextracted from the sample may cause matrix interferences. The extent of matrix interferences will vary considerably from sample to sample, depending upon the nature of the environment being investigated. An interference which is unique to SIM techniques can arise from the presence of a coeluting compound which contains the quantification mass ion. This event results in a positive interference to the reported value for the compound of interest. Interferences are controlled to some degree by acquiring data for a confirmation ion. If the ion ratios between the quantification ion and the confirmation ion are not within the specified limits, then interferences may be present. Quantification and confirmation ions should agree within +/- 50% of the calibration standard ion ratios. However, the stability of confirmatory/primary ion abundance ratios may decrease as the IDL is approached. Analysts must apply judgment in evaluating apparent interferences as SIM provides less mass spectral information compared to a full-scan method. Recommended confirmation ions are listed in Table II.

The presence of a large amount of a single alkyl homolog group without the presence of the other related groups may be indicative of an interference. For example, the presence of an apparent C_2 - Naphthalenes in the absence of C_1 - Naphthalenes or C_3 - Naphthalenes may be an analytical interference. The analyst should use the retention times of major peaks and pattern recognition when determining whether to select a homolog group.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

All relevant Material Safety Data Sheets (MSDSs) are kept alphabetically in the centrally located file storage.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in 1L or 500mL amber glass bottles with a Teflon lined cap.

Soil/sediment and tissue samples are collected in glass soil jars. For soil/sediment matrices the amount of sample needed is 5-30 grams, for tissues 5-15grams, and for petroleum products the amount of sample needed is 0.1 grams.

Additional sample is needed (approximately 3X the minimum amount) if MS/MSD analyses are to be performed.

6.2 Sample Preservation

Aqueous samples are stored without preservative at 4°C. Soil/sediment and tissue samples are stored at 4°C, or if desired, frozen.

6.3 Sample Shipping

Refer to the Sample Receipt and Login SOP/1559 for sample shipping requirements.

6.4 Sample Handling

The hold time for this method is 7 days for the extraction of aqueous samples and 14 days for the extraction of soil/sediment and tissue samples. There is no extraction holding time applied to petroleum product samples. If sediment or tissue samples are frozen, this suspends the holding time until removal from the freezer. All extracts must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph: Model Agilent/HP6890 or equivalent. The instrumentation includes a temperature-programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for split or splitless injection onto a capillary column. The injection port includes a Phenomenex drilled uniliner with a hole on the top and contains a small plug of silanized glass wool. The injector port will require maintenance on an as needed basis if degradation or contamination is apparent.

7.2 Column: Restek or Phenomenex 60-m x 0.25 mm ID, 0.25 um film thickness, fused-silica capillary column with RTX-5 or ZB-5 bonded phase, or equivalent.

7.3 Mass Spectrometer – Agilent/HP5973, or equivalent. The mass spectrometer must operate at 70ev (nominal) electron energy in the electron impact ionization mode and be tuned to optimize the sensitivity of the instrument to the mass range being monitored (30 - 550 amu). The GC capillary column is fed directly into the ion source of the mass spectrometer. The source will require cleaning and/or filament replacement on an as needed basis. Please refer to the instrument hardware manual for detailed procedures, located in the laboratory next to the instrument.

7.4 Auto sampler: Agilent/HP 7683 series autosampler and tray, or equivalent.

7.5 Computer: with Windows NT version 4.0 operating software utilizing Agilent/HP Enviroquant G1701BA Version E.02.00 software, or equivalent/higher versions.

7.6 Helium: Ultra high purity grade (99.9999% pure).

8. Reagents and Standards

Use reagent grade chemicals for all reagents. Deionized (DI) water is ASTM Type II laboratory reagent grade water.

All stock standard solutions are purchased from commercial vendors as ampulated certified solutions or prepared in house from neat materials. Standards are stored according to the vendor recommendations. When an ampulated stock solution is opened, or prepared fresh from neat materials, it must be transferred into a PTFE-lined screw capped vial. All working standard solutions are stored at -10°C or less, away from light, when not in use. They are discarded after 1 year unless the vendor expiration date states otherwise or if breakdown is observed. Expired standards are prohibited from use.

8.1 Methylene Chloride, ACS approved, Pesticide grade, see Alpha Analytical SOP Reagent, Solvent and Standard Control (SOP/1816) for additional details regarding solvent purity.

8.2 Hexane, ACS approved, Pesticide grade, see Alpha Analytical SOP Reagent, Solvent and Standard Control (SOP/1816) for additional details regarding solvent purity.

8.3 Acetone, ACS approved, Pesticide grade, see Alpha Analytical SOP Reagent, Solvent and Standard Control (SOP/1816) for additional details regarding solvent purity.

8.4 Methanol, Purge and Trap grade, see Alpha Analytical SOP Reagent, Solvent and Standard Control (SOP/1816) for additional details regarding solvent purity.

8.5 Custom Mix Calibration Standard prepared by Supelco which contains the parent PAH and Heterocyclic compounds at 1000ug/mL.

8.6 5B(H)-Cholane (CAS# 80373-86-0) and 17A(H), 21B(H)-Hopane (CAS# 13849-96-2): Obtained from Chiron AS Norway at 1000ug/mL. These compounds are used for calibration and quantitation of all steranes, triterpanes and triaromatic steroids, and 5B(H)-Cholane is also used as a surrogate compound.

8.7 Surrogates:

8.7.1 **Surrogates:** Naphthalene-d8, Phenanthrene-d10, Benzo(b)fluoranthene-d12 and Benzo(a)pyrene-d12 from AccuStandard custom mix in Methylene Chloride with a concentration of 2000ug/mL. Take 500uL of stock solution into 100mL of Methylene Chloride for a spiking solution at 10ug/mL. This solution must be assayed for use by analysis before release to the preparation lab. All compounds must be within 10% of their true value. A manager's approval is needed for all surrogate spiking solutions prior to use. 100uL of solution is spiked into each QC and field sample. This amount may be adjusted to meet project specific requirements, or if a sample is suspected to contain high concentration PAHs or oil is observed.

8.7.2 **Alternate Surrogates:** This solution may be used for some programs. 2-Methylnaphthalene-d10, Pyrene-d10, and Benzo(b)fluoranthene-d12 from Restek, custom mix in Methylene Chloride with a concentration of 2000ug/mL. Take 500uL of stock solution into 100mL of Methylene Chloride for a spiking solution at 10ug/mL. This solution must be assayed for use by analysis before release to the preparation lab. All compounds must be within 10% of their true value. A manager's approval is needed for all surrogate spiking solutions prior to use. 100uL of solution is spiked into each QC and field sample. This amount may be adjusted to meet project specific requirements, or if a sample is suspected to contain high concentration PAHs or if oil is observed.

8.7.3 Biomarker surrogate: 5B(H)-Cholane from Chiron AS Norway solution at 1000ug/mL in iso-octane. Take 1,000uL of stock solution into 100mL of Methylene Chloride for a spiking solution at 10ug/mL. This solution must be assayed for use by analysis before release to the preparation lab. All compounds must be within 10% of their true value. A manager's approval is needed for all surrogate spiking solutions prior to use. 100uL of solution is spiked into each QC and field sample. This amount may be adjusted to meet project specific requirements, or if a sample is suspected to contain high concentration PAHs or oil is observed.

8.8 Internal Standards (IS): Acenaphthene-d10 and Chrysene-d12 from Restek, custom mix in Methylene Chloride with a concentration of 2000ug/mL. Prepare a 250ug/mL intermediate solution by spiking 3125uL into 25mL Methylene Chloride. Then prepare a working solution by adding 2000uL to 100mL for a 5ug/mL solution. 100uL is spiked into each 1mL of QC sample or field sample, for a concentration of 500ng/uL on column.

8.9 Laboratory Control Spike and Matrix Spike (LCS/LCSD/MS/MSD): A solution of 17 priority pollutant parent PAH's from Restek, or equivalent, at 1000ug/mL. This solution is from a separate source than the calibration solutions. Prepare the spike mix by adding 250uL of the solution to 25mL of Methylene Chloride for a 10ug/mL LCS/LCSD/MS/MSD spiking solution. The solution must be assayed for use by analysis before release to the preparation lab. All compounds must be within 10% of their true value. 100uL is spiked into the LCS/LCSD and each designated MS/MSD field sample. This amount may be adjusted to meet project specific concentrations, as needed.

8.10 Alaska North Slope Crude Oil (ANS): Weigh approximately 0.5g neat oil, add 10mL each PAH and biomarker surrogates, and 200uL internal standard stock solution into 100mL of Methylene Chloride for a working solution of approximately 5mg/mL (with surrogates at 1.0µg/mL and internals at 0.5µg/mL).

8.11 Independent Calibration Verification (ICV): Prepared as below in Section 8.13 at 0.5ug/mL, but from a different source, lot, or vendor. Independent Check analyses must agree within 20% of their true value.

8.12 SRM 1941b, 1944 – PAH's in sediment, SRM 1974a – PAH's in Tissue and SRM 1582 PAHs' in Crude Oil: From the National Institute of Standards & Technology (NIST). Please refer to the individual certifications for the assigned true values. These SRMs may be extracted and analyzed with sample batches as part of the overall QC evaluation if requested by the client. Other certified SRMs may be used on a project specific basis.

8.13 Working Stock Standard: Prepare the Working Stock Standard (all resulting concentrations at 5µg/mL) in 50mL of Methylene Chloride (CH_2Cl_2) as follows. (**Note:** The following is just one way an analyst may make up calibration standards. Limitations may exist that would cause the method to be adjusted. Problems with standard availability, solubility, or expiration may affect how the following 6 level calibration standards are prepared.)

Component	Volume Added	Final Volume in (CH_3Cl_2)
Custom Supelco Mix (PAH)	250 µL of 1000 µg/mL	50 mL
5B(H)-Cholane	250 µL of 1000 µg/mL	50 mL
Custom Deuterated Surr Mix	125 µL of 2000 µg/mL	50 mL

7 Level Curve Preparation for Individual Components

Calibration Level	Volume of Working Std. Added (5 ug/mL)	Volume of IS Stock added	Final Volume in (CH_3Cl_2)
Level 1 - 10 ng/mL	200 µL	200 uL	100 mL
Level 2 - 25 ng/mL	500 µL	200 uL	100 mL
Level 3 - 100 ng/mL	2000 µL	200 uL	100 mL

Calibration Level	Volume of Custom PAH mix Std. Added (1000 ug/mL)	Volume of IS Stock added	Final Volume in (CH_3Cl_2)
Level 4 – 500 ng/mL	25 µL	100 uL	50 mL
Level 5 - 5,000 ng/mL	125 uL	50 uL	25 mL
Level 6 - 10,000 ng/mL	250 uL	50 uL	25 mL
Level 7 - 20,000 ng/mL	500 uL	50 uL	25 mL

Note: A minimum of a 5-level curve must be analyzed, but more levels may be analyzed and evaluated.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Quality Control (QC) samples are necessary to monitor both the sample extraction and instrument analysis procedures. The Quality Control samples described below are considered the method defaults, and are the minimum requirements, except where noted. Client and Project specific Data Quality Objectives (DQOs) supersede the requirements in this section where applicable. Client or Project specified DQOs shall be included, or referenced, in the final report to the client.

9.1 Blank(s)

A method blank must be extracted (spiked with surrogates and internal standards) and analyzed once per every 20 samples or per extraction batch, whichever is more frequent.

Acceptance Criteria: Method Blanks must not contain any individual compound at or above the concentration of the reporting limit. If a blank does contain target compounds greater than the reporting limit, they should be less than 10% of any sample results for the same compound(s). Individual compounds may be detected in the blank below the reporting limit. Associated sample results are flagged with a “B” qualifier if the concentration of the analyte in the sample is less than 10x the concentration in the blank.

Corrective Action: If the blank does not meet the above criteria, all efforts must be made to identify and eliminate any source of contamination, and all samples associated with the

contaminated blank should be re-extracted and reanalyzed. Exceptions may be made with the approval of the Department Supervisor. Any exceedances are noted in the case narrative.

9.2 Laboratory Control Sample and Laboratory Control Sample Duplicate (LCS/LCSD)

The laboratory control samples (LCS/LCSD) contain 17 priority pollutant parent PAHs. The spiking solution is from a second/separate source, to verify the accuracy of the calibration curve. The LCS/LCSD are extracted along with the samples. An LCS/LCSD must be extracted and analyzed once per every 20 samples or per extraction batch, whichever is more frequent.

Acceptance Criteria: All LCS/LCSD compound recoveries must be between 50-130% of the true values, with <30%RPD.

Corrective Action: If the LCS/LCSD do not meet the QC limits, check to see if an analytical or spiking error has occurred. If the LCS/LCSD recoveries are still out of control, re-extraction of the entire extraction set may be necessary. If the samples are also associated with a matrix spike and matrix spike duplicate that are in control, re-extraction may not be necessary, as this demonstrates an isolated problem pertaining to the LCS/LCSD only. Exceptions may be made with the approval of the Department Supervisor. Any exceedances are noted in the case narrative.

9.3 Initial Calibration Verification (ICV)

The analysis of an ICV standard must follow the initial calibration curve.

After final processing, calculate the percent recovery of each PAH by using the following calculation:

% Recovery = Found Amount / True Value x 100

Acceptance Criteria: All recoveries must be +/- 20% of the true values.

Corrective Action: If the ICV fails, perform instrument maintenance and repeat the initial calibration..

9.4 Continuing Calibration Verification (CCV)

A continuing calibration verification (CCV) standard, at the concentration of a mid-level initial calibration standard, must be analyzed at the beginning and end of every analytical sequence, and every 24 hours within the sequence, to confirm instrument stability, via response factor, for each calibrated PAH. Only the parent PAH compounds are monitored for %D and the following acceptance criteria.

Acceptance Criteria: Compare the CCV response factor(RF) against the average RF for the initial calibration for each calibrated PAH. The %D for each calibrated PAH must be below 25%, with no more than 10% of all compounds greater than 25% but less than 35%. If multiple CCVs are analyzed within an analytical sequence, each CCV must be analyzed within 24 hours of the previous CCV. Each CCV, including the ending CCV must meet the acceptance criteria.

Corrective Action: If the CCV does not meet the acceptance criteria for each calibrated PAH, perform instrument maintenance and repeat the continuing calibration. Re-analyze all affected samples. Reported results for the failing CCV may be “J” qualified; including all alkylated compounds quantified using the suspect response. If the failure of the suspect response

appears related to a loss in MS sensitivity, instrument maintenance and repeat analysis of the continuing calibration, and all affected samples, must be performed. Exceptions may be made with the approval of the Department Supervisor. Any exceedances are noted in the case narrative.

9.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Matrix spike and matrix spike duplicate analyses are performed per client request and may only be performed if sufficient sample is supplied. It is preferable to extract samples that have been selected specifically by the client. If none have been assigned than the laboratory analyst must choose a representative sample.

Acceptance Criteria: All matrix spike/matrix spike duplicate compound recoveries must be between 50-150% of the true values, with <30%RPD.

Corrective Action: If the MS/MSD do not meet the QC limits, check to see if an analytical or spiking error has occurred. If the recovery still exceeds the control limits, re-extraction of the set may be necessary. If the associated LCS/LCSD is within control, re-extraction may not be necessary, as this demonstrates that there may be matrix effects on the accuracy of the affected results as evidenced by the matrix spike and matrix spike duplicate exceedance. Exceptions may be made with the approval of the Department Supervisor. Any exceedances are noted in the case narrative.

9.6 Sample Duplicate

A sample duplicate (DUP) is extracted per client request.

Acceptance Criteria: The QC limit is 30% RPD for target compounds found above 5 times the reporting limit.

Corrective Action: If the %RPD exceeds the 30% control limit, checkto see if an analytical or spiking error has occurred. Also check sample preparation notes for heterogeneity comments or for possible calculation errors. If the RPD still exceeds the control limits, re-extraction of the set may be necessary. If the associated LCS/LCSD is within control, re-extraction may not be necessary, as this demonstrates that there may be matrix effects on the precision of the affected results as evidenced by the sample and duplicate exceedance. Exceptions may be made with the approval of the Department Supervisor. Any exceedances are noted in the case narrative.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

Acceptance Criteria: All surrogate recoveries must be between 50-130% of the true values.

Corrective Action: If the surrogate does not fall within the QC limits, check to see if an analytical or spiking error has occurred. If only one surrogate falls below the 50% recovery limit, but is above 10% recovery, re-extraction may not be necessary. If all surrogates are recovered below the 50% limit, re-extract the sample and report the re-extract results along with the original results, if re-extraction occurred beyond the holding time, and the re-extract surrogates are within the QC limits. If the surrogates are recovered below 50% in the re-extract, this confirms suspected matrix interference on the surrogates, and only the original analysis needs to be reported. If the chromatogram shows obvious matrix interference, no re-analysis or re-extraction is necessary.

9.7.2 Internal Standards

Internal standards are added to every field sample, QC sample, and method blank.

Acceptance Criteria: The acceptance limits are 50-200% of the internal standard response (or area) of the daily continuing calibration verification standard.

Corrective Action: If the internal standard areas fall outside the QC limits, check to see if an analytical, dilution or spiking error occurred.

- If internal standards are low and surrogate recoveries are acceptable reanalyze the extract.
- If internal standards are low and surrogate recoveries are high, there may have been a spiking error; re-spike a pre-internal standard extract with internals and re-analyze.
- If internal standards are high and surrogate recoveries are low, there may have been a spiking error; re-spike a pre-internal standard extract with internals and re-analyze.
- If internal standards are high and surrogate recoveries are acceptable, the extract may have concentrated while on the instrument, then:
    - If no obvious interference is present, re-analyze the extract. If internal standards are now within the acceptance limits, report only the re-analysis, as long as the re-analysis occurred within the 40-day analytical hold time. If the re-analysis occurred outside of the 40-day analytical hold time, both the original and re-analysis must be reported. If the internal standards again are outside the acceptance limits, and either within or outside of the 40-day hold time, try re-analyzing at a 1:5 or greater dilution (see below).
    - If the chromatogram shows obvious matrix interference that cannot be avoided when integrating, a re-analysis at a 1:5 or greater dilution may be helpful in minimizing the interference while ensuring better quantitation.

Exceptions may be made with the approval of the Department Supervisor. Any exceedances are noted in the case narrative.

9.7.3 Standard Reference Materials (SRMs)

Standard reference materials (SRM) are available from the National Institute of Standards and Technology (NIST) and are extracted and analyzed with samples on a project specific basis. These are not used as controls, but to evaluate potential matrix effects in associated samples for the target compounds being evaluated.

Acceptance Criteria: Acceptance criteria for SRM analysis will vary from project to project depending upon client data quality objectives (DQOs). Generally, ± 35% difference (%D) based on the true certified values of the target compounds of interest, or 65% - 135% recovery, serve as advisory acceptance criteria.

Corrective Action: Repeat analysis and/or check to see if an analytical error has occurred. If the % recovery or %D still exceeds the control limits, re-extraction of the set may be necessary. If the associated LCS/LCSD and/or MS/MSD are

within control, re–extraction may not be necessary as this may be isolated to this sample as evidenced by the LCS/LCSD and/or MS/MSD acceptance. Exceptions may be made with the approval of the Department Supervisor. Any exceedances are noted in the case narrative.

9.8 Method Sequence

Samples are prioritized for analysis by the Department Supervisor or GC/MS Team Leader based on client due date and sample analytical hold time. Samples are retrieved from the sample storage refrigerator.

The sequence is prepared and run using the Enviroquant software. Printouts of all sequences are kept in a three-ring binder for each instrument. The sequence printouts are used to document analytical runs; notations are manually added of any reruns or dilutions that will need to be performed.

10. Procedure

10.1 Equipment Set-up

Prior to the analysis of any standards or samples, the instrument acquisition and process methods must be set up. This includes the GC run parameters and the SIM mode acquisition ion entries into the different SIM acquisition retention time windows. The mass spectrometer must be tuned to the meet the abundance criteria for PFTBA (then DFTPP if required per client request or project specific DQOs) and an initial calibration must be analyzed to establish linearity of the instrument.

10.1.1 PFTBA Manual Tuning

10.1.1.1 Prior to initial calibration, tune the mass spectrometer using PFTBA (Perfluorotributylamine - calibration gas) to maximize the sensitivity of the instrument in the mass range of interest, 35-525 amu.

10.1.1.2 The following PFTBA mass intensity criteria must be met:

PFTBA Ion	Relative Abundance
m/e 69	Base Peak with > 200,000 counts
m/e 219	30% to 60% of Base Peak
m/e 502	5% to 11% of Base Peak

10.1.2 GC Instrumental Conditions

Inject an aliquot of 1uL into the capillary column of the gas chromatograph at the following conditions. Larger injection volumes (to 50uL using the Large Volume Injector, LVI) will be dictated by project specific DQOs.

GC Parameter	Setting
Injector Temp:	300 °C
Transfer Line Temp:	300 °C
Initial Oven Temp:	40 °C
Initial Hold Time:	1 minute
Ramp Rate:	6 °C / minute
Final Temperature:	315 °C
Final Hold Time:	30 minutes
Total runtime:	76 minutes
Mode:	Splitless / Constant Flow 1.0ml/min
Purge:	20 mL / minute – on at 1.0 minutes
MS Temperature	300

10.1.3 Mass Spectrometer Conditions

The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the SIM mode using appropriate retention time windows to include the quantification and confirmation ions for each PAH and Biomarker compound as shown in Table II. For each retention time window the ions 191, 217, and 218 are included for sterane and triterpane quantification, if requested by the client.

10.1.4 Data Acquisition Parameters

10.1.4.1 SIM Windows must be set up that bracket the expected retention times for each target analyte. These windows include the quantitation (primary) and confirmation ions for each parent PAH and Alkyl homolog group. To establish the expected retention time window ranges, the mid-level calibration standard must be analyzed in full scan mode. The resulting full scan analysis will dictate the windows in which the selected ions will be monitored. Depending upon the length of the analytical GC column, the time each window is selectively monitored may vary. The retention time windows must be shifted accordingly, when instrument maintenance is performed, (*i.e.,* the column is clipped).

10.1.4.2 The “dwell” time for each window should be set to 18, and the resolution should be set to “high”. Each window must contain a total of 28 ions, and must contain the appropriate quantification ions, and if possible, confirmation ions, as determined by full scan analysis of the mid-level standard from 10.1.4.1, Specific projects may require the addition of ions to one or more SIM windows; in these instances it is acceptable to remove confirmation ions as necessary to keep the total number of ions in each window at 28 and the dwell time per ion at 18.

10.2 Initial Calibration

10.2.1 Before analysis of sample extracts, establish a multi-point response factor calibration curve showing the linear range of the analysis for all target analytes in Table IA and IB. Use standard concentrations of 10, 25, 100, 500, 5000, 10,000 and 20,000 ng/mL to construct the curve. See Section 8.13 for the preparation of the standard solutions for the initial calibration curve.

10.2.2 Run a sequence with the initial calibration standards and the retention time standard.

- Create a processing method from a previous method on that instrument. Set retention time windows using a mid level standard.

- Quantify and QEDIT the initial calibration standards. Update the response factors for each level of the method with these standards. Use each parent PAH compound response for any associated homolog group. Use hopane response for associated triterpane biomarkers and 5B(H)cholane response for associated sterane and triaromatic steroid biomarkers.

  Acceptance Criteria: 25% RSD for 90% of all target compounds, with the exception for 10% to be between 25%RSD and 35%RSD. All calibration standards must be analyzed within 24 hours. An alternative calculation may be employed by the use of linearity calibraton model as long as the correlation coefficient is >0.995 and orgin is NOT forced through 0.

10.2.3 Initial Calibration Verification (ICV)

- The analysis of an ICV standard must follow the initial calibration curve.

- After final processing, calculate the percent recovery of each PAH by using the following calculation:

  % Recovery = Found Amount / True Value x 100

- Acceptance Criteria: All recoveries must be +/- 20% of the true values.

10.2.4 If the initial calibration fails, perform instrument maintenance and repeat.

10.2.5 Alaska North Slope Crude Reference Oil (ANS)

- The ANS crude oil reference standard is analyzed following each initial calibration curve. Analysis of this reference oil is to establish the integration patterns of the Alkyl PAH homolog groups, and to establish the current instrument quantitation and confirmation ion ratios. Analysis of this standard following the initial calibration ensures the retention time windows in the SIM mode of acquisition have been set up properly.

- The analysis of the ANS standard is for reference. If instrument maintenance is performed, such as removing a significant section of the analytical column consisting of one “loop” or more, the ANS must be analyzed to update and/or confirm the SIM acquisition windows.

- The ANS reference standard is processed against the completed initial calibration curve method. See Section 11.1 for information regarding manual integration of the

Alkyl PAH homolog clusters. This standard will be used for reference when processing field samples for this method, until such a time that a new ANS standard needs to be analyzed. Other project/client specific source oils may be used. These specific oils will likely display patterns that differ from ANS, but will aid analysts in pattern identification of the related field samples.

10.2.6 ICALs are documented on the *Alkylated PAH ICAL Checklist (Form/11443)* . The initial calibration must be secondary reviewed before analyzing samples.

10.3 Equipment Operation and Sample Processing

10.3.1 Daily PFTBA Tuning is not required. See Section 10.1.1 for details.

10.3.2 If the on-column concentration of any single peak PAH compound exceeds the calibration range of 20,000 ng/mL, the sample must be diluted and re-analyzed.

10.4 Continuing Calibration

A continuing calibration verification (CCV) standard, at the concentration of a mid-level initial calibration standard, must be analyzed at the beginning and end of every analytical sequence, and every 24 hours within the sequence, to confirm instrument stability, via response factor, for each calibrated PAH.

10.4.1 Quantitate and QEDIT the continuing calibration standard. **Note:** The Alkyl homolog groups may be deleted from the report. Only the parent PAH compounds are monitored for %D and the following acceptance criteria.

10.4.2 Acceptance Criteria: Compare the CCV response factor(RF) against the average RF for the initial calibration for each calibrated PAH. The %D for each calibrated PAH must be below 25%, with no more than 10% of all compounds greater than 25% but less than 35%. If multiple CCVs are analyzed within an analytical sequence, each CCV must be analyzed within 24 hours of the previous CCV. Each CCV, including the ending CCV must meet the acceptance criteria.

10.4.3 If the CCV does not meet the criteria for each calibrated PAH, the following *corrective actions* are recommended:

- Perform instrument maintenance and repeat the continuing calibration, and re-analyze all affected samples, OR,
- Qualify all results reported for the failing CCV with an appropriate qualifier, including all alkylated compounds quantified using the suspect response, and any non-detects. If the failure of the suspect response appears related to a loss in MS sensitivity, instrument maintenance and repeat analysis of the continuing calibration, and all affected samples, must be performed.

The choice of corrective action must be made in consultation with the Department Manager, Project Manager and/or the client. The reasoning for choosing the second option must be documented in the project narrative to the client.

10.5 Preventive Maintenance

If performing any maintenance on any piece of equipment it must be documented in the *Instrument Maintenance Logbook* located in the laboratory specific to each instrument. Specific instrumentation service contracts or warranties differ from each instrument. See the Department Manager for specific instrument details.

Injection Port and Column Maintenance: Maintenance should be done when the daily CCV starts to demonstrate degradation. The type of samples analyzed will have an effect on how

soon maintenance should be performed. The injection port can be cleaned using cotton swabs and DCM until no contamination is observed. Generally maintenance is performed by trimming 6 cm off the front of the column. The column is then installed into the injection port liner and the inlet nut is tightened. After cleaning the instrument must be checked for any air leaks while cool. The first injection should be a primer to remove any active sites and to check if any of the windows need updating.

Merlin Maintenance: The Merlin High Pressure Microseal (Part #410 Phenomenex) should be replaced when it cannot hold back pressure. A septum may be used as a backup or for troubleshooting.

11. Data Evaluation, Calculations and Reporting

11.1 Identification of the priority pollutant PAH compounds is based on gas chromatographic relative retention times (RRTs) from the analysis of a mid-level initial calibration standard. For these compounds, manual quantitations may be performed, if necessary, by integrating the area of the quantitation ion or peak. For *alkylated* PAHs, the homolog groupings (*i.e.,* C_3-Naphthalenes) appear in the extracted ion current profiles (EICPs) as a cluster of isomers. Integrate peaks within the cluster by straight-line integration to the baseline, taking into account background noise in the EICPs. Reference the Alaska North Slope Crude Oil pattern book, for a cluster by cluster example of each integration for each alkylated PAH homolog group. Table II, in Section 16, lists the representative ion(s) used for quantitation and confirmation of each parent PAH and alkylated PAH homolog group.

> **Note:** Manual integration is not to be used solely to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system.

11.2 From EICP of the quantification (primary) mass ions and the confirmatory mass ions, identify all target analytes according to the following criteria:

- The characteristic masses of each analyte of interest should maximize in the same, or within one scan of each other.
- The retention time should fall within ± 10 seconds of the retention time of the parent PAH from the preceding CCV. **Note:** When evaluating alkyl homolog groups, the retention time of the most intense peak within the group may not have the exact retention time of the most intense peak in the ANS reference standard. Analyst judgement and referral to each homolog groups' retention time window is essential for identification. Apply analyst judgment regarding corrective action when this criterion is not met.
- The relative peak heights of the primary ion compared to the confirmation or secondary ion masses for parent compounds should fall within ± 50 percent of the relative intensities of these masses in the reference mass spectrum (*i.e.,* the mid-level of the initial calibration curve and/or the Alaska North Slope Crude Oil).

Note: The relative intensities of the primary and secondary ions may vary widely within a given group of alkyl homologs (*i.e.,* C_3 - Naphthalenes). Thus, the pattern of each alkyl homolog cluster, and the retention time window for the cluster, will be the primary identification criteria for alkyl homologs. In some instances, a parent compound that does not meet secondary ion confirmation criteria may still be determined to be present in a sample after close inspection of the data by the experienced mass spectrometrist. Supportive data includes the presence of the secondary ion, but ratio value greater than ± 50 percent of the primary ion, may be caused by an interference of the secondary ion.

11.3 To calculate the ***Relative Standard Deviation*** (RSD) of all target analytes and surrogate compounds for the initial calibration use the formula below. The RSD of each target compound and surrogate must be below 25% with the exception for 10% to be between 25%RSD and 35%RSD. Additionally, use the initial seven-point calibration and determine ***Relative Response Factors*** (RRF_Is) at each concentration level. Average the RRF_Is, to generate mean RRF_Is, for quantification of all target analytes and surrogate compounds. The RRF_Is are based on the internal standard compounds, and are calculated using the formula below. (The relative response factors for the continuing calibration verifications (RRF_Cs) are calculated using the same formula). See Section 16, Table IA and Table IB, for the listing of target compounds and their associated internal standards for quantification.

$$\mathbf{RSD = SD / mean\ RRF_I \times 100}$$

where:

SD = Standard deviation between the five points, for that target analyte.

$$\mathbf{RRF_I = (A_c \times C_{IS}) / (A_{IS} \times C_c)}$$

where:

A_c = Area of the characteristic ion for the standard compound to be measured.
A_{IS} = Area of the characteristic ion for the representative internal standard compound.
C_{IS} = Concentration of the representative internal standard compound (ng/mL).
C_c = Concentration of the standard compound to be measured (ng/mL).

Note: Assign the response factor of the parent compound to the alkyl homolog cluster.

11.4 Based on the mean RRF_Is, calculate the ***Sample Extract Amount*** for each target analyte and surrogate in the extracts using the following formula:

$$\mathbf{Q_e = (A_a \times Q_{IS}) / (A_{IS} \times RRF_I)}$$

where:

Q_e = Sample extract amount (ng) of target analyte, from quantitation report.
A_a = Area of the characteristic ion for the target analyte.
A_{IS} = Area of the characteristic ion for the representative internal standard compound.
Q_{IS} = Amount of internal standard compound added to each extract (ng).

11.5 Calculate the ***Sample Concentration*** (C) for each compound by the following formula:

$$\mathbf{C = (Q_e / V_s) \times DF}$$

where:

C = Concentration in sample (ng/L water, ug/Kg sediment/tissue, or mg/Kg product).

V_s = Original volume or weight of sample extracted.

DF = Dilution factor or fraction of the original extract to which internal standard added.

11.6 If the response of any individual target compound in a sample exceeds the linear response range, as defined by the initial calibration standards in Section 10.2.1, dilute the extract so that the concentrations of all individual target compounds fall within the range of the calibration curve. If that compound is also part of an alkyl homolog group, the group is considered to exceed the calibration range and thus, would also require dilution. Reported concentrations that are above the highest standard concentration in the initial calibration are qualified with an "E". If the response of any target compound in a sample exceeds the MDL but is below the reporting limit (RL), qualify the reported concentration with a "J". If any target compound is found in the associated sample(s) at or below 10x the concentration of the method blank, qualify the reported concentration with a "B".

11.7 Compare response factors for each compound in the ***Continuing Calibration Verification*** (CCV), to those of the initial calibration curve by determining the percent difference.

$$\textbf{Percent Difference (\%D)} = ([RRF_I - RRF_C] / RRF_I) \times 100$$

where:

RRF_I = Mean response factor from initial calibration.

RRF_C = Response factor from CCV.

11.8 All results must be reported to three significant figures. All solids including soils, sediments, and sludges must be reported on a dry-weight basis. Tissue results may be reported in wet-weight depending upon client request. Petroleum results are reported "as received" or on a wet-weight basis.

11.9 The analyst does data entry, or upload of the data, into the LIMS system. The LIMS is linked to the instrument, so the analyst must choose the sample(s) to be reported from that instrument's analytical sequence. All associated preparation and instrumental QC samples and dilutions are also chosen. Once the data/samples have been selected and with the proper QC samples, the batched data set is reported.

11.10 The laboratory generates two types of data packages from the LIMS: "Standard" for routine projects and "CLP-like" for fully data validated projects. A standard package consists of sample results and the associated QC sample results. A CLP-like package includes all sample results, all preparation and instrumental QC results, and the associated supporting raw data. A secondary review is performed on all data.

11.11 Procedures for data and record management must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP (1729). All records shall be stored in such a manner as to be safe and accessible for at least 10 years.

11.12 Notebooks: Laboratory notebooks are designed to accommodate the specific analysis. Instrument printouts are used to document run sequences, and each sequence printout is filed in a three-ring binder. Each sequence notebook page is numbered. If a sample requires re-analysis or re-extraction for any reason, a notation is made next to the sample entry on the sequence log. The sequence run log is permanently bound, assigned an internal ID number, and filed accordingly. Such files shall be archived so as to remain available for at least 10 years. All laboratory notebooks must follow the specifications in the *Laboratory Notebook Usage* Work Instructions, WI 1556, and all record keeping and document control practices.

11.13 Electronic records: All data files from computers, attached to instruments, shall be backed up daily onto the proper directory on the server. The backups shall be stored so as to be accessible for 10 years. Movement of the data files to the server is the responsibility of the primary analyst. Server backup and storage is the responsibility of the IT department.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All Alkylated PAH-SIM results are reportable without qualification if analytical holding times are met, preservation requirements (including cooler temperatures) are met, and all QC criteria defined in the table below are met. If any of the below QC parameters are not met, all associated samples must be evaluated for re-analysis or re-extraction. See Sections 9, 10.2, 10.3 and 10.4 for additional QC discussion including corrective actions for any QC outliers.

QC Parameter	Acceptance Criteria
Initial Calibration Curve	≤ 25%D for all target analytes with exception for 10% of target analytes can be >25%, but ≤ 35%
Independent Calibration Verification	+/- 20% recovery of the true values
Continuing Calibration Verification	Analyzed every 10 samples or 24 hours, ≤ 25%D for all target analytes with exception for 10% of target analytes can be >25%, but ≤ 35%
Method Blank	No analyte at or above the RL, “B” qualify analyte if detected in the samples ≤ 10x the concentration in the blank
Laboratory Control Sample/Laboratory Control Sample Duplicate	50-130%R for all target analytes, <30%RPD between duplicates
Matrix Spike / Matrix Spike Duplicate	50-150%R for all target analytes, <30% RPD between the duplicates.
Sample / Sample Duplicate	<30% RPD between the duplicates, for compounds >5X RL.
Surrogates	50% - 130% recovery
Internal Standards	50% - 200% of the daily CCV area for the Internal Standards
SRM	+/- 35% D or 65% - 135% recovery

Section 9, Quality Control, defines the corrective actions that must be taken in instances where QC outliers exist.

If non-compliant Alkylated PAH-SIM results are to be reported, the Department Manager and/or the Laboratory Director must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

Hazardous Waste and Sample Disposal SOP/1797

SOP/1729 Document Control

Logbook Useage Work Instructions, WI/1556

16. Attachments

Table IA: PAH, Alkyl PAH, and Heterocyclic Target Compounds Internal Standard Reference

Table IB: Sterane and Triterpane Target Compounds Internal Standard Reference

Table II: PAH, Alkyl PAH, Sterane and Triterpane Quantification Primary and Confirmation Ions

Table IA: PAH, Alkyl PAH, and Heterocyclic Target Compounds Internal Standard Reference

Compound	IS Reference	Compound	IS Reference
cis/trans-Decalin	1	Fluoranthene	1
C_1-Decalins	1	Benzo(b)fluorene	1
C_2-Decalins	1	Pyrene	1
C_3-Decalins	1	C_1-Fluoranthrenes/Pyrenes	1
C_4-Decalins	1	C_2-Fluoranthrenes/Pyrenes	1
Naphthalene	1	C_3-Fluoranthrenes/Pyrenes	1
C_1-Naphthalenes	1	C_4-Fluoranthrenes/Pyrenes	1
C_2-Naphthalenes	1	Naphthobenzothiophenes	1
C_3-Naphthalenes	1	C_1-Naphthobenzothiophenes	1
C_4-Naphthalenes	1	C_2-Naphthobenzothiophenes	1
2-Methlynaphthalene	1	C_3-Naphthobenzothiophenes	1
1-Methlynaphthalene	1	C4-Naphthobenzothiophenes	1
Benzothiophene	1	Benz[a]anthracene	2
C_1-Benzo(b)thiophenes	1	Chrysene/Triphenylene	2
C_2-Benzo(b)thiophenes	1	C_1-Chrysenes	2
C_3-Benzo(b)thiophenes	1	C_2-Chrysenes	2
C_4-Benzo(b)thiophenes	1	C_3-Chrysenes	2
Biphenyl	1	C_4-Chrysenes	2
2,6-Dimethylnaphthalene	1	Benzo[b]fluoranthene	2
Dibenzofuran	1	Benzo[j]+[k]fluoranthene	2
Acenaphthylene	1	Benzo[a]fluoranthene	2
Acenaphthene	1	Benzo[e]pyrene	2
2,3,5-Trimethylnaphthalene	1	Benzo[a]pyrene	2
Fluorene	1	Perylene	2
C_1-Fluorenes	1	Indeno[1,2,3-c,d]pyrene	2
C_2-Fluorenes	1	Dibenz[ah]+[ac]anthracene	2
C_3-Fluorenes	1	Benzo[g,h,i]perylene	2
Dibenzothiophene	1	C_1-Dibenzothiophene isomers	1
C_1-Dibenzothiophenes	1	C_1-Phenanthrenes/Anthracene isomers	1
C_2-Dibenzothiophenes	1	C1-Fluoranthenes/Pyrenes isomers	
C_3-Dibenzothiophenes	1	**Surrogate Compounds**	
C_4-Dibenzothiophenes	1	Phenanthrene-d_{10}	1
Phenanthrene	1	Benzo(b)flouranthene-d12	2
C_1-Phenanthrenes/Anthracenes	1	Benzo(a)pyrene-d_{12}	2
C_2-Phenanthrenes/Anthracenes	1	5B(H) - Cholane	2
C_3-Phenanthrenes/Anthracenes	1	Methylnaphthalene-d10	1
C_4-Phenanthrenes/Anthracenes	1	Pyrene-d10	1
Anthracene	1	Naphthalene-d8	1
Retene	1	**Internal Standards**	
Carbazole	1	Acenapthene-d_{10}	1
1-Methylphenanthrene	1	Chrysene-d_{12}	2

Table IB: Sterane and Triterpane Target Compounds Internal Standard Reference

Compound	IS Reference	Compound	IS Reference
C23 Tricyclic Terpane	2	30,31-Trishomohopane-22S	2
C24 Tricyclic Terpane	2	30,31-Trishomohopane-22R	2
C25 Tricyclic Terpane	2	Tetrakishomohopane-22S	2
C24 Tetracyclic Terpane	2	Tetrakishomohopane-22R	2
C26 Tricyclic Terpane-22S	2	Pentakishomohopane-22S	2
C26 Tricyclic Terpane-22R	2	Pentakishomohopane-22R	2
C28 Tricyclic Terpane-22S	2	13b(H),17a(H)-20S-Diacholestane	2
C28 Tricyclic Terpane-22R	2	13b(H),17a(H)-20R-Diacholestane	2
C29 Tricyclic Terpane-22S	2	13b,17a-20S-Methyldiacholestane	2
C29 Tricyclic Terpane-22R	2	14a(H),17a(H)-20S-Cholestane/ 13b(H),17a(H)-20S-Ethyldiacholestane	2
18a-22,29,30-Trisnorneohopane-TS	2	14a(H),17a(H)-20R-Cholestane/ 13b(H),17a(H)-20R-Ethyldiacholestane	2
C30 Tricyclic Terpane-22S	2	Unknown sterane	2
C30 Tricyclic Terpane-22R	2	13a,17b-20S-Ethyldiacholestane	2
17a(H)-22,29,30-Trisnorhopane-TM	2	14a,17a-20S-Methylcholestane	2
17a/b,21b/a 28,30-Bisnorhopane	2	14a,17a-20R-Methylcholestane	2
17a(H),21B(H)-25-Norhopane	2	14a(H),17a(H)-20S-Ethylcholestane	2
30-Norhopane	2	14a(H),17a(H)-20R-Ethylcholestane	2
18a(H)-30-Norneohopane-C29Ts	2	14b(H),17b(H)-20R-Cholestane	2
17a(H)-Diahopane	2	14b(H),17b(H)-20S-Cholestane	2
30-Normoretane	2	14b,17b-20R-Methylcholestane	2
18a(H)&18b(H)-Oleananes	2	14b,17b-20S-Methylcholestane	2
17a(H),21B(H)-hopane-C30H52 or Hopane	2	14b(H),17b(H)-20R-Ethylcholestane	2
Moretane	2	14b(H),17b(H)-20S-Ethylcholestane	2
30-Homohopane-22S	2	C26,20R- +C27,20S- triaromatic steroid	2
30-Homohopane-22R	2	C28,20S-triaromatic steroid	2
Gammacerane/C32-diahopane	2	C27,20R-triaromatic steroid	2
30,31-Bishomohopane-22S	2	C28,20R-triaromatic steroid	2
30,31-Bishomohopane-22R	2		

Table II: PAH, Alkyl PAH, Sterane and Triterpane Quantification Primary and Recommended Confirmation Ions

Compound	Quantification & Confirmation Ions	Compound	Quantification & Confirmation Ions
cis/trans-Decalin	138, 96	Fluoranthene	202, 101
C_1-Decalins	152	Benzo(b)fluorene	216, 215
C_2-Decalins	166	Pyrene	202, 101
C_3-Decalins	180	C_1-Fluoranthrenes/Pyrenes	216, 215
C_4-Decalins	194	C_2-Fluoranthrenes/Pyrenes	230, 215
Naphthalene	128, 127	C_3-Fluoranthrenes/Pyrenes	244, 229
C_1-Naphthalenes	142, 141	C_4-Fluoranthrenes/Pyrenes	258
C_2-Naphthalenes	156, 141	Naphthobenzothiophene	234, 189
C_3-Naphthalenes	170, 155	C_1-Naphthobenzothiophenes	248
C_4-Naphthalenes	184, 169, 183	C_2-Naphthobenzothiophenes	262
2-Methylnaphthalene	142, 141	C_3-Naphthobenzothiophenes	276
		C4-Naphthobenzothiophenes	290
1- Methylnaphthalene	142, 141	Benz[a]anthracene	228, 226
Benzothiophene	134	Chrysene/Triphenylene	228, 226
C1-Benzo(b)thiophenes	148	C_1-Chrysenes	242, 241
C2-Benzo(b)thiophenes	162	C_2-Chrysenes	256, 241
C3-Benzo(b)thiophenes	176	C_3-Chrysenes	270, 255
C4-Benzo(b)thiophenes	190	C_4-Chrysenes	284, 269
Biphenyl	154, 153	Benzo[b]fluoranthene	252, 253
2,6-Dimethylnaphthalene	156, 155	Benzo[j]+[k]fluoranthene	252, 253
Dibenzofuran	168, 139, 169	Benzo[a]fluoranthene	252, 253
Acenaphthylene	152, 153	Benzo[e]pyrene	252, 253
Acenaphthene	153, 154	Benzo[a]pyrene	252, 253
2,3,5-Trimethylnaphthalene	170, 155	Perylene	252, 253
Fluorene	166, 165	Indeno[1,2,3-c,d]pyrene	276, 138, 277
C_1-Fluorenes	180, 165	Dibenz[ah]+[ac]anthracene	278, 139, 279
C_2-Fluorenes	194, 179,195	Benzo[g,h,i]perylene	276, 277
C_3-Fluorenes	208, 197	7H-Benzo(c)fluorene	216, 215
		C_1-Fluoranthrenes/Pyrenes isomers	216, 215
Dibenzothiophene	184, 152	**Biomarker Compounds**	
C_1-Dibenzothiophene isomers	198, 197	17a(H),21B(H)-hopane-C30H52 or Hopane	191, 177
C_1-Dibenzothiophenes	198, 197	Triterpanes	191
C_2-Dibenzothiophenes	212, 197	Steranes	217, 218
C_3-Dibenzothiophenes	226, 211	Triaromatic steroids	231
C4-Dibenzothiophenes	240, 225	**Surrogate Compounds**	
Phenanthrene	178, 176	2-Methylnaphthalene-d_{10}	152, 150
C_1-Phenanthrenes/Anthracene isomers	192, 191	Pyrene-d_{10}	212, 211
C_1-Phenanthrenes/Anthracenes	192, 191	Benzo(b)fluoranthene-d_{12}	264, 260

C_2-Phenanthrenes/Anthracenes	206, 191, 207	Naphthalene-d_8	136, 134
C_3-Phenanthrenes/Anthracenes	220, 205	Phenanthrene-d_{10}	188, 184
C_4-Phenanthrenes/Anthracenes	234, 219	Benzo(a)pyrene-d_{12}	264, 260
Anthracene	178, 176	5B(H) - cholane	217, 218
Retene	234, 219	**Internal Standards**	
Carbazole	167, 139	Acenapthene-d_{10}	164, 162
1-Methylphenanthrene	192, 191	Chrysene-d_{12}	240, 241

1,4-Dioxane

By Gas Chromatography / Mass Spectrometry in Selected Ion Mode (GC/MS-SIM) with Isotope Dilution Modification

References:

EPA 8270E, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision VI (Phase II), June 2018.

EPA 8000C, SW-846,Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846. Update III, March 2003.

1,4-Dioxane Analytical Notes, Appendix II-B-4, WSC-CAM-II-B, Revision 1, July 2010, (Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup).

1. Scope and Application

Matrices: Aqueous, soil/sediment and non-aqueous waste matrices

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the quantification of 1,4-Dioxane extracted from aqueous samples in methylene chloride and analyzed by GC/MS-SIM. The extraction method is listed below and should be referenced for more details. Detection limits will vary with instrument calibration range, and volume of sample analyzed. 1,4-Dioxane detected over the calibration ranges of the instrument it is being analyzed on will be diluted and re-analyzed for accurate quantification.

The following extraction method applies:

- *Extraction of Water Samples by Separatory Funnel* (SOP 2165)
- *Microscale Solvent Extraction (MSE)* (SOP 2172)
- *Organic Waste Dilution Extraction* (SOP 2265)

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/MS-SIM and in the interpretation of GC/MS-SIM data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

250mL, 500mL or 1000mL of aqueous samples are serially extracted with methylene chloride in a 2 Liter Separatory Funnel (Method 3510) at a neutral pH. The extract is concentrated in MeCl2 to a 2.5mL to 10mL final volume respectively depending on the volume of sample extracted. Soil/sediment samples are extracted by Microscale Solvent Extraction (MSE Method 3570). Approximately 5g of sample is extracted and concentrated to a 4mL final volume. Non-aqueous waste samples are extracted by Organic Waste Dilution Extraction (Method 3580). Approximately 1g is diluted to 10mL

final volume.

Analytes are introduced into the GC/MS using a large volume injector and injecting 3ul of the calibration standards, quality control samples, and sample extracts into the GC equipped with a narrow-bore capillary column. The GC column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer (MS) in selective ion mode. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of the calibration standards. Concentrations are determined using mean relative response factors from a multi-level calibration curve. Response factors for target analytes and surrogate compounds are determined relative to the internal standards.

Isotope dilution quantification is achieved by spiking 1,4-Dioxane-d8 at extraction which is then in turn used as both an internal standard (IS) and surrogate. For quantification, the 1,4-Dioxane-d8 IS quantifies 1,4-Dioxane in samples. An additional IS added prior to analysis, 1,4-Dichlorobenzene-d4, quantifies 1,4-Dioxane-d8 as a surrogate.

2.1 Method Modifications from Reference

SIM option with Isotope dilution is utilized to increase sensitivity for this analyte. Note that while this method may be used for the evaluation of 1,4-Dioxane in soil/sediment samples, the MA DEP CAM does not approve of this method for determining presumptive certainty for MA DEP cleanup sites. Method 8260 should be employed in these cases.

3. Reporting Limits

Concentrations for 1,4-Dioxane can be detected in water samples in the range of 150ng/L to 100,000ng/L. Soil/sediment samples can be detected in the range of 8ug/Kg to 8,000ug/Kg. Standard reporting limits for aqueous samples are 150ng/L and for soil/sediment samples 8ug/kg respectively.

4. Interferences

4.1 Phthalate esters can be a major source of contamination if any material containing plasticizers (phthalates) comes in contact with the sample during the extraction process. Use of plastic or any material containing plasticizers (phthalates) should be avoided during extraction or analysis.

4.2 The injection port of the gas chromatograph can become contaminated with high boiling compounds resulting in the loss of sensitivity. It may be necessary to replace the injection port liner routinely to prevent this loss of sensitivity. Clipping off approximately four inches of the column at the injection end may also increase sensitivity. Low instrument response can be detected during the daily tuning procedure by including pentachlorophenol and benzidine in the daily tuning mix.

4.3 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

4.4 Solvents, reagents and glassware may introduce interferences. These must be demonstrated to be free of interferences by the analysis of a method blank. See the SOP *Reagent, Solvent and Standard Control* (G-008) and *Laboratory Glassware Cleaning* (G-002), for additional details.

4.5 It should be noted that there are some chromatographic consequences observed due to the acetone used in the MSE extraction process. This is characterized by a shift in retention time

for 1,4-Dioxane and the 1,4-Dioxane-d8 surrogate (~0.4minutes), as well as the presence of a large peak (likely acetone) in all chromatograms.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Solid samples; A minimum of 100grams of sample must be collected in a glass jar with a Teflon lined screw cap.

Water samples: A minimum of 0.25 to 1 liters of sample must be collected in amber glass bottles.

6.2 Sample Preservation

Solid samples: The sample must be refrigerated and maintained at 4±2 °C until extraction and analysis. Sediment samples can be frozen at -20±5 °C until extraction to extend hold time. The extracts must be refrigerated and maintained at 4±2 °C until analysis.

Water samples: The samples must not be preserved except by refrigeration at 4±2 °C until extraction and analysis. The extracts must be refrigerated and maintained at 4±2 °C until analysis.

Non-aqueous waste samples: Concentrated sample extracts must be stored in contaminant-free containers and preserved in a refrigerator when not used for more than four hours.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Solid samples: All solid samples must be extracted within 14 days from the date of collection. Frozen sample hold times are monitored up to 14 days from the date removed from freezer. The extracts must be refrigerated and maintained at 4±2 °C until analysis. Sample extracts must be analyzed within 40 days from date of extraction.

Water samples: All water samples must be extracted within 7 days from the date of collection. Sample extracts must be analyzed within 40 days from date of extraction.

Non-aqueous waste samples: Hold times do not apply to neat oils/NAPL/product samples.

7. Equipment and Supplies

7.1 Gas chromatograph – Programmable, heating range from 40C to 350C; splitless-type inlet system, (Hewlett Packard 6890N Series II or similar); mass selective detector (Hewlett Packard 5973, or similar); automatic injector (Hewlett Packard 7683B or similar).

7.2 Chromatography Column – Fused silica capillary column, 0.25mm ID x 60m length, 0.25um film thickness RTX-5, Restek Corporation, 5% diphenyl-95% dimethyl polysiloxane, Fused silica capillary column, 0.18mm ID x 60m length, 0.18um film thickness RTX-PCB, Restek Corporation, Fused silica capillary column, 0.25mm ID x 30m length, .25um film thickness (Zebron ZB-SemiVolatiles, Phenomenex Corporation, 5% Polysilarylene - 95% Polydimethylsiloxane) , or equivalent.

7.3 Gerstel Large Volume Injection System – Temperature programmable range from 0C to 350C; pressure programmable; capable of split or splitless injection; Injection volumes range from 1 to 50uL and Cryo cooling availability to allow for cold injections.

7.4 Agilent Split/Splitless injector System

7.5 Data Acquisition System - Computerized system for collecting, storing, and processing detector output (Hewlett Packard Enviroquant target software) or equivalent.

7.6 Gases - BIP Ultra high purity helium (99.9995%); Compressed nitrogen for N-Evap. Carbon dioxide (siphon type) for Gerstel.

7.7 Syringes – 10uL to 1.0mL

7.8 Vials- including 2ml, 4ml, 10ml, 40ml and other sizes as necessary.

7.9 Hamilton Gas tight Syringes - varying sizes

7.10 Gerstel Single baffle injection port liners- packed lightly with glass wool.

7.11 GC Injection Port Liner: Phenomenex Direct Connect Top Hole

7.12 Class A Volumetric flasks: Including 10ml, 20 ml, 50 ml, 100ml and other sizes as necessary

8. Reagents and Standards

Use reagent grade chemicals for all reagents. Deionized (DI) water is ASTM Type II laboratory reagent grade water.

8.1 Solvents: All solvent expirations determined as indicated by manufacturer guidelines

8.1.1 Methylene Chloride, ACS approved, Pesticide grade, see SOP *Reagent, Solvent and Standard Control* (SOP 1816) for additional details regarding solvent purity. Used to extract samples and prepare instrument/analytical standards.

8.1.2 Acetone, ACS approved, Pesticide grade, see SOP *Reagent, Solvent and Standard Control* (SOP 1816) for additional details regarding solvent purity. This water soluble solvent is used for surrogate and LCS/MS preparation.

8.1.3 Methanol, ACS approved, Pesticide grade, *Reagent, Solvent and Standard Control SOP* (SOP 1816) for additional details regarding solvent purity.

8.2 Analytical Standards: Standards should be stored at –10C or less, away from light when not in use. They should be discarded after 1 year unless the vendor expiration date states otherwise or, if degradation is observed. Stock standards are given a 1 year expiration from the preparation date or the expiration of the primary vendor solution, whichever occurs first. Working standards are given six month expiration from the preparation date or the expiration of the primary solution whichever occurs first. All analytical standards are made up in Methylene Chloride. All prep standards are made up in Acetone.

8.3 Surrogate/Internal Standard (IS):

8.3.1 A 1,4-Dioxane-d8 Primary neat standard is commercially obtained from Cambridge Isotope (Cat #DLM-28-10 or equivalent). A stock surrogate/internal standard solution is prepared by weighing 0.1 g of the primary neat standard and diluting volumetrically in 10 mls methylene chloride (or equivalent preparation) to obtain a concentration of ~10,000 ug/ml.

8.3.2 From this stock (Section 8.3.1), the Surrogate spiking solution is made by a serial dilution (1 ml diluted up in a 50 ml volumetric flask, followed by a 0.5 ml of this solution diluted up in a 20 ml volumetric flask or equivalent preparations) in Acetone to achieve a concentration of 5 ug/mL. Of this surrogate solution, 1 mL is spiked into each water sample, and 0.4 mL is spiked into each soil/sediment sample for a surrogate/IS concentration of 500 ng/mL in samples.

8.3.3 The initial stock solution (Section 8.3.1) is diluted volumetrically (250 uL diluted up in a 100 ml volumetric flask or equivalent preparation) in methylene chloride to obtain an IS solution at a concentration of 25 ug/ml for the spiking of only the analytical standards (i.e. calibration curve and continuing calibration).

8.4 Internal Standard: 1,4-Dichlorobenzene-d4 commercially obtained from Restek (Cat #31206 or equivalent). This primary solution is at 2000 ug/mL and contains other Semivolatile Internal standards, however this method only utilizes the 1,4-Dichlorobenzene-d4. This solution is diluted volumetrically (250 uL diluted up in a 50 ml volumetric flask or equivalent preparation) in methylene chloride to obtain a SIM-IS solution at a concentration of 25 ug/ml. All samples and standards are spiked with 20uL of internal standard before analysis. This IS is intended to be used for both quantitation (of the surrogate 1,4-Dioxane-d8) and the establishment of relative retention times.

8.5 Matrix Spike/Laboratory Control Spike Solutions (MS/LCS) – 1,4-Dioxane primary spike solution is commercially obtained from Restek at 2000 ug/mL (Cat #31853 or equivalent). To prepare the working spike solution, syringe measure 0.25 mL of stock and bring to 100 mL in acetone (or equivalent preparation) for a 5 ug/mL concentration. From this solution, 1 mL is spiked into each MS/LCS QC water sample and 0.4 mL is spiked into each soil/sediment sample. The final concentration in a 10 mL water extract and in a 4 mL soil/sediment sample will be 500 ng/mL.

8.6 Stock Calibration Standards are prepared from a primary standard commercially obtained from Restek at a concentration of 2000 ug/ml (Cat #31853 or equivalent). The primary standard is used to appropriately prepare a stock standard at a concentration of 10,000 ng/ml (125 uL diluted up in a 25 ml volumetric flask or equivalent preparation). The stock solution is generally made up in 25 ml increments. The stock solution then doubles as a high level standard for the calibration curve, as well as a stock solution by which to serially dilute to prepare the other calibration standards. Except for the high level standard, the calibration curve levels are generally each made at 10 ml. Refer to the table below for example calibration curve levels. All the calibration curve levels, once aliquoted at 1 ml increments for injection, have 20 ul of 1,4

Dichlorobenzene IS (SIM IS – Section 8.4) added as well as 20 uL of 1,4 Dioxane-d8 IS/Surrogate (Section 8.3.3) added.

8.7 Curve Preparation: The following 7 levels are the possible levels that can be analyzed for an ICAL.

Calibration Level	*Volume of Stock Std Added Into 10 ml of DCM*
Level 1 (10 ng/mL)	10 uL
Level 2 (50 ng/mL)	50 uL
Level 3 (100 ng/mL)	100 uL
Level 4 (500 ng/mL)	500 uL
Level 5 (1000 ng/mL) - CCV	1.0 mL
Level 6 (5000 ng/mL)	5.0 mL
Level 7 (10,000 ng/mL)	NA

8.8 The Independent Check Verification Standard is prepared from a Primary standard solution commercially obtained from Ultra (Cat NV-150-1 or equivalent) at a concentration of 100 ug/ml. The primary solution is diluted volumetrically (100 ul diluted up in a 10 ml volumetric flask or equivalent preparation) in methylene chloride to create a working ICV solution at a concentration of 1000 ng/ml. The working ICV solution is aliquoted into 1 ml increments as needed to inject with each calibration curve. Additionally, 20 ul of SIM IS (Section 8.4) and 20 ul of 1,4-Dioxane-d8 IS (Section 8.3.3) is added into the 1 ml aliquot.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A method blank must be prepared once per every 20 samples or per extraction batch, whichever is more frequent.

Organic compounds of interest must not be detectable in the method blank at a concentration greater than the reporting limit.

Corrective Action: For contaminated blanks, all efforts must be made to identify and eliminate the source of contamination. The presence of analytes at concentrations at or above the reporting limit will warrant application of a “B” qualifier to that target compound(s) on all associated report forms, and perhaps re-extraction of all associated samples. Re-extraction of the method blank and all associated samples must be performed until the blank is in control. Surrogate recoveries must meet the QC limits for the method blank. Re-extraction must be initiated immediately so that minimum time is wasted before re-extraction can occur - if at all possible-this re-extraction should take place within holding time. Re-extraction *corrective action* that would exceed the sample holding time criteria should be discussed with the Organics Supervisor, Project Manager, client, and Operations Manager prior to implementation. Exceptions may be made with approval of the Organics Section Supervisor if the samples associated with an out of control method blank are non-detect for the affected compound(s) or if the concentration of the affected compound(s)in the sample is greater than 10x the blank level. In such cases, the sample results are accepted without corrective action for the high method blank result. The client must be notified, via the project narrative, of any method blank non-compliance associated with sample results

9.2 Laboratory Control Sample/Laboratory Control Sample Duplicate (LCS/LCSD)

Laboratory control samples (LCS/LCSD) must be prepared once per every 20 samples or per extraction batch, whichever is more frequent, and spiked with 1,4 Dioxane spike solution (Section 8.5) and surrogate (Section 8.3.2) before extraction. The IS (Section 8.4) is spiked after extraction and before the analysis.

Acceptable Recovery limits are 40% - 140%. The relative percent difference (RPD) between the LCS/LCSD is 30%. Limits are adapted from MCP protocol and are generally monitored and documented in-house through control charts.

Corrective Action: Analysis must be repeated if an analytical error is suspected. If the LCS/LCSD recoveries and/or %RPD are still out of control, re-extract and re-analyze the LCS/LCSD and all associated samples. Samples cannot be reported until an acceptable LCS is obtained.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

Matrix spike / matrix spike duplicate (MS/MSD) samples are performed upon project specifications. They are performed per client request. The sample is spiked with 1,4-Dioxane spike solution (Section 8.5) and surrogate (Section 8.3.2) before extraction. The IS (Section 8.4) is spiked after extraction and before the analysis. The recovery limits are 40% - 140% and the RPD limit is 30%. Limits are adapted from MCP protocol and are generally monitored and documented in-house through control charts.

Corrective Action: Analysis must be repeated if an analytical error is suspected. If the % recovery and/or %RPD still exceeds the control limits and the LCS/LCSD is compliant; include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the reported results as evidenced by MS/MSD recoveries and/or %RPD outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses are performed upon client and/or workplan request. *For Organic analyses, the matrix duplicate is usually in the form of the matrix spike duplicate, see Section 9.5.*

Acceptable relative percent difference (RPD) of duplicates is 30%. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate the RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:

R1 = sample Replicate #1
R2 = sample Replicate #2

The RPD limits should be monitored and documented in-house through control charts and updated as needed.

Corrective Action: Analysis must be repeated if an analytical error is suspected.. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the reported results as evidenced by the matrix duplicate % RPD exceedence.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

Surrogate spikes (Section 8.3.2 and Section 8.3.3) must be added to QC and field samples to evaluate the extraction method performance.

The acceptable surrogate recovery limits are 15% - 110%. Limits are adapted from MCP protocol.

Corrective Action: Analysis must be repeated if an analytical error is suspected. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm the sample matrix. If *obvious* matrix interferences are noted, consultation with the Organic Supervisor or Operations Manager may be in order to confirm the need for sample re-extraction. If no re-extraction occurs, the surrogate results and reasons for the decision not to re-extract must be discussed in the project narrative to the client. Due to the isotope dilution nature of this method, the analyst must pay close attention to surrogate recoveries and areas as this recovery is then used to surrogate correct (as an Internal standard) the response of 1,4-Dioxane.

9.7.2 Internal Standards

Internal standards must be added to all sample extracts, QC samples and standards for quantitation purposes. For sample extracts, only the 1,4-Dichlorbenzene-d4 IS (SIM IS – Section 8.4) should be added since the extraction surrogate is then used as an Internal Standard upon analysis. However, all Calibration standards must be spiked with both 1,4-Dichlorobenzene-d4 as well as 1,4-Dioxane-d8 (Section 8.3.3). In the sample extracts the area counts for 1,4-Dioxane-d8 will vary based on the extraction, however the 1,4-Dichlorobenzene-d4 area counts should remain fairly constant. The 1,4-Dichlorobenzene-d4 internal standard should also remain constant with respect to the continuing calibration analyzed at the beginning of the run. Sample IS areas must be –50% to +100% of the Internal Standards in the Continuing Calibration – applies only to 1,4-Dichlorobenzene-d4. Additionally, the IS retention times should not differ more than 30 seconds from the Continuing Calibration.

Corrective Action: Analysis must be repeated once unless there are obvious samples matrix interferences, i.e., the sample extract was very colored and viscous, or there are obvious chromatographic interferences. If *obvious* matrix interferences are noted, consultation with the Organic Supervisor or Operations Manager may be in order to confirm the need for sample re-analysis or re-extraction.

9.8 Method Sequence

- Tune
- CCV
- Method Blank
- LCS
- LCSD
- Samples

10. Procedure

10.1 Equipment Set-up

10.1.1 The instrument used for the analysis is a HP 6890N Series gas chromatograph. The HP system is equipped with a Gerstel large volume injection system, and a 7683B-type autosampler or equivalent. The mass spectrometer is an HP 5973 or 5975 with the HP Enviroquant data system. The method is modified for selective ion monitoring. The table below lists the ions monitored in one SIM window. This method must only be set up and analyzed by an experienced mass spectrometrist.

Compound	*Primary Ion*	*Secondary Ion*
1,4-Dioxane	88	58, 43
1,4-Dioxane-d8	64	96
1,4-Dichlorobenzene-d4	152	115

10.1.2 The basic GC parameters are as follows for the Gerstel Large Volume Injection System:

Oven Equib Time: 0.10 min
Oven Max: 325
Initial Temp.: 45°C
Initial Time: 6.00 min

Level	Rate (°C/min)	Final Temp. (°C)	Final Time (min)
1	19.00	120	1.0
2	11.00	150	1.00
3	19.00	305	4.00

Final Time: 26.83

10.1.3 The basic injection port parameters are as follows for the Gerstel Large Volume Injection System:

"Splitless" mode
Initial Temp: 46 °C
Initial Time: 0.30 min
Ramp Rate: 300°/second
Final Temp: 300°C
Final Time: 30.0
Cryo: ON
Cryo Use temp: 25 °C
Cryo Timeout: 30.0 min
Cryo Fault: ON
Purge Flow: 25 ml/min

Purge Time: 2.50 min
Gas Saver: off
Gas: Helium

Mode: Constant Pressure
Average Velocity: 30cm/sec
Initial flow: 1.3 ml/min

10.1.4 The basic GC parameters are as follows for the Agilent Split/Splitless injector System:

Oven Equib Time: 0.20 min
Oven Max: 325°C
Initial Temp.: 30°C

Level	Rate (°C/min)	Final Temp. (°C)	Final Time (min)
1	0	30	2.00
2	5	50	0.00
3	50	300	2.00

Final Time: 13.00

10.1.5 The basic injection port parameters are as follows for the Agilent Split/Splitless injector System:

:

Mode: Splitless
Temperatuire: 200 °C
Flow: 1.0 mL/min
Velocity: 36.074 cm/sec
Septum Purge: 3mL/min
Purge Flow to Split Vent: 60 mL/min at 0.3 min

10.1.6 MS Acquisition Information:

Tune file: dftpp.u
Acquisition Mode: SIM
Solvent Delay: 7.70 min
MS Source temp: 280°C

10.1.7 Tuning

10.1.7.1 Before the analytical standards are analyzed the mass spectrometer must be adjusted to meet the proper ion criteria for DFTPP. This is demonstrated by injecting into the GC/MS system 1uL of a 50ug/mL DFTPP solution. After the analysis of the DFTPP, evaluate the tune as follows:

- Enter into the “Environmental Data Analysis” (off-line) screen.
- Go to “File” and select the tune data file.
- Go into “Tuner” and select “Eval DFTPP”, then select “AutoFind DFTPP to Screen,” to evaluate the tune file, based on the pre-set SW-846 criteria**.** The software will evaluate the tune by selecting three scans of the DFTPP peak

and will display the ion intensities on the screen. That is, one scan at the apex, one scan directly preceding the apex and one scan following the apex and averages them, then takes one background subtracted scan, 20 seconds before the beginning of the DFTPP peak. If the criteria below are met, repeat, select "AutoFind to Printer", for a hardcopy of the tune evaluation for the record.

If the "AutoFind" tune evaluation does not meet the criteria below, manual evaluation of the tune can be performed by attempting either of the options below:

- Blow up the DFTPP peak on the screen and select either one single scan at the apex of the peak, or a scan immediately preceding or following the apex. Go into "Tuner" and select "Evaluate DFTPP to Screen," or "Evaluate DFTPP to Printer," as described above, OR,
- Take the average of the scans across the entire peak. Go into "Tuner" and select "Evaluate DFTPP to Screen," or "Evaluate DFTPP to Printer," as described above.

10.1.7.2 The following DFTPP mass intensity criteria should be used.

DFTPP KEY MASSES AND ABUNDANCE CRITERIA

Mass	m/z Abundance criteria
51	10-80 percent of mass 198.
68	Less than 2 percent of mass 69.
70	Less than 2 percent of mass 69.
127	10-80 percent of mass 198.
197	Less than 2 percent of mass 198.
198	Base peak, or >50 percent of Mass 442.
199	5-9 percent of mass 198.
275	10-60 percent of mass 198.
365	Greater than 1 percent of mass 198.
441	Present but less than 24 percent of mass 442.
442	Base Peak, or > 50 percent of mass 198.
443	15-24 percent of mass 442.

10.1.8 Tune acceptance should be verified at the beginning of every 12 hour analytical shift. The DFTPP may be combined with the calibration verification standard as long as both tuning and calibration acceptance criteria are met.

10.2 Initial Calibration

10.2.1 After the DFTPP passes criteria, a set of multi-level calibration standards listed in Section 8.7 are analyzed, from low concentration to high. A minimum of five calibration levels are analyzed. The calibration standards are stored in amber vials in the standards freezer. The labeling convention allows each standard to have a unique identifier which distinguishes it from field samples. The naming convention used throughout the laboratory identifies the standard as semivolatile, hydrocarbon, pesticide/PCB or volatile. An example of this would be SW042407E, meaning it is a semivolatile (S) working (W) standard made on April 24, 2007 and that it was the fifth standard made that day. All certificates of analysis that are shipped with standards are filed with their receipt ID written on it to insure traceability.

10.2.2 Once the standards have been analyzed, they are reduced by the search software of the Enviroquant data system. Once all the components are identified, a linear curve is calculated for the components. The criteria for evaluation are as follows:

10.2.2.1 The average RF for each compound must be greater than 0.05.

10.2.2.2 The %RSD of each compound must not exceed 20%. If they do this may be an indication that the chromatographic system is too reactive for analysis to begin. This indicates the instrument may need maintenance.

10.2.2.3 Alternatively, a linear regression model may be employed, provided that the coefficient of determination (COD or r2) is ≥0.99. Otherwise, construct a nonlinear calibration of no more than a third order equation. Statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approach. A quadratic (second order) model requires six standards, and a third order polynomial requires seven standards. In setting model parameters, do not force the line through the origin. The COD or r^2 must be greater than or equal to 0.99.

10.2.2.4 Once the calibration curve is reviewed, an Initial Calibration Checklist must be completed.

10.2.3 All samples and standards are spiked with Internal Standards (IS) before analysis. Refer to section 8.4 for specific internal standard spiking information. The IS is intended to be used for both quantitation and the establishment of relative retention times. Internal standard acceptance criteria can be found in Section 9.7.2.

10.2.4 Independent check standards (Section 8.8) from a separate source or different lot are analyzed after every initial calibration for evaluation against calibration standard solutions. The % Difference (%D) should not be greater than ±30%.

10.3 Equipment Operation and Sample Processing

10.3.1 Tuning

A DFTPP standard must be analyzed and pass criteria before a continuing calibration verification standard or any samples are analyzed. The DFTPP may be combined with the calibration verification standard as long as both tuning and calibration acceptance criteria are met. A DFTPP tune standard must be analyzed before each 12 hour analytical shift. Please refer to Section 10.1.5 for tuning criteria and other information.

10.3.2 Daily Calibration

On a daily basis after the DFTPP has passed, a mid-level (usually 1000 ng/mL) continuing calibration standard which contains all of the analytes of interest is analyzed. The criteria for acceptance are:

10.3.2.1 All analytes must have response factors greater than 0.05.

10.3.2.2 The % D must be ±20% D from the initial calibration.

10.3.3 Sample Analysis

10.3.3.1 The prep lab staff will transfer the samples to the instrument laboratory. The samples are generally brought to a 10 mL final volume for liquids or 4 ml for soil samples; 1 mL is transferred and the remaining sample volume is archived. One aliquot of each sample is then placed in the sample extract holding refrigerator located in the instrument laboratory.

10.3.3.2 All of the samples at 1 mL (including the batch QC samples) are spiked with 20 uL

internal standard (see section 8.4 for specifics regarding the internal standard). The samples are shaken briefly after the internal standard is added to ensure mixing. A sample will need a dilution for target analytes that are over calibration.

10.3.3.3 After the daily tune and CCAL have passed criteria, the analyst places the samples onto the autosampler tray. (Generally, the samples will be analyzed in order of color (lightest to darkest.) with QC samples being analyzed first. The instrument sequence is typed into the HP Chemstation Software. Next run "Simulate Sequence" (also under the "Sequence" dropdown list) without clicking the "Overwrite Files" box. Compare the order of the vials on the instrument versus the sequence to confirm all the samples and standards are in the right places. Next click "Run Sequence" also under the HP Chemstation "Sequence" dropdown list.

10.3.3.4 After the samples have been analyzed, the data files from the MS are quantitated versus the proper quantitation method. The QCPRN1.MAC macro creates a form with which to easily check internal standard and surrogate criteria are met. The following should be reviewed initially:

10.3.3.4.1 Are all the surrogates within QC criteria? Please see Section 9.7.1 for surrogate information.

10.3.3.4.2 Is the internal standard- 1,4 Dichlorobenzene-d4, within 50-200% of the daily CCAL? If not, the samples should be checked for matrix interferences that may be causing these issues. The IS peaks should also be evaluated for peak splitting or incorrect integration by the software. A sample may not need to be reanalyzed if it can be determined (with guidance from a supervisor) that the QC is exceeded due to matrix interference.

10.3.3.4.3 Are all target analytes within calibration range? If not, the sample(s) should be diluted and re-analyzed. If a dilution is performed after the internal standard has already been added, it will be necessary to add additional IS in order to make up for the impact of the original IS added also being diluted. Conversely, if a sample has been over-diluted, it may need to be analyzed at less of a dilution to detect target analytes that may have been diluted out. Note: for this method, due to the isotope dilution, it is necessary to refer to area counts to determine if dilution is required. The area response for 1,4-Dioxane should be compared to the area of the highest level of the ICAL standard for that target. If the area of 1,4-Dioxane in the extract is greater than the area of 1,4-Dioxane in the Highest level of the Calibration Curve, then a dilution is necessary. The analyst **CAN NOT** use the concentration of 1,4-Dioxane found in the extract to determine if dilution is required because the concentration of 1,4-Dioxane in the sample is surrogate corrected in Chemstation based on the use of the extraction surrogate as an Internal Standard. Once it is determined based on the peak area that a sample requires a dilution, the analyst must pay attention to the concentration of IS in the dilution. Although the analyst will adjust the 1,4-Dichlorobenzene-d4 concentration by adding the appropriate amount of additional IS to the dilution, there is no way for the analyst to make up for the dilution of the extraction surrogate 1,4 dioxane-d8 (also used as internal standard). To account for this the analyst must change the concentration of the 1,4-Dioxane-d8 within the calibration table to a value which accounts for the dilution. (Example: If the analyst performs a 10x dilution then the concentration of 1,4-Dioxane–d8 must be changed within the ICAL to 50.) The analyst must ensure that only the diluted sample is calculated against this

value and that the method is returned to the proper concentration once the analysis of the dilution is complete.

10.3.3.4.4 Are all analyses within 12 hour tune time? If a sample is analyzed outside tune time, it will need to be re-analyzed in another tune clock.

10.3.3.4.5 The sequence should also be printed out from Chemstation, initialed and dated, and placed in the logbook.

10.3.3.4.5.1 If anything in the initial review of the data indicates that there should be a re-analysis, the reason for re-analysis should be noted on the sequence.

10.3.3.4.5.2 Also, note the time the tune standard was analyzed, the time of the last sample analysis. If there are samples analyzed outside tune time, the time the last sample within tune time ran should be noted, as well as the last sample analyzed.

10.3.3.4.5.3 If a re-extract is required, the "Request for Repreparation/Reclean" book should be filled out and a photocopy of the appropriate page should be given to the Preparation Group leader or the Organics Section Head.

10.4 Continuing Calibration

Continuing Calibration Criteria is outlined in section 10.3.2

10.5 Preventive Maintenance

If performing any maintenance on any piece of equipment it must be documented in the *Instrument Maintenance Logbook* located in the laboratory specific to each instrument.

Daily

Injection port maintenance: Maintenance should be done when the daily CCAL starts to demonstrate degradation either by %D outliers or area responses <50% as compared to the ICAL areas. Several tune clocks may be injected before maintenance is needed. The type of samples analyzed will have an effect on how soon maintenance should be performed. Injection port maintenance should be done as needed. General maintenance includes replacing the single baffle liner packed lightly with glass wool, cutting about 2-4 inches off the head of the column, and replacing the septa. Refer to maintenance log for more specific information.

The Gerstel Injection port should be handled with care. The liners are quite thin. Do not force the Gerstel weldmen into place as the threads are soft metal and will cross thread. Always make sure the weldmen goes on straight. The Gerstel injection port does not require significant tightening of either the weldmen or column nut, tighten enough to seal but there is no need to crank down on it.

11. Data Evaluation, Calculations and Reporting

11.1 Qualitative Analysis

11.1.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of mass spectrum, after background correction, with

characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the two ions of greatest relative intensity, and are over 30% relative intensity. Compounds are identified when the following criteria are met.

11.1.2 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. A peak selected by the data system, based on the presence of target specific ions at a target specific retention time will be accepted as meeting these criteria.

11.1.3 The relative retention time of the sample component is within $\pm$ 0.06 RRT units of the RRT of the standard component.

11.1.4 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.) The relative intensities are monitored daily. The relative intensities will be updated when they exceed established values from the reference spectrum.

11.1.5 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

11.1.6 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e. a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important. Selective ion monitoring eliminates this potential.

11.2 Quantitative Analysis

11.2.1 Response factors and % RSD to evaluate Initial Calibration acceptability.

Calculate RF by:

$$RF = \frac{area_{cmp}}{area_{is}} \times \frac{conc_{is}}{conc_{cmp}}$$

where:

area cmp = Area of the characteristic ion for the compound being measured.
area is = Area of the characteristic ion for the specific internal standard.
conc is = Concentration of the specific internal standard.
conc cmp = Concentration of the compound being measured.

Calculate % RSD by:

$$\%RSD = \frac{SD}{\overline{x}} \times 100 \qquad SD = \sqrt{\sum_{i=1}^{N} \frac{(x_i - \overline{x})^2}{N-1}}$$

where:

% RSD = percent relative standard deviation
x = average of RF's
SD = standard deviation
xi = analytical results of each level in the final reporting units
N = number of results (levels)

11.2.2 Calculate % Difference (%D) by:

$$\%D = \frac{\overline{RF}_i - RF_c}{\overline{RF}_i} \times 100$$

where:

RFi - Initial Calibration average RF
RFc = Continuing Calibration RF

11.2.3 Results of Water Analysis - calculation as performed in report form:

$$\text{Concentration (ug/L)} = \frac{(\text{Conc})\ (\text{Vf})\ (\text{DF})}{(\text{Vi})} \times 1000$$

11.2.4 Results of Soil/Sediment Analysis – calculation as performed in report form:

$$\text{Concentration (ug/Kg)} = \frac{(\text{Conc})\ (\text{Vf})\ (\text{DF})}{(\text{Vi})\ \%S} \times 1000$$

where:
Conc = Raw on-column concentration obtained from the quantitation report using Initial Calibration results.
Vf = Final volume of extract (mL)
Vi = Volume of sample extracted (mL), or weight of sample extracted in grams (g)
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".
%S = percent solids, as a decimal

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9.0 outlines sample batch QC acceptance criteria. If non-compliant organic compound results are to be reported, the Organic Section Head and/or the Laboratory Director, and the Operations Manager must approve the reporting of these results. The laboratory Project Manager

shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst, Data Reviewer, or Department Supervisor performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

All results for the organic compounds of interest are reportable without qualification if extraction and analytical holding times are met, preservation requirements (including cooler temperatures) are met, all QC criteria defined in the table below are met, and matrix interference is not suspected during extraction or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

QC Parameter	Acceptance Criteria
Method Blank	No analyte above the reporting limit The results are qualified with a "B" for any associated sample concentrations that are less than 10x the blank concentration for this analyte
Surrogate Recovery	15% - 110%
Laboratory Control Samples	40% - 140% and 30% RPD
Matrix Duplicate	30% RPD
Matrix Spike	40% - 140%
Matrix Spike Duplicate	40% - 140% and 30% RPD

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP 1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)
SOP 1739 Demonstration of Capability (DOC) Generation
SOP 1731 Manual Integration & Compound Rejection
SOP 1797 Hazardous Waste and Sample Disposal

SOP 1816 Reagent, Solvent and Standard Control

16. Attachments

None

Acid Volatile Sulfides and Simultaneously Extracted Metals in Sediments

Reference: "Determination of acid-volatile sulfide and simultaneously-extracted metals in sediments using sulfide-specific electrode detection," Warren S. Boothman, USEPA Environmental Research Laboratory, Narragansett, RI, and Andrea Helmstetter, Science International Corp., Narragansett, RI. September 4, 1992. AVSSEM SOP v2.0.

EPA-821-R-91-100 – DRAFT Analytical Method of the Determination of Acid Volatile Sulfide in Sediment, US EPA Offices of Water, and Science and Technology, 1991.

1. Scope and Application

Matrices: Sediments

Definitions: Refer to Alpha Analytical Quality Manual.

Acid volatile sulfides (AVS) are amorphous or moderately crystalline metal monosulfides, primarily FeS. It has been demonstrated that AVS may be the most important factor controlling the availability of heavy metals to benthic organisms in anoxic sediments. The relative concentrations of AVS and simultaneously extractable metals (SEM) is postulated as an indicator of metal availability.

The classification of sulfides as "acid volatile" is an operational definition; i.e., the extent to which mineral sulfide phases are volatilized by the analysis will depend on the analytical conditions used (acid concentrations, time, etc.). For AVS data to be comparable, the reaction conditions used to volatilize sediment sulfides must be similar. Simultaneously extracted metals are operationally defined as the metals extracted from the sediment into solution by the acid volatilization procedure. Soluble metals are those passing through a 0.45 μm filter.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of experienced analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

To determine AVS in sediments, an aliquot of wet sediment is acidified with HCl under nitrogen gas to produce gaseous hydrogen sulfide (H_2S). The H_2S is purged from the flask and trapped in buffer solution. The sulfide concentration is measured electrometrically using an ion-selective electrode against a sulfide standard solution curve.

Simultaneously extracted metals (SEM) are determined by filtration of the acid-sediment slurry and analysis of the extract by ICP, ICP-MS, or CVAF as per laboratory SOPs which describe analysis of dissolved metals in aqueous solutions.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Sulfide – 200 ug/L (0.624 µmol/g)

Cd – 2 µg/L (0.00178 µmol/g)

Cu – 10 µg/L (0.0157 µmol/g)

Pb – 20 µg/L (0.00965 µmol/g)

Ni – 20 µg/L (0.0341 µmol/g)

Zn – 20 µg/L (0.0306 µmol/g)

µmol/g values are based on 1 g wet weight.

4. Interferences

Volatilized sulfide will be lost if oxygen is present in apparatus or reagents. The apparatus used must be completely purged with oxygen-free nitrogen gas prior to beginning of analysis.

Volatilization of sulfides from sediments by acidification can be affected by sediment matrix which may consume the acid and increase the slurry pH. This may occur in sediments with high carbonate content. In such cases, the acid extracts should be tested periodically, especially if effervescence is observed during acidification with HCl, to insure that the pH < 2.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Because the sulfide ion is unstable in the presence of dissolved O_2, storage containers should minimize the air space above the sediment. Samples should be collected in soil jars with sealed caps to minimize volatile losses. Approximately 20 to 30 g are required (to allow for replicate and QC analyses, as required).

6.2 Sample Preservation

Samples should be stored at 4° ± 2° C following collection.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples should be analyzed within 14 days of collection.

7. Equipment and Supplies

7.1 pH/Ion selective electrode meter

7.2 Sulfide selective electrode

7.3 Flow controller

7.4 Flasks (250 mL round-bottom; 100 mL volumetrics), beakers (150 mL), graduated cylinders (50 mL and 100 mL)

7.5 Filter holders with receivers (250 mL)

7.6 47 mm filters, 0.45 μm nominal pore size

7.7 Tygon tubing

7.8 Air sampling impingers and impinger bottles

7.9 20-30 ml polyethylene barrel syringes with needles

8. Reagents and Standards

8.1 Reagents

8.1.1 Nitrogen gas - oxygen-free. Ultra pure N_2 gas can be purchased and/or an oxygen trap attached to the N_2 gas tank in-line to remove the trace oxygen.

8.1.2 Hydrochloric Acid (HCl). Instra-Analyzed Grade. 6N HCl prepared by diluting 250 mLs HCl into 250 mLs DI water.

8.1.3 Sulfide Anti-Oxidant Buffer Reagent (SAOB). (2M NaOH, 0.2M EDTA, 0.2M ascorbic acid) (note - prepare in order indicated). Dissolve 80.00 g NaOH *slowly* in 700 mL DDI water. When cool, add 74.45 g EDTA (disodium form) and stir until dissolved. Add 35.23 g ascorbic acid and stir until dissolved. Pour solution into a 1000 mL beaker and dilute to volume with DDI water. For use in impinger.

8.1.3.1 Dilute SAOB. Mix 350 ml of SAOB with 350 ml DDI water. For use with standards only.

8.1.4 Sulfide Stock Solution

8.1.4.1 Primary Stock Preparation (Approx. 1650 ppm). Wash crystals of $Na_2S \bullet 9H_2O$ with deionized water and blot dry. Weigh approximately 12 g of $Na_2S \bullet 9H_2O$ and dissolve in 900 ml DDI water. Pour into a 1000 ml volumetric flask and dilute to volume with DDI water. Standardize daily.

8.1.4.2 PrimaryStock Standardization. Pipette 10.00 ml of standard iodine solution into each of two 125 ml Erlenmeyer flasks. Pipette 2.00 ml of sulfide stock into one flask. Pipette 2.00 ml of DDI water as a method blank into the other flask. Add 5.0 ml of 6M HCl into each flask, swirl lightly, then cover and place in the dark for 5 minutes. Titrate each with 0.025N thiosulfate solution, adding soluble starch indicator when the yellow iodine color fades. The end point is reached when the blue color disappears.

$$\text{sulfide } (\mu\text{mol/ml}) = \frac{(T_{bl}-T_s) \times N \times \frac{1 \text{ mole } S^{2-}}{2 \text{ equiv } S^{2-}} \times \frac{1000 \ \mu\text{moles}}{1 \text{ mole}}}{\text{ml sample}}$$

sulfide (ppm) = sulfide (Φmol/ml) x 32
where T = ml titrant used for blank and spike
where N = normality of thiosulfate titrant

8.1.5 Standard Iodine solution (0.025N). This solution is purchased commercially.

8.1.6 Thiosulfate titrant (0.025N). This solution is purchased commercially and diluted. The solution is then standardized against the 0.025N standard iodine solution.

8.1.7 Soluble starch indicator. Prepare by dissolving 1.0 g starch in 100 ml boiling deionized water.

8.2 Calibration and QC Solutions

8.2.1 Calibration standards are prepared by diluting primary sulfide stock solution to produce a working standard. The primary stock concentration *must* be standardized by iodometric titration before preparing the working standards. See section 8.1.4.2.

8.2.2 Working standard is made by 6.0 ml primary sulfide stock and diluting to a final volume of 200 ml with dilute DI WATER. The working standard exact concentration is determined by the following calculation:

$$\text{working standard (ppm)} = \frac{(6) \text{ (Primary stock conc. *)}}{}$$

200

* as determined by standardization

8.2.3 Calibration curve consists of the following aliquots of working standard diluted to 100 ml with dilute SAOB: 0.20, 0.50, 2.0, 5.0, 20.0, and 50.0 ml. Exact concentrations are determined daily by the following equation:

$$\text{ppm sulfide} = \frac{(\text{ml working standard}) \times (\text{conc. working standard})}{100}$$

8.2.4 Initial Calibration Verification (ICV) Continuing Calibration Verification (CCV). The mid point of the calibration curve must be analyzed at the beginning of an analytical sequence, after a minimum of every ten samples and at the end of each analytical sequence. The calibration curve need not be generated daily. If a calibration curve is used over more than a one day period then an acceptable CCV must be generated prior to continuing with the analysis.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A preparation blank should be prepared with each batch. The concentration should be less than the reporting limit of the analysis. Corrective Action. Determine the cause of the contamination, reprepare batch if reporting limit can not be raised above the level of the blank.

9.2 Laboratory Control Sample (LCS)

A LCS or blank spike should be analyzed with every batch to verify the analytical system. Recovery should be within the range of 85-115%. Corrective Action: If LCS recovery is outside this range, determine source of error before analyzing samples. An acceptable LCS recovery must be obtained prior to the analysis of samples.

9.3 Initial Calibration Verification (ICV)

The ICV should be analyzed at the beginning of each run, after calibration, to verify the accuracy of the electrode. Acceptable recovery is ± 15% of true value. Corrective Action: Repeat the determination if recovery is still out determine cause. Re-calibrate electrode meter. Reanalyze ICV. Analysis cannot proceed until an acceptable ICV recovery is obtained.

9.4 Continuing Calibration Verification (CCV)

The CCV should be analyzed after a maximum of ten samples with a recovery of ± 15%. Corrective action. Repeat the determination if recovery is still out determine cause. Re-calibrate electrode meter. Reanalyze all samples from last passing QC standard.

9.5 Matrix Spike

Analyze a matrix spike (MS) for every ten environmental samples(10% or more frequent) using the working standard as a spiking solution. Add the sulfide metals spike with the 6N HCl through the sidearm. Acceptable recovery is 75-125%. Corrective Action: If blank spike is in-control and the MS is not; matrix interference is suspected. Include a narrative explanation to

client that there is evidence of a matrix effect on the accuracy of the results. If project-specific QC requires, the analysis of the MS could be repeated using a smaller sample aliquot to minimize matrix effects.

9.6 Laboratory Duplicate

Duplicate analyses (matrix duplicate) should be performed at a frequency of 5% or each analytical batch, whichever is more frequent. The RPD should not exceed 20%. Corrective Action: If the RPD exceeds 20%, include narrative to client that there is evidence of a matrix effect on the precision of the results.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

Calibration

ICV

ICB

Method Blank

LCS

Sample 1

Duplicate

Matrix Spike

(Matrix Spike Duplicate)

Sample 2

Sample 3 etc.

CCV

CCB

10. Procedure

10.1 Equipment Set-up

10.1.1 **Extraction**.

10.1.1.1 Weigh 2-3 g sediment into an acid washed 250 ml round bottom flask with septum sidearm. Add 50 ml DDI water and an acid washed stir bar. It is important to try to extract enough sample to generate a positive Sulfide response in order to evaluate the SEM/AVS ratio. Some samples (such as clean sand) will not contain enough sulfide for detection.

10.1.1.2 To the impinger (trap) add 50 ml SAOB solution and 30 ml DDI water. Purge system with nitrogen for 10 minutes. Slowly add 10 ml of 6N HCl through sidearm. Turn on stir plate.

10.1.1.3 Purge system with nitrogen for 60 minutes.

10.1.1.4 Remove impinger. and pour solution into 100 ml volumetric flask while rinsing impinger trap with DI water. Dilute to 100 ml with DDI water.

10.1.2 Measurement of sulfide by ion-specific electrode.

10.1.2.1 Prepare calibration curve after standardizing primary stock to determine exact sulfide concentration. A 6-point curve is generated by regressing log sulfide concentration against mvolt reading from the probe. The blank is not used in the regression. The correlation coefficient r must be $\leq$ -0.995.

10.1.2.2 Pour standard or sample from flask into 150 ml beaker, add stir bar and stir with minimum agitation (to avoid oxidation of sample). Using DDI water, rinse electrodes and blot dry. Immerse electrode in sample solution.

10.1.2.3 Allow electrode response to stabilize. Record millivolt reading. Plot calibration curve of standards. Use standard curve to convert sample millivolt readings to log sulfide concentration of sample. The sample sulfide concentration is calculated as the antilog of the result generated from the calibration curve.

10.1.2.4 If the measured sulfide concentration exceeds the range of calibration standards, repeat the analysis using a smaller amount of sediment. If the measured concentration is less than the low standard of the calibration curve, repeat the analysis for that sample using more sediment.

10.2 Initial Calibration

Refer to Section 10.1.2.1 for sulfide calibration curve information.

10.3 Equipment Operation and Sample Processing

10.3.1 Simultaneously extracted metals.

10.3.1.1 Assemble the acid washed filtration unit with a 0.45μm pore size filter and apply pressure.

10.3.1.2 Pour the acid-sediment slurry of the round-bottom flask into the upper chamber of the filtration unit and apply pressure. Rinse the round bottom flask several times with DDI water and add the rinsed to the filtration unit. When all the solution has been filtered, wash the walls of the upper chamber of the filtration unit with DDI water.

10.3.1.3 Pour the filtrate into a clean 100 mL graduated cylinder. Bring to volume with DDI water. Mix the sample and pour into a labeled, acid-cleaned polyethylene bottle and cap. The sample is ready for metals analysis.

10.3.1.4 Weigh out aliquot and perform dry-weight (percent solids test) determination following laboratory SOP.

10.4 Continuing Calibration

CCVs must be 90-110% for SEM metals by ICPMS, sulfide must be 85-115%.

10.5 Preventive Maintenance

Clean impinge exterior with Kimwipes to remove sediment/residue. Clean interior with cotton swabs.

11. Data Evaluation, Calculations and Reporting

11.1 AVS concentration in sediments.

$$\text{Acid Volatile Sulfide (mg/Kg dry wt)} = \frac{\text{mg/L from curve x trap volume}}{\text{initial g sediment x dry wt conversion}}$$

11.2 SEM concentrations in sediments.

$$\text{SEM } (\mu\text{g/g dry wt}) = \frac{\text{concentration metal x final volume of 100 mL}}{\text{initial g sediment x dry wt conversion}}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Refer to Section 9.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP #1732 MDL/LOD/LOQ Generation

SOP# 1739 IDC/DOC Generation

SOP# 1728 Waste Management and Disposal

16. Attachments

None.

Chemical Oxygen Demand (COD)
Including: High (0-1500ppm) and Low (0-150ppm) Levels
Closed Reflux, Colorimetric Method (Total and Soluble in Liquid)

References: **SM 5220 D,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 410.4, Methods for the Determination of Inorganic Substances in Environmental Samples, August 1993.

1. Scope and Application

Matrices: This method is applicable to liquid samples.

Definitions: Refer to Alpha Analytical Quality Manual.

The Chemical Oxygen Demand (COD) is used as a measure of the oxygen equivalent of the organic matter content of a sample that is susceptible to oxidation by a strong chemical oxidant. For samples from a specific source, COD can be related empirically to BOD, organic carbon, or organic matter. The test is useful for monitoring and control after correlation has been established. The dichromate reflux method is preferred over procedures using other oxidants because of superior oxidizing ability, applicability to a wide variety of samples, and ease of manipulation. Oxidation of most organic compounds is 95 to 100% of the theoretical value. Pyridine and related compounds are oxidized only to the extent that they remain in contact with the oxidant. Ammonia, present either in the waste or liberated from nitrogen-containing organic matter, is not oxidized in the absence of significant concentration of free chloride ions.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Most types of organic matter are oxidized by a boiling mixture of chromic and sulfuric acids. A sample is refluxed in strongly acid solution with a known excess of potassium dichromate ($K_2Cr_2O_7$). After digestion, the remaining unreduced $K_2Cr_2O_7$ is determined colorimetrically and the oxidizable organic matter is calculated in terms of oxygen equivalent. Keep ratios of reagent weights, volumes, and strengths constant. The standard two-hour reflux time may be reduced if it has been shown that a shorter time yields the same results.

2.1 Method Modifications from Reference

2.1.1 Soluble COD (SCOD): Filter the sample through 0.45µm filter followed by preservation to pH $\leq$2 using 1:1 H_2SO_4.

2.1.2 Low Level COD: Analyze samples in low level range COD tubes for more precise results on lower concentrations of target analyte.

3. Reporting Limits

The reported detection limit for chemical oxygen demand, closed reflux, colorimetric method, is 20.0mg/L for (High) COD method, and 10.0mg/L for (Low) COD method.

4. Interferences

4.1 Instrumental

The Spectrophotometer and COD Reactors are subject to preventative maintenance, which will minimize instrumental interferences. (Refer to Section 10.6)

4.2 Parameters

4.2.1 **Volatile Straight-Chain Aliphatic Compounds:** These compounds are not oxidized to any appreciable extent. This failure occurs partly because volatile organics are present in the vapor space and do not come in contact with the oxidizing liquid. Straight-chain aliphatic compounds are oxidized more effectively when silver sulfate (Ag_2SO_4) is added as a catalyst. However, Ag_2SO_4 reacts with chloride, bromide, and iodide to produce precipitates that are oxidized only partially. The difficulties caused by the presence of the halides can be overcome largely, though not completely, by complexing with mercuric sulfate ($HgSO_4$) before the refluxing procedure. Although 1g $HgSO_4$ is specified for 50mL sample, a lesser amount may be used where sample chloride concentration is known to be less than 2000mg/L, as long as a 10:1 ratio of $HgSO_4$ is maintained. Do not use the test for samples containing more than 2000mg Cl/L. Techniques designed to measure COD in saline waters are available.

4.2.2 **Nitrite (NO_2^-)** exerts a COD of 1.1mg O_2/mg NO_2^- -N. Because concentrations of NO_2^- -N in waters rarely exceed 1 or 2mg NO_2^- -N/L, the interference is considered insignificant and usually is ignored. To eliminate a significant interference due to NO_2^-, add 10mg sulfamic acid for each mg NO_2^- -N present in the sample volume used; add the same amount of sulfamic acid to the reflux vessel containing the distilled water blank.

4.2.3 **Reduced Inorganic Species,** such as ferrous iron, sulfide, managanous manganese, etc., are oxidized quantitatively under the test conditions. For samples containing significant levels of these species, stoichiometric oxidation can be assumed from known initial concentration of the interfering species and corrections can be made to the COD value obtained.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

NOTE: COD tubes contain Mercury, Dichromate and Sulfuric Acid. General laboratory safety practices must be observed

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Preferably, collect samples in glass bottles. Blend samples containing settleable solids with a homogenizer to permit representative sampling.

6.2 Sample Preservation

If delay before analysis is unavoidable, preserve sample by acidification to pH ≤2 using 1:1 H_2SO_4. Preferably acidify any sample that cannot be analyzed the same day it is collected.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Test unstable samples without delay. Preserved samples are analyzed within 28 days of sampling. Store samples under refrigeration at 4 ± 2°C.

7. Equipment and Supplies

7.1 COD Reactor: HACH Company, Environmental Express or equivalent, 150ºC

7.2 Spectrophotometer: For use at 620nm.

7.3 Computer and related accessories: with LIMS access and Excel

7.4 Automatic Pipettor: Capable of 2.0mL

7.5 Pipets: 10mL and 1mL, borosilicate glass.

7.6 Centrifuge.

7.7 Kim Wipes.

7.8 Volumetric flasks, assorted sizes (1000mL, 100mL)

8. Reagents and Standards

8.1 Stock Calibration Standard (for calibration curve generation): 5000ppm Potassium Hydrogen Phthalate (KHP) Stock Calibration Standard: 4.25g of dry KHP and 1ml H3PO4 brought up to 1L with DI. Prepare stock calibration standard every six months and generate a new calibration curve (Section 10.1). Store stock calibration standard under refrigeration at 4 ± 2 °C. Standard expires after six months.

8.1.1 Working Calibration Standards for High COD: Prepare fresh standards for each calibration curve. The following Potassium Hydrogen Phthalate (KHP) standards are prepared in volumetric flasks:

8.1.1.1 400ppm: 8ml of 5000ppm (Section 8.1) to 100ml with DI.

8.1.1.2 800ppm: 16ml of 5000ppm (Section 8.1) to 100ml with DI.

8.1.1.3 1000ppm: 20ml of 5000ppm (Section 8.1) to 100ml with DI.

8.1.1.4 1250ppm: 25ml of 5000ppm (Section 8.1) to 100ml with DI.

8.1.1.5 20ppm: 2ml of 1000ppm (Section 8.1.1.3) to 100ml with DI.

8.1.1.6 50ppm: 5ml of 1000ppm (Section 8.1.1.3) to 100ml with DI.

8.1.1.7 100ppm: 10ml of 1000ppm (Section 8.1.1.3) to 100ml with DI.

8.1.1.8 200ppm: 20ml of 1000ppm (Section 8.1.1.3) to 100ml with DI.

8.1.2 Working Calibration Standards for Low COD:

8.1.2.1 500ppm: 10ml of 5000ppm (Section 8.1) to 100ml DI.

8.1.2.2 5ppm: 1ml of 500ppm (Section 8.1.2.1) to 100ml DI.

8.1.2.3 10ppm: 2ml of 500ppm (Section 8.1.2.1) to 100ml DI.

8.1.2.4 20ppm: 4ml of 500ppm (Section 8.1.2.1) to 100ml DI.

8.1.2.5 50ppm: 10ml of 500ppm (Section 8.1.2.1) to 100ml DI.

8.1.2.6 100ppm: 20ml of 500ppm (Section 8.1.2.1) to 100ml DI.

8.1.2.7 125ppm: 25ml of 500ppm (Section 8.1.2.1) to 100ml DI.

8.2 Stock Verification Standard (for calibration curve verification): 5000ppm Potassium Hydrogen Phthalate (KHP) Stock Verification Standard: 4.25g of dry KHP and 1ml H3PO4 brought up to 1L with DI. Prepare stock verification standard every six months. Stock verification standard must be of a different source than that used for the stock calibration standard (Section 8.1). Store stock verification standard under refrigeration at 4 ± 2 °C. Standard expires after six months.

8.2.1 Working Verification Standards [Including LCS (Laboratory Control Standard), ICV (Initial Calibration Verification), and CCV (Continuing Calibration Standard), (Section 9.2)]: Prepare fresh standards monthly or as needed. Standards are stored at room temperature in complete darkness. Standards expire after one month. The following Potassium Hydrogen Phthalate (KHP) standards are prepared in volumetric flasks:

8.2.1.1 1000ppm: 20ml of 5000ppm (Section 8.2) to 100ml with DI. (Also Low Cod Spike Solution).

8.2.1.2 500ppm Hi LCS/ICV Solution: 10ml of 5000ppm (Section 8.2) to 100ml with DI.

8.2.1.3 200ppm CCV Solution: 4ml of 5000ppm (Section 8.2) to 100ml with DI.

8.2.1.4 400ppm CCV Solution: 8ml of 5000ppm (Section 8.2) to 100ml with DI.

8.2.1.5 50ppm Low LCS Solution: 5ml of 1000ppm (Section 8.2.1.1) to 100ml with DI.

8.2.1.6 60ppm CCV Solution: 6ml of 1000ppm (Section 8.2.1.1) to 100ml with DI.

8.2.1.7 20ppm ICV/CCV Solution for Low Cod: 4ml of 500ppm (Section 8.2.1.1) to 100ml DI.

8.3 COD Tubes: High Cod Range 0 – 1500 mg/L, Low Cod Range 0-150 mg/L. Follow the manufacturer's directions for use and date of expiration. Store at room temperature in a complete darkness. NOTE: A new calibration curve must be generated for each brand of COD tubes purchased. The same curve cannot be used for different brands.

8.3.1 High Level COD Tubes (0-1500ppm): Environmental Express, Catalog # B1015

8.3.2 High Level COD Tubes (0-1500ppm): CPI International, Catalog #4380-1500-150

8.3.3 Low Level COD Tubes (0-150ppm): Environmental Express, Catalog # B1010

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

9.1.1 Method Blank/Initial Calibration Blank (ICB)

Add 2 mL DI to COD tube and digest. One Method Blank is analyzed per batch of 20 samples or less. The Method Blank result must be less than the RL. If the Method Blank fails acceptance criteria, the entire batch must be reset and reanalyzed. This Blank is used to zero the spectrometer before reading the cod batch samples and associated QC.

9.1.2 Continuing Calibration Blank (CCB)

The tube used the as the ICB is the same one used as the CCB. One CCB is analyzed every 10 samples. If the CCB fails acceptance criteria, the previous 10 samples need to be reread. If it fails a second time, then the entire batch must be reset and reanalyzed.

9.2 Laboratory Control Sample (LCS)

9.2.1 ICV / LCS

One Low LCS and one Hi LCS/ICV pair are analyzed per batch of 20 samples or less.

For method 5220, the LCS must be recovered within laboratory Control Limits.

For method 410.4, the results of the low LCS and Hi LCS/ICV must be within ± 10% of the true value.

If recovery is outside of acceptance criteria, the low LCS and Hi LCS/ICV are re-made and reanalyzed. If these criteria are still not met, a new calibration curve must be generated (Section 10.1), and the entire batch of samples is reset.

9.2.1.1 High COD:

9.2.1.1.1 Low LCS: 2.0mL of 50ppm standard (Section 8.2.1.5) is added to a COD tube.

9.2.1.1.2 High LCS/ICV: 2.0mL of 500ppm standard (Section 8.2.1.2) is added to a COD tube.

9.2.1.2 Low Cod:

9.2.1.2.1 Low LCS: 2.0mL of 20ppm standard (Section 8.2.1.7) is added to a COD tube.

9.2.1.2.2 High LCS/ICV: 2.0mL of 50ppm standard (Section 8.2.1.5) is added to a COD tube.

9.3 Initial Calibration Verification (ICV)

Refer to Section 9.2.

9.4 Continuing Calibration Verification (CCV) (Applies to High Cod only).

A CCV standard is analyzed after a Blank sample in the sample batch and after every 10 samples. The CCV is to verify that the calibration curve is valid over the range of use. The CCV concentration is rotated daily, with a different concentration each day.

To a COD tube, add 2.0mL from one of the CCV standards: 60ppm (Section 8.2.1.6), 200ppm (Section 8.2.1.3) or 400ppm (Section 8.2.1.4).

% Recovery must be within ± 10% of the true value. If % recovery is outside of these limits, the CCV Standard is re-made and reanalyzed. If these criteria are still not met, a new calibration curve must be generated (Section 10.1), and the entire batch of samples is reset.

9.5 Matrix Spike

One matrix spike is analyzed per batch of **20 samples** or less for method **5220**. One matrix spike is analyzed per batch **of 10 samples or less for method 410.4**. :

9.5.1 High COD: 100µL of 5000ppm KHP stock working standard (Section 8.2) is added to 2.0mL of sample in a COD tube. (Base value before dilutions = 238.1 mg/L).

9.5.2 Low COD: 100uL of 1000ppm KHP stock working standard (Section 8.2.1.1) is added to 2.0mL of sample in a COD tube. (Base value before dilutions = 47.6 mg/L).

% Recovery must be within **laboratory control limits** for method **5220** and be within **± 10%** of the true value for method **410.4.** If % recovery is outside of these limits, and the LCS recovery is acceptable, the matrix spike sample is reset. If failure continues, the matrix spike is reset on dilution. If recovery is acceptable, a narrative is submitted with the data indicating a dilution was necessary due to sample matrix. If failure still continues, and the LCS is acceptable, a narrative is submitted with the data indicating failure is due to sample matrix.

9.6 Laboratory Duplicate

One duplicate sample is analyzed per batch of 20 samples or less. The % RPD must be within laboratory control Limits for method 5220. The % RPD must be ≤ 20% for method 410.4. If % RPD is outside of these limits, the sample and its duplicate are reset. If failure continues, a narrative is submitted with the data indicating the sample is non-homogeneous.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Blank (ICB)
- Low LCS
- Hi LCS (ICV)
- Samples/QC Samples (1-10 or less)
- CCV
- CCB

- Samples/QC Samples (11-20 or less)
- CCV
- CCB

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation of Calibration Curve: A Calibration Curve is generated every six months when new Stock Calibration Standard is prepared (Section 8.1), upon purchase of a new brand and **new lot** of COD tubes, or sooner if necessary for method 5220. A Calibration Curve must be in place prior to sample analysis. The curve is generated using the eight calibration standards included within Section 8.1.1. for High Cod, or Section 8.1.2 for Low Cod.

Note: New calibration curve must be generated with every new lot of tubes purchased for method 5220.

NOTE for Method 410.4: Calibration curve is generated daily using Blank and 4 calibration standards: 20 mg/l, 100 mg/l, 400 mg/l and 1250mg/l.

Correlation coefficient will be calculated. All calibration points are back calculated on excel and should be within 10% from true concentration, except lowest point of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient must be > or = 0.995.

If calibration curve criteria failed, then new calibration standards have to be re-analyzed and new calibration curve has to be generated and all calibration points have to be recalculated to meet criteria.

If second calibration curve fails acceptance criteria, then analysis have to stopped, problem has to be identified; new calibration standards made and analyzed.

10.2 Initial Calibration

Analyze these standards according to Section 10.3.

Plot the absorbance values of the standards versus the corresponding COD concentrations. The correlation coefficient must be $\geq$ 0.995 for the curve to be considered valid.

10.3 Equipment Operation and Sample Processing

10.3.1 Preparation of COD tubes:

Preheat the COD reactor to 150°C.

Measure 2.0mL of sample or diluted sample into a COD tube. (NOTE: Use appropriate range level of tubes for High or Low COD analysis). In a similar manner, prepare the QC samples to be digested with the samples (refer to Sections 9.2 – 9.4 for High COD, and Sections 9.2 – 9.3 for Low Cod).

Tightly cap tubes and invert several times to mix completely. **CAUTION! Protect hands from heat produced when the contents of the vessel are mixed.** In order

to prevent local heating of vessel bottom and a possible explosive reaction, mix vessel contents thoroughly before placing in the COD reactor.

Place the prepared COD tubes into the heated COD reactor. Record the "Time in" and the reactor temperature on the batch sheet for later entry onto excel sheet. Reflux for 2 hours. Remove tubes from the COD reactor. Record the "Time out" and the reactor temperature on the batch sheet for later entry onto excel sheet. Allow tubes to cool to room temperature.

10.3.2 Measurement of Dichromate Reduction: If any liquid is visible on the walls of the cooled COD tubes, invert the tubes to incorporate this liquid. Allow solids to settle before measuring absorbance. If solids do not settle, the COD tubes must be centrifuged. Refer to section 10.3.3 for instructions on how to enter the data directly onto the Cod excel sheet.

10.3.3 Entering data onto the COD excel sheet

10.3.3.1 Turn on the computer and the spectrophotometer, at least 30 minutes prior to use.

10.3.3.2 On the computer desktop, double-click with the mouse to open the Gensys1 shortcut, hit "okay" when the box that says "device error" appears, then minimize the box which shows that the device has been activated.

10.3.3.3 Next, open the \\Fileserv\wetchem shortcut, followed by the cod_xlt file for High Cod, and cod_low_xlt file for Low Cod. Once these excel sheet opens, located at the top, right of the page, enter the WG batch number from the LIMS printout (done prior to Cod analysis). Enter the technician's initials in the appropriate area. Enter the date and time of the Cod preparation, then the date and time of the cod analysis.

10.3.3.4 Select which corresponding COD product is being analyzed: (ex. COD-5210), from the "buttons" area.

10.3.3.5 In the reagents/media information section of the excel sheet, enter the date of the preparation of the working standards (the High LCS/ICV, Low LCS, and CCV) used during the procedure (in order for the excel sheet to accept these dates, a " ' " must be typed first, followed by the date in an only numerical fashion, (for example: '091108 would express that the standards were made on 09-11-08 on the excel sheet) note that when this is typed, the " ' " will not be visible, but it is required to enter on the sheet).

10.3.3.6 On the MS line, enter the date of preparation of the original stock standard by typing it the same way.

10.3.3.7 On the COD Tubes line, enter the WC # which is the Wet Chemistry reagent log identifier, (for example: on the exterior of the cod box, a handwritten identifier saying WC-596 is visible).

10.3.3.8 Use the mouse to hit the "Get Samples" button on the screen. This will call up all the batch information and place it onto the excel sheet.

10.3.3.9 Below the column heading entitled Time, count down 10 samples; use the mouse to click on the far left, row numbers column, right-click on the mouse to activate the choice to insert another row. Do this process twice. In these newly added rows, type in (under column heading entitled Sample number), and the concentration of the (rotating for High Cod only) CCV standard, which was used

for the Cod batch to be read. If the batch is a full batch of 20 samples, this process of inserting extra rows will need to be repeated below the next 10 samples also. (Therefore, when reading the Cod tubes, the Blank, ICV, and first 10 samples will be read, then the CCV and CCB, followed by the next 10 samples, the CCV and CCB a second time, followed by the Duplicate and Spike, followed by the CCV and CCB a third time to finish the readings for that batch. If there are less than 20 samples, the rows are entered accordingly where necessary, allowing for the CCV and CCB to be read every 10 samples.)

10.3.3.10 Once all of the initial setup of the excel sheet has been accomplished, set the curser above the Time column and use the arrows to move the cursor down to the first box in the column. It is imperative that the box does NOT have a blinking cursor present in it when readings are begun.

10.3.3.11 Ensure that the spectrophotometer is set to the 620 wavelength and that all Cod tubes are ready for reading as described in section 10.3.1

10.3.4 Reading COD tubes (including Initial and Continuing Calibration requirements)

10.3.4.1 Wipe the exterior of the digested Method Blank COD tube with a Kim Wipe. Insert the Blank COD tube into the light path of the spectrophotometer set at 620nm. Set the absorbance to Zero.

10.3.4.2 Initial Calibration for the analysis requires that all initial QC (i.e. Blank, Low and High LCS) pass method requirements before proceeding on to read samples.

10.3.4.3 Read the absorbance of all samples and QC samples, wiping each tube with a Kim Wipe prior to reading.

10.3.4.4 On the lower right side of the face of the spectrophotometer, there is a print button. Once zeroing the Blank, and making sure to have the correct box highlighted on the excel sheet, hit the print button and it will beep as the date, time, and absorbance information are transferred to the excel sheet. The batch should be read in order as it occurs on the excel sheet, to enable the time sequence to be accurate.

10.3.4.5 Continuing Calibration requires that, following each 10 samples, the CCV (rotating value for High Cod only) is read, followed by the CCB, in order to demonstrate that the curve is valid. If the recovery result is valid according to the method criteria, then continue reading the next 10 samples. If the CCV and/or CCB fail, the previous 10 samples are no longer valid and must be reanalyzed as well as the CCV, CCB, and future unread samples in the batch.

10.3.4.6 When all readings have been taken, manually fill in the spike result based on the result shown in the above readings. Then hit the Save To LIMS button. Once this has been done, the data will be available in the LIMS, and a hardcopy will automatically print to review.

10.3.4.7 To repeat this process for a new COD batch, return to section 10.3.3.3.

10.4 Continuing Calibration

See Section 10.3.4.5

10.5 Preventive Maintenance

All spectrophotometers are calibrated on a semi-annual basis and an instrument service company calibrates the COD Reactors on an annual basis. Records are kept on file.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

11. Data Evaluation, Calculations and Reporting

$$\text{COD as mg } O_2 / L = \frac{\text{Absorbance - Y-intercept}}{\text{Slope}} \times \text{dilution factor}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the LCS recovery falls outside the designated acceptance range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

If the Matrix Spike acceptance criteria is not met, the spiked sample is reanalyzed (if possible). If failure continues and if all other QC performance criteria are met, the data is reported and a narrative is included with the final report.

If the RPD for the Duplicate measurements falls outside the Laboratory defined control limits, the sample is reanalyzed (if possible). If failure continues, and if all other QC performance criteria are met, the data is reported and a narrative is included with the final report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

The quarterly method reporting limit (MRL) verification requirement is satisfied by the quarterly LOQ analysis, as per SOP/1732.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734 and 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1734, 1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None

Chloride

References: **Method SM 4500Cl-E,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 9251, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1997.

Method 10-117-07-1-B, Chloride in Water, Lachat Instruments, 6645 West Mill Road, Milwaukee, WI 53218 (1993).

1. Scope and Application

Matrices: The method is applicable to potable water, surface water, saline water, domestic and industrial wastewaters. The concentration range can be varied by using the colorimeter controls. Soil samples can be analyzed following water extraction.

Definitions: See Alpha Laboratories Quality Manual Appendix A.

Chloride, in the form of chloride (Cl-) ion, is one of the major inorganic anions in water and wastewater. In potable water, the salty taste produced by chloride concentrations is variable and dependent on the chemical composition of water. Some waters containing 250mg Cl-/L may have a detectable salty taste if the cation is sodium. On the other hand, the typical salty taste may be absent in waters containing as much as 1000mg/L when the predominant cations are calcium and magnesium. The chloride concentration is higher in wastewater than in raw water because sodium chloride (NaCl) is a common article of diet and passes unchanged through the digestive system. Along the seacoast, chloride may be present in high concentrations because of leakage of salt water into the sewerage system. It also may be increased by industrial processes. A high chloride content may harm metallic pipes and structures, as well as growing plants.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Thiocyanate ion is liberated from mercuric thiocyanate by the formation of soluble mercuric chloride. In the presence of ferric iron, free thiocyanate ion forms the highly colored ferric thiocyanate, of which the absorbance is proportional to the chloride concentration. Samples are analyzed directly against a nonlinear calibration curve.

2.1 Method Modifications from Reference

None.

3. Detection Limits

The reported detection limit is 1.0mg Cl^-/L for water samples or 10.0 mg/kg for soils.

4. Interferences

4.1 Turbid samples and samples containing suspended solids are filtered through a 0.45µm filter.

4.2 Substances, which reduce iron (III) to iron (II) and mercury (II) to mercury (I), (e.g. sulfite, thiosulfate), will interfere.

4.3 Halides which also form strong complexes with mercuric ion (e.g. Br-, I-) give a positive interference.

4.4 Calcium and Magnesium ion may precipitate if present in sufficient concentration.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in clean, chemically resistant glass or plastic bottles. The maximum sample portion required is 100mL. Soil samples may be collected in glass or plastic containers.

6.2 Sample Preservation

Refrigerate at 4 ± 2 ºC until analysis.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

The maximum holding time is 28 days from collection.

7. Equipment and Supplies

7.1 Automated Ion Analyzer: Lachat Instruments.

7.2 Volumetric Flasks: Various volumes.

7.3 Volumetric Pipets: Various volumes.

7.4 Filtering Apparatus

7.5 0.45 µm Filters

7.6 Data Station

7.7 Analytical balances

7.8 Stirring plate

7.9 Plastic cups.

7.10 Ottawa sand.

7.11 10 ml syringes

8. Reagents and Standards

8.1 Stock Calibration Standard, 1000mg Cl^-/L: Commercially prepared. A Certificate of Analysis is kept on file. Stored at room temperatures and expires per manufacturer's expiration date.

8.1.1 Working Calibration Standards: Stable for period of six months. Store at room temperature.

8.1.1.1 100ppm Working Standard: To a 200mL volumetric flask, pipet 20.0mL of 1000ppm stock standard, (Section 8.1). Bring to volume with DI.

8.1.1.2 80ppm Working Standard: To a 100mL volumetric flask, pipet 8.0mL of 1000ppm stock standard, (Section 8.1). Bring to volume with DI.

8.1.1.3 20ppm Working Standard: To a 200mL volumetric flask, pipet 4.0mL of 1000ppm stock standard, (Section 8.1). Bring to volume with DI.

8.1.1.4 10ppm Working Standard: To a 200mL volumetric flask, pipet 2.0mL of 1000ppm stock standard, (Section 8.1). Bring to volume with DI.

8.1.1.5 5ppm Working Standard: To a 200mL volumetric flask, pipet 1.0mL of 1000ppm stock standard, (Section 8.1). Bring to volume with DI.

8.1.1.6 1ppm Working Standard: To a 200mL volumetric flask, pipet 2.0mL of 100ppm working standard, (Section 8.1.1.1). Bring to volume with DI.

8.1.1.7 Calibration Blank: DI water.

8.2 Stock Mercuric Thiocyanate Solution: In a 1L volumetric flask, dissolve 4.17g mercuric thiocyanate ($Hg(SCN)_2$) in about 500mL methanol. Dilute to the mark with methanol and stir for 20 minutes until dissolved. **CAUTION!** Mercury is a very toxic metal. Work under a hood and wear gloves! Solution is prepared every six months, and stored at room temperature.

8.2.1 Mercuric Thiocyanate, $Hg(SCN)_2$: Purchase ACS Grade. Store at room temperature. Expires upon manufacturer's expiration date.

8.2.2 Methanol: Reagent grade. Store at room temperature. Expires upon manufacturer's expiration date.

8.3 Stock Ferric Nitrate Reagent 0.5M: In a 1L volumetric flask, dissolve 202g ferric nitrate $Fe(NO_3)_3$ x $9H_2O$ in approximately 800mL water. Add 25mL concentrated nitric acid and dilute to the mark. Stir until dissolved. Reagent is prepared every 6 months, and stored at room temperature.

8.3.1 Ferric Nitrate, $Fe(NO_3)_3$ x $9H_2O$: Purchase ACS Grade. Store at room temperature. Expires upon manufacturer's expiration date.

8.3.2 Concentrated Nitric Acid, HNO_3: Reagent Grade. Store at room temperature. Expires upon manufacturer's expiration date.

8.4 Combined Color Reagent: In a 500mL volumetric flask, mix 75mL stock mercuric thiocyanate solution (Section 8.2) with 75mL stock ferric nitrate reagent (Section 8.3) and dilute to the mark with water. Invert three times to mix. Solution is prepared monthly, and stored at room temperature.

8.5 Stock LCS Standard, 1000mg Cl/L: Commercially prepared. A Certificate of Analysis is kept on file. The LCS Stock Standard must be from a different source than the Calibration Stock Standard. Store at room temperature, expires per manufacturer's expiration date.

8.5.1 5ppm (Low-level) LCS: Pipet 1mL of the 1000ppm stock LCS into a 200mL volumetric flask and bring to volume with DI. Standard is stable for six months. Store at room temperature.

8.5.2 30ppm (Mid-range) LCS: Pipet 3mL of the 1000ppm stock LCS into a 100mL volumetric flask and bring to volume with DI. Standard is stable for six months. Store at room temperature.

8.5.3 80ppm (High-level) LCS: Pipet 8mL of the 1000ppm stock LCS into a 100mL volumetric flask and bring to volume with DI. Standard is stable for six months. Store at room temperature.

8.5.4 Soil LCS: weigh 5 g(record exact weight) of Ottawa sand into plastic cup, pipet 2 ml of 1000 ppm stock LCS and add 48 ml of DI. Stir for 30 min, analyze water portion.

Final LCS concentration @ 400 mg/kg.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

9.1.1 Method Blank / ICB

Analyze one DI Blank per batch of 20 samples or less. An Initial Calibration Blank is analyzed at the start of each analytical sequence. The ICB consists of DI that has been filtered through a 0.45µm filter.
For soil samples: weigh 5 g of Ottawa sand (record exact weight in Log book), add 50 ml of DI, stir for 30 min, and filter. Analyze water portion.

Results must be less than the RL. If the ICB fails, re-run the ICB. If it passes criteria, the analysis can continue. If the ICB fails again, re-filter the ICB. If failure continues, analyze the ICB without filtering the DI, to determine if the contamination is coming from the filter or the water. If the ICB passes criteria, then check the filters. Any contaminated filters must be discarded; samples are only filtered through clean filters. Samples that don't have any trace of turbidity may be analyzed without filtering.

9.2 Laboratory Control Sample (LCS)

The calibration curve is verified at three concentrations at the start of each analytical run (ICV standards). Middle standard (30 mg/L) used for reportable LCS. The LCS for aqueous matrix must fall between 90-110% of the true value.

The LCS recovery criteria for the soil matrix is determined by calculating laboratory control limits, currently 89-109%.

If the LCS falls outside acceptance criteria, reprepare the solution and reanalyze. If the LCS falls outside for a second time, re-calibrate the system.

9.3 Initial Calibration Verification (ICV)

Refer to Section 9.2.

9.4 Continuing Calibration Verification (CCV)

The calibration curve is verified at 20 mg/l standard after every 10th sample, and at the end of the analytical run. Recovery for the CCV must be within ±10% of the true value.

If the CCV fails criteria, reanalyze the CCV. If it passes criteria, then continue with the analytical run. If the CCV fails for a second time, reprepare the CCV and reanalyze. If it passes criteria, reanalyze all samples that were analyzed since the last acceptable CCV.

9.5 Matrix Spike

Pipet 0.5mL of the 1000ppm Calibration Stock Standard (Section 8.1) into a 25mL volumetric flask and bring to volume with sample. The spike concentration will be 20ppm. Analyze one spike per batch of **20** samples or less for method **4500**. Analyze one matrix Spike sample for the batch of **10** or less samples for method **9251**.

For soil samples: weigh 5 g of sample (record exact weight), pipet 2 ml of 1000 ppm stock LCS and add 48 ml of DI. Stir for 30 min, analyze water portion. Final spike concentration is 400 mg/kg.

The MS recovery must fall within in-house control limits. If the MS falls outside of the recovery criteria, re-analyze both the MS and the associated sample. If the % recovery is still outside acceptance criteria, narrate the nonconformance due to sample matrix.

9.6 Laboratory Duplicate

Analyze one sample in duplicate per batch of **20** samples or less for method **4500**. Analyze one Duplicate sample for the batch of **10** or less samples for method **9251**.

The duplicate must be within in-house control limits. If the RPD falls outside acceptance limits, re-run both the duplicate and out of control sample. If the RPD is still outside acceptance criteria, narrate the nonconformance.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Calibration of the instrument
- ICV/LCS analysis (5, 30, 80ppm standards)
- Curve verification (DI blank and 20ppm calibration standard)
- Auto dilutor check
- Sample analysis, up to ten samples
- Curve verification (DI blank and 20ppm calibration standard) after every tenth sample and at the end of the analytical run.

10. Procedure

10.1 Equipment Set-up

Turn on instrument and connect appropriate board.

10.2 Initial Calibration

10.2.1 Calibrate the Lachat flow injection analyzer according to the manufacturer's instructions.

10.2.2 Connect the chloride board in the appropriate channel position.

10.2.3 Using the six calibration standards and a DI blank (see Section 8.1.1.1 to 8.1.1.7), calibrate the instrument. A correlation coefficient ≥ 0.995 must be achieved using a second order quadratic calibration, otherwise recalibration will be necessary. This can be done by re-analyzing the standards used previously, or preparing the standards again. The calibration curve is prepared by plotting the peak areas of each standard processed through the manifold against the true chloride concentrations in each standard.

All calibration points are back calculated by Lachat software and should be within 10% from true concentration, except lowest point of calibration curve. %r recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse than 0.995.

10.2.4 **Initial Calibration Verification (ICV/LCS):** Prior to sample analysis, the calibration curve is checked at low, mid and high ends of the curve with 5ppm LCS, 30ppm LCS and 80ppm LCS respectively (Sections 8.5.1 through 8.5.3). Recovery for each standard must be within ±10% of their true value, otherwise recalibration is necessary.

10.2.5 **Auto dilutor check:** Prior to sample analysis, following ICV verification, auto dilutor check has to be performed (if dilutor will be used during analytical run). Analyze 100 mg/l calibration standard (8.1.1.1) using 10x auto dilution function. Recovery for

standard must be within ±10% of true value, otherwise repeat auto dilution. If recovery still will be outside of 10%, then recalibration is necessary. If after re-calibration recovery still will be outside of 10%, auto dilution function cannot be used and manual dilutions must be performed when needed.

10.3 Equipment Operation and Sample Processing

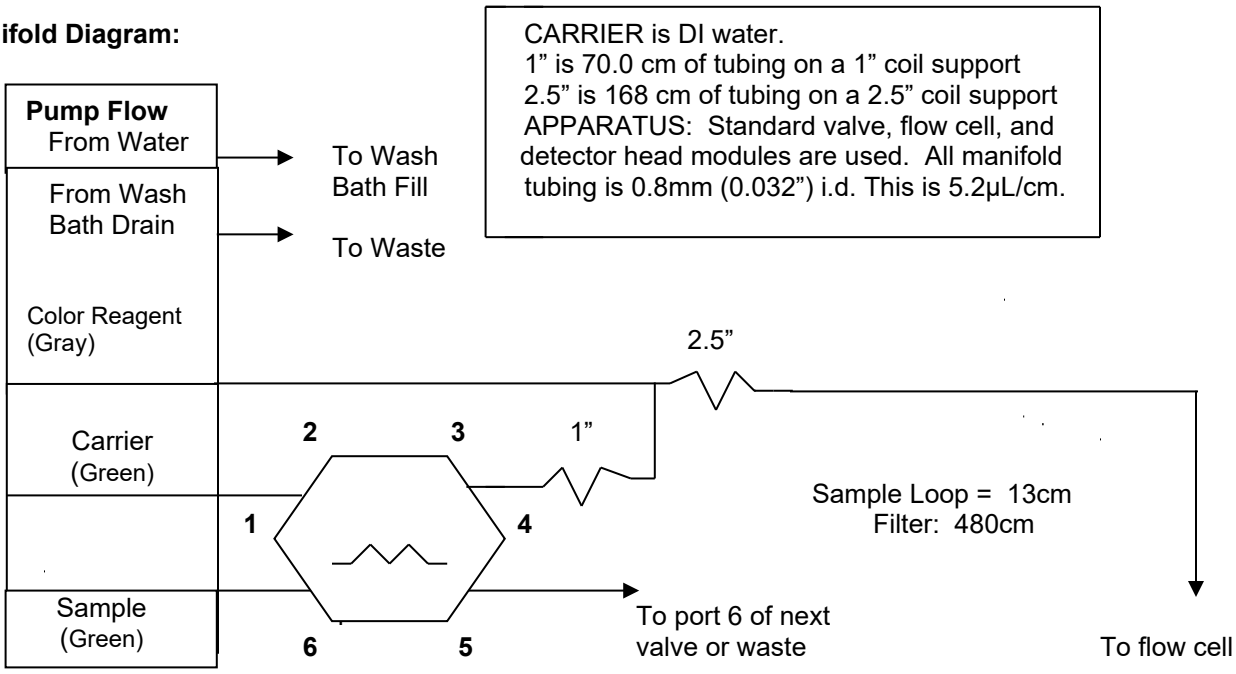
10.3.1 Follow the instrument manual for instrument operating instructions. Water samples should be poured in sample tubes for analysis. If sample is turbid or has sediment, sample has to be filtered prior analysis using 0.45 nm acrodisk filters. For soil samples: weigh 5 g of sample (record exact weight in log book), add 50 ml of Di stir for 30 min, filter and analyze water portion.

Note: 1:10 ratio should be used for soil:water extraction

10.3.2 The following are the specific parameters for this method:

Sample throughput:	120 samples/h; 30 s/sample
Pump speed:	35
Cycle period:	30 s
Inject to start of peak period:	8 s
Inject to end of peak period:	30 s
System IV Gain Setting	149 x 1
Presentation, Data Window	
Top Scale Response:	1.00
Bottom scale response:	0.00

Manifold Diagram:



10.3.3 A volumetric dilution is performed on any sample that has a result greater than 100ppm (the highest point on the calibration curve), so that the sample concentration is near the midpoint of the curve. All dilution factors are recorded.

10.3.4 Any sample with a result >1ppm, that follows a sample with a result of 100ppm or greater, should be considered suspect due to possible carry-over contamination and must be reanalyzed.

10.4 Continuing Calibration

Before sample analysis begins, after every 10 samples, and at the end of the sample run, the following must be analyzed:

- DI blank (Section 8.1.1.7)
- 20ppm calibration working standard (CCV) (Section 8.1.1.3)

The blank must yield a non-detectable result and the standard must be recovered within ±10% of its true value. If recoveries fall outside of this range, the cause for the failure is determined and corrected. When the CCV fails low, the CCV is re-made and re-analyzed. If failure continues, all samples analyzed since the last valid CCV, are considered invalid and must be re-analyzed. However, prior to re-analysis, a new calibration curve must be generated. If the CCV fails high, and all sample are considered ND, then the failure is narrated.

10.5 Preventative Maintenance

10.5.1 All lines are flushed with DI at the end of each run.

10.5.2 Tubing is replaced as necessary.

10.5.3 All equipment is kept clean.

10.5.4 All maintenance is recorded in the Instrument Maintenance Logbook.

11. Data Evaluation, Calculations and Reporting

Prepare standard curves by plotting peak heights of standards processed through the manifold against chloride concentrations in standards. The Lachat software will compute the sample chloride concentration by comparing the sample peak height with the standard curve.

The analyst has two options for final calculation: 1) Manually multiply the Lachat result times the dilution factor, if any, or 2) Input the dilution factor into the Lachat software and allow it to compute the corrected results.

Note: Chloride calibration curve is not linear; it's second order quadratic calibration. To ensure that result is accurate toward high end of the calibration curve, all samples with concentration over 90 mg/l, have to be re-analyzed with dilution.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

The quarterly method reporting limit (MRL) verification requirement is satisfied by the quarterly LOQ analysis, as per SOP/1732.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734 and 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1734,1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

Technical Information

Reference Number:	ASTM D4318-17
Test Method Title:	Standard Test Method for Liquid Limit, Plastic Limit and Plasticity Index of Soils
Test Property:	Plasticity of soils
Test Specimen Size:	150-200 grams of material passing the #40 sieve 100 grams of as received soil for Moisture Content Determination
Number of Test Specimens:	1 (representative sample obtained by quartering, mixing or splitting)
Test Equipment:	Liquid limit device Flat grooving tool Drying containers Balance readable to 0.01 gram with 500 gram capacity Ground glass plate Spatula #40 sieve Wash bottle Distilled Water Drying oven capable of maintaining 110 $\pm$ 5 °C Drying Plate/vacuum pump apparatus Soil Blender with mixing cup

Standard Operating Procedure

The Atterberg Limit Test requires the As Received Moisture content determination to be reported. If no other assigned testing requires the As Received Moisture content determination, follow ASTM D 2216 and obtain the result for the sample.

GTX requires a daily IRM check of the equipment to include, but is not limited to: the wear of the grooving tool, the drop height of the Liquid Limit device cup and the wear spot on the base under the cup at the point of impact of the Liquid Limit device. Do not use equipment that is not comply with the IRM tolerances, found on the IRM log.

SAMPLE PREPARTION (wet method)

1. If sample has little or no material retained on the #40 sieve, prepare specimen of 150-200 g by mixing thoroughly with distilled water on glass plate or mixing dish using a spatula. If desired, soak the material in a mixing/storage dish with a small amount of water to soften the material before the start of mixing. Adjust the water content of the soil and mix again to bring to a consistency that would require between 25-35 blows. Remove >#40 sieve material by hand if small percentage is present. If water has been added to specimen place in a covered dish and allow to cure for a minimum of 16 hours.

2. If large percentage of >#40 sieve material is present, obtain a large enough specimen which will contain 150-200 g of material passing the #40 sieve. Place specimen in mixing cup and cover with distilled water. Mix into slurry, pass specimen through a #40 sieve and discard >#40 material. Reduce the amount of water using the drying plate/vacuum pump apparatus. Do not allow sample to over dry. Dry until sample is at a consistency that would require between 25-30 blows. Water has been added to the specimen so place in a covered dish and allow to cure for a minimum of 16 hours.

SAMPLE PREPARATION (dry method)

1. If sample is received dry, pass the material through a #40 sieve, and adjust the moisture content and mix thoroughly to bring the soil to a consistency that would require between 25-35 blows. Place specimen in a covered dish and allow to cure for a minimum of 16 hours.

LIQUID LIMIT DETERMINIATION (multipoint method)

1. Before starting test, remix thoroughly in its dish adjusting the water content slightly, as necessary.

2. Fill the liquid limit cup even with front rim, to a depth that allows a full height cut with grooving tool. Keep the unused material covered with a wet towel in the mixing dish.

3. Form a groove in the soil from the highest point to the lowest point on the rim of the cup using the beveled edge facing forward of the grooving tool. Keep the tool perpendicular to cup from back to front by using an arc type motion. If the groove cannot be made in one stroke without tearing the soil, cut the groove in layers a little deeper with each cut, without tearing the soil.

4. Inspect the underside of the Liquid Limit Device, making sure the underside of the cup and base are clean. Lift and drop the cup at a rate of 1.9-2.1 drops per second until the two halves of the soil pat come in contact at the bottom of the groove along a distance of 0.5 inch. The base of the machine shall not be held with a hand while the crank is being turned. Record number of blows required. Take approximately 10 g of material from the closed area, perpendicular to the groove and place into a pre-weighed drying container. Measure and record mass to nearest 0.01 g. Cover the container.

5. Remove balance of material from cup and remix with soil in dish. Clean the cup.

6. By wetting the remaining material, adjust the blow counts so a distribution of blow counts is obtained. One point between 35 and 25 blows, another point between 20 and 30 blows, and a third point between 25 and 15 blows. For each point repeat steps 1 through 6.

PLASTIC LIMIT DETERMINATION

1. Obtain about 20 grams of soil prepared for the Liquid Limit test and dry by blotting with paper towels until it can be rolled without sticking to the glass plate.

2. To determine plastic limit, roll three to four threads of 1.5-2.0 grams of material down to a 1/8 inch diameter for a total sample weight of at least 6 grams.

3. Roll the material on a glass plate with the palm of your hand until a thread of uniform diameter of 1/8 inch is obtained (80-90 strokes per minute). This should take no longer than 2 minutes.

4. Ball up and repeat step 3 again. Repeat until 1/8 inch diameter cannot be obtained (thread crumbles under the pressure required for rolling). The only requirement for continuing is that the threads are able to be reformed into a ball and rolled out again.

5. Gather portions of the crumbled thread together and place into a pre-weighed container. Measure and record mass to nearest 0.01 g.

6. Repeat steps 2 through 5 one more time.

7. Dry all materials to a constant mass in oven with the temperature required for the material type being tested. Weigh again to the nearest 0.01 g.

8. Calculate the plasticity index as follows:

   PI = LL-PL

   where:

   PI = Plasticity Index
   LL = Liquid Limit
   PL = Plastic Limit

9. If either the liquid limit or plastic limit could not be determined, or if the plastic limit is equal to or greater than the liquid limit, report the soil as non-plastic.

10. Report: sample identification, as received moisture content, sample preparation procedure, liquid limit, plastic limit and plasticity index to the nearest whole number, and non-plastic if applicable.

Determination of Inorganic Anions by Ion Chromatography

Reference: **EPA 300.0,** Methods for the Determination of Inorganic Substances in Environmental Samples, EPA/600/R-93/100, August, 1993.

Method 9056A, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision IV, February 2007

1. Scope and Application

Matrices: Drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (after extraction) leachates (when no acetic acid is used).

Definitions: Refer to Alpha Analytical Quality Manual.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the ion chromatography and in the interpretation of ion chromatography data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Parameter	CAS
Bromide	24959-67-9
Chloride	16887-00-6
Fluoride	16984-48-8
Nitrate – N	14797-55-8
Sulfate	14808-79-8

2. Summary of Method

A small volume of sample is introduced into an ion chromatograph. The anions are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

2.1 Method Modifications from Reference

Use of other eluents that improve method performance are minor modifications of the method and are considered by the method to be acceptable.

3. Reporting Limits

The laboratory follows the procedure found in 40CFR Part 136 to determine the MDL on a semi-annual basis.

Parameter	RL
Bromide	.05 mg/L
Chloride	.5 mg/L
Fluoride	.05 mg/L
Nitrate – N	.05 mg/L
Sulfate	1 mg/L

4. Interferences

4.1 Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or fortification can be used to solve most interference problems associated with retention times.

4.2 Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

4.3 Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

4.4 Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5mg/L, this interference may not be significant, however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

4.5 The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

4.6 The quantitation of unretained peaks should be avoided, such as low molecular weight organic acids (formate, acetate, propionate, etc.) which are conductive and coelute with or near fluoride and would bias the fluoride quantitation in some drinking and most waste waters.

4.7 Any residual chlorine dioxide present in the sample will result in the formation of additional chlorite prior to analysis. If any concentration of chlorine dioxide is suspected in the sample, purge the sample with an inert gas (argon or nitrogen) for about five minutes or until not chlorine dioxide remains.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

Sulfuric acid used in this method has the potential to be highly toxic or hazardous.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass or plastic bottles of sufficient volume to allow replicate analyses of the anions of interest.

6.2 Sample Preservation

Samples are refrigerated at 4°C.

6.3 Sample Shipping

No specific requirement.

6.4 Sample Handling

The sample holding time is 48 hours for the following anions: Nitrate –N.

The sample holding time is 28 days for the following anions: Bromide, Chloride, Fluoride, and Sulfate

7. Equipment and Supplies

7.1 Balance: Analytical, capable of weighing to 0.0001g.

7.2 Ion Chromatograph: Analytical system (Dionex ICS-2000 or Dionex Integrion HPLC) complete with ion chromatograph and all required accessories including syringes, autosampler, analytical columns, compressed gasses and detectors.

7.2.1 Anion guard column: AG-18 (Dionex PN 060551) or AG-18-Fast (Dionex PN 076035) A protector of the separator column. If omitted from the system the retention times will be shorter. Usually packed with a substrate the same as that in the separator column.

7.2.2 Anion analytical column: AS-18 (Dionex PN 060549) or AS-18-FAST (Dionex PN 076034) . This column produces the separation shown in Figure 1.

7.2.3 Anion supressor: ASRS Ultra II 4mm (PN 061561). The supressor column is packed with a high capacity cation exchange resin that is capable of converting the eluent and separated anions to their respective acid forms.

7.2.4 Detector: DS6 (PN 057985) Temperature controlled, heated conductivity cell

7.2.5 Eluent Generator: EG40 (Dionex PN 058900) Prepares the eluent electronically, controlled by the software; equipped with KOH cartridge.

7.3 Software: The Dionex IC Instrument uses Chromeleon Software.

7.4 0.45µm Membrane Filter Syringes.

7.5 Volumetric Flasks: Various volumes.

7.6 Volumetric Pipets: Various volumes.

7.7 0.5mL Vials with Caps: Dionex PN 038142

7.8 Vial kit: 30 ml PP w/Slit septum (Dionex PN 055428)

8. Reagents and Standards

Note: All analytical standards used for calibration and calibration verification must be traceable to NIST. Each standard is recorded in a Logbook and unique ID is assigned to each standard. The unique IDs must also be included in all analytical sequences.

8.1 Reagent Water: Deionized water, free of the anions of interest. Water should contain particles no larger than 0.20 microns.

8.2 Eluent Solution: 32mM KOH, Prepared by the Eluent Generator.

8.3 Stock Calibration / ICV Standard Solutions, 1000mg/L (1mg/mL): Stock standards for all analytes are usually purchased as certified solutions. Certificates of analysis are kept on file.

However, if it is necessary, the Stock Solutions may be prepared from ACS reagent grade materials (dried at 105°C for 30 minutes) as listed below. The ICV Standards must be prepared from a different source than the calibration standards.

NOTE: Stock calibration/ ICV standards are stable for at least six months when stored at 4°C. Dilute working standards are prepared weekly.

8.3.1 Standard 1: Fluoride Stock Standard, 1000mg F^-/L

In a 250mL volumetric flask, dissolve 0.5526g of sodium fluoride (NaF, CASRN 7681-49-4) in about 200mL reagent water. Dilute to the mark with reagent water, and invert to mix.

8.3.2 Standard 2: Fluoride Stock Standard, 100mg F^-/L

In a 250mL volumetric flask, pipet 25mL of Standard 1, dilute to the mark with reagent water, and invert to mix.

8.3.3 Standard 3: Chloride Stock Standard, 1000mg Cl^-/L

In a 250mL volumetric flask, dissolve 0.4121g of sodium chloride (NaCl, CASRN 7647-14-5) in about 200mL reagent water. Dilute to the mark with reagent water, and invert to mix.

8.3.4 Standard 4: Bromide Stock Standard, 1000mg Br^-/L

In a 250mL volumetric flask, dissolve 0.3219g of sodium bromide (NaBr, CASRN 7647-15-6) in about 200mL reagent water. Dilute to the mark with reagent water, and invert to mix.

8.3.5 Standard 5: Bromide Stock Standard, 100mg Br^-/L

In a 250mL volumetric flask, pipet 25mL of Standard 4, dilute to the mark with reagent water, and invert to mix.

8.3.6 Standard 8: Nitrate Stock Standard, 1000mg NO_3^--N /L

In a 250mL volumetric flask, dissolve 1.5170 g of sodium nitrate ($NaNO_3$, CASRN 7631-99-4) in about 200mL reagent water. Dilute to the mark with reagent water, and invert to mix.

8.3.7 Standard 9: Nitrate Stock Standard, 100mg NO_3^--N /L

In a 250mL volumetric flask, pipet 25mL of Standard 8, dilute to the mark with reagent water, and invert to mix.

8.3.8 Standard 12: Sulfate Stock Standard, 1000mg SO_4^{2-} /L

In a 250mL volumetric flask, dissolve 0.4535 g of anhydrous dibasic, potassium sulfate (K_2SO_4, CASRN 7778-80-5) in about 200mL reagent water. Dilute to the mark with reagent water, and invert to mix.

8.3.9 Stock Bromide Standard, 1000ppm

Commercially available. Certificate of analysis required and kept on file. Use separate sources for the ICV and Calibration standards listed below.

8.3.9.1 ICV Bromide Standard, 100ppm

In a 100mL volumetric flask, add 1mL of Stock 1000ppm Bromide standard (Section 8.4.13). Bring to volume with reagent water.

8.4 Working Mixed Stock Standard A (Calibration Stock)

In a 200mL volumetric flask, transfer using volumetric pipets, 2mL each of Standards Bromide 1000ppm and Nitrate 1000ppm; 20mL of Chloride 1000ppm and Fluoride 100ppm (Section 8.3). Dilute to the mark with reagent water and invert to mix. Store at 4 ± 2°C for up to one month.

This makes Standard A containing F^-, Cl^-, NO_2^- -N, Br^-, NO_3^- -N and SO_4^{2-} at the concentrations of 10, 100, 10, 10 and 200ppm respectively.

8.4.1 Analyte Matrix Spike Solution

Volumetrically prepare the spike solution by bringing 1.0 mL of the calibration stock standard (Section 8.4) up to a 25mL final volume with the sample.

8.5 Working Mixed Standards B through G (Calibration Curve)

Working mixed standards B through F are prepared by diluting Standard A as summarized in the following Table. These are prepared fresh as needed for calibration.

Std.	Std. A (mL)	Final Vol. (mL)	F^-	Cl^-	Br	NO_3^- -N	SO_4^{2-}
B	5	10	5.0	50.0	5.0	5.0	100.0
C	5.0	25	2.0	20.0	2.0	2.0	40.0
D	1.25	25	0.5	5.0	0.5	0.5	10.0
E	0.5	25	0.2	2.0	0.2	0.2	4.0
F	1 of Std D	10	0.05	0.5	0.05	0.05	1.0
G	0.5 of Std D	10	0.025	0.25	0.025	0.025	0.5

Example: To make up Standard B, take 25mL of Standard A in a 10mL volumetric flask and dilute to the mark with reagent water.

Note: The dynamic range for the method is two orders of magnitude. The concentrations for the standards could be changed to bracket the concentrations of the samples to be analyzed.

8.6 ICV Stock Standard (Second Source Verification)

To a 200mL volumetric flask add 20mL of the following standards: Chloride 1000ppm and Sulfate 1000ppm, and 2mL of Fluoride 1000ppm, Bromide 1000ppm and Nitrate 1000ppm (Section 8.3). Dilute to volume with reagent water.

8.6.1 ICV Working Standard / LCS Solution

In a 25mL volumetric flask, add 2mL of the ICV Stock Standard. Bring to volume with reagent water. Store at 4 ± 2°C. Prepare weekly.

ICV working standard will have the following concentrations: 0.8 mg/L for Fluoride, Nitrate and Bromide; 8.0 mg/L for Chloride and Sulfate.

8.7 CCV Working Solution

The CCV Working Solution is made by diluting 2.5 ml of Mixed Stock Standard A up to 25 ml of DI Section 8.4. Final concentrations are: 1.0 mg/L for Fluoride, Nitrate and Bromide; 10.0 mg/L for Chloride and 20.0mg/l for Sulfate. Store at 4 ± 2°C. Prepare weekly.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

One Method Blank consisting of an aliquot of reagent water is analyzed with each batch of 20 samples or less. Data produced are used to assess contamination from the laboratory environment. Method Blank results must be less than the Reporting Limit (RL) for the analyte.

Note: If samples have to be filtered prior to analysis, all associated batch QC must also be filtered.

9.2 Laboratory Control Sample (LCS)

The ICV/LCS is analyzed at the beginning of each run, but after the CCV standard. This standard is prepared from a different source than that used to prepare the calibration standards (Section 8.6). The % Recovery of this standard must be within ± 10% of the calibration standard.

9.3 Initial Calibration Verification (ICV)

See Section 9.2.

9.4 Continuing Calibration Verification (CCV)

The CCV Standard is the equivalent of Standard D in Section 8.5. Store at 4 ± 2°C. Prepare weekly. The CCV is analyzed at the beginning of each run, after every tenth sample, and at the end of the sample run. The % Recovery of this standard must be within ± 10% of the calibration standard. If the Continuing Calibration cannot be verified within the specified limits, reanalyze the CCV solution. Record the reason for re-injection. If the second analysis of the CCV solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable CCV solution must be reanalyzed. The analysis data of the calibration blank and CCV solution must be kept on file with the sample analyses data

9.5 Matrix Spike

Prepare and analyze one spiked sample per batch of 10 samples or less (Section 8.4.1). Recovery of the Matrix Spike must be within +/-10% of the true value for method 300 and +/-20% for method 9056.

9.6 Laboratory Duplicate

Prepare and analyze one duplicate sample per batch of 20 samples or less. The RPD for the duplicate measurements must be ≤ 15%.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Instrument calibration
- Rinse
- CCV
- CCB
- ICV
- ICB
- Blank
- LCS
- Six samples
- CCV
- CCB
- Ten samples
- CCV
- CCB
- Shut-down

10. Procedure

10.1 Equipment Set-up

10.1.1 Determination of Linear Calibration Range (LCR)

The LCR must be determined initially and verified every six months or whenever a significant change in instrument response is observed or expected. The initial demonstration of linearity must use sufficient standards to ensure that the resulting curve is linear. The verification of linearity must use a minimum of a blank and three standards. If any verification data exceeds the initial values by ± 10%, linearity must be

reestablished. If any portion of the range is shown to be nonlinear, sufficient standards must be used to clearly define the nonlinear portion.

10.1.2 Prime Pump: The pump must be primed prior to analysis, to ensure that there is no gas entering the column.

10.1.3 Monitor Baseline: From the main Panel screen, press the "Startup" Button. This will turn on, in order, the pump, the eluent generator, and the conductance cell. Allow the instrument to warm up for 10 – 20 minutes to ensure the baseline is stable and flat.

10.1.4 While the baseline stabilizes, the sample sequence can be written and the autosampler may be loaded.

10.1.5 When the baseline is stabilized, the sample sequence may be loaded into the analytical run, and started from the Chromeleon software.

10.1.6 Operating Conditions: Dionex IC Instrument

Eluent Concentration: 32mM KOH

Flow rate: 1.0mL / minute

Injection volume: 100µL

ASRS: ON

Conductivity Cell Temperature: 30 °C

10.1.7 Monitor instrument stability: Prior to QC sample and sample analysis, analyze a DI water blank to ensure the instrument is stable.

10.1.8 Sample filtration: Autosampler vials are equipped with a filter. If additional filtration is necessary, samples may be filtered through a 0.45µm membrane filter attached to a syringe. **NOTE:** If samples require filtration, all associated batch QC samples must also be filtered.

10.1.9 Extraction of solid materials: Add a volume of reagent water equal to 10 times the weight of dry solid material taken as a sample. This slurry is mixed for 10 minutes using a magnetic stirring device. Filter the resulting slurry before injecting using a 0.45µ membrane filter attached to a syringe. Care should be taken to show that good recovery and identification of peaks is obtained with the user's matrix through the use of matrix spikes (Section 9.5).

10.2 Initial Calibration

For each analyte of interest, prepare calibration standards at a minimum of three concentration levels and a blank by adding accurately measured volumes of one or more stock standards (Section 8.5) to a volumetric flask and diluting to volume with reagent water. If a sample analyte concentration exceeds the calibration range, the sample may be diluted to fall within the range.

Using injections of 100µL of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte. During this procedure, retention times must be recorded.

The calibration curve for each analyte is prepared by plotting instrument response against the standard concentration. A correlation coefficient of 0.995 or greater is considered acceptable for all analytes.

All calibration points are back calculated by Dionex software and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 50% and correlation coefficient will not be worse than 0.995.

If calibration curve criteria failed, then new calibration standards have to be re-analyzed and new calibration curve has to be generated and all calibration points have to be recalculated to meet criteria.

If second calibration curve fails acceptance criteria, then analysis have to stopped, problem has to be identified; new calibration standards made and analyzed.

10.2.1 Initial Calibration Verification (ICV/LCS): The calibration curve must be verified on each working day, and after every 20 samples. The ICV/LCS sample is prepared from a different source than that used for the calibration standards (Section 8.6). If the response or retention time for any analyte varies from the expected values by more than ± 10%, the analysis must be repeated, using fresh calibration standards. If the results are still more than ± 10%, a new calibration curve must be prepared for that analyte.

10.3 Equipment Operation and Sample Processing

10.3.1 An automated constant volume injection system is used. Load and inject a fixed amount of well-mixed sample. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units.

10.3.2 The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.3.3 If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of reagent water and reanalyze.

10.3.4 If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

Note: Retention time is inversely proportional to concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases this peak migration may produce poor resolution or identification.

10.4 Continuing Calibration

This standard (Standard D: Section 8.5) is prepared weekly. The CCV is analyzed at the beginning of each run, after every tenth sample, and at the end of the sample run. The % Recovery of this standard must be within ± 10% of the calibration standard. Refer to Section 9.4 if % Recovery falls outside of the acceptance range.

10.5 Preventive Maintenance

Follow the Preventative Maintenance Schedule as outlined on the Dionex ICS-2000 CDROM.

As Needed

- Check the eluent reservoir to see if it needs to be refilled.

Daily

- Check the ICS-2000 component mounting panel for leaks or spills. Wipe up spills. Isolate and repair leaks. Rinse off any dried eluent with reagent water.

- Check the waste container daily and empty when needed.

Weekly

- Once a week, check fluid lines for crimping or discoloration. Relocate any pinched lines. Replace damaged lines.
- Check the junctions between the pump heads and the pump casting for evidence of liquid leaks. If piston seal wash tubing is not connected, check the drain tubes at the rear of the pump heads for evidence of moisture. Normal friction and wear may gradually result in small liquid leaks around the piston seal. If unchecked, these leaks can gradually contaminate the piston housing, causing the pump to operate poorly. If leaks occur, replace the piston seals.

- Check the end-line filter (PN 045987) and change if needed. When new, end-lline filters are pure white. If the system is in continuous operation, change the end-line filter weekly, or whenever it becomes discolored. Replace the filter more often if bacterial buildup is visible or if the eluent does not contain solvent.

> **NOTE:** It is especially important to regularly replace end-line filters when using aqueous eluents, which may contaminate the filter with bacteria or algae. The bacterial buildup may not be visible.

Yearly (performed by Dionex technician)

- Calibrate the cell.
- Calibrate the vacuum degas assembly
- Replace the pump piston rinse seals and piston seals

11. Data Evaluation, Calculations and Reporting

11.1 Compute the sample concentration by comparing sample response with the standard curve. Multiply the result by the appropriate dilution factor.

11.2 Report only those values that fall between the lowest and the highest calibration standards. Samples exceeding the highest standard should be diluted and reanalyzed.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

If the Method Blank result exceeds the Reporting Limit (RL) for the analyte, the Blank is reanalyzed. If the second result remains > RL, notify the Laboratory Manager to ensure maintenance is performed on the water filtration system and seek an alternate reagent water source within the laboratory. If the alternate reagent water source is acceptable, this source must be utilized for all blanks, standards and sample dilutions for the sample batch. If the second source reagent water also fails, the Laboratory Manager is notified.

If the Continuing Calibration cannot be verified within the specified limits, reanalyze the CCV solution. Record the reason for re-injection. If the second analysis of the CCV solution confirms calibration to be outside the limits, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable CCV solution must be reanalyzed. The analysis data of the calibration blank and CCV solution must be kept on file with the sample analyses data.

If the ICV/LCS acceptance criterion cannot be met, reanalyze the standard. If failure continues, the instrument is recalibrated.

If the Matrix Spike acceptance criteria is not met, the spiked sample is reanalyzed (if possible). If failure continues and if all other QC performance criteria are met, the data is reported and a narrative is included with the final report.

If the RPD for the Duplicate measurements falls outside the Laboratory defined control limits, the sample is reanalyzed (if possible). If failure continues, and if all other QC performance criteria are met, the data is reported and a narrative is included with the final report.

Holding time exceedance and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CCV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

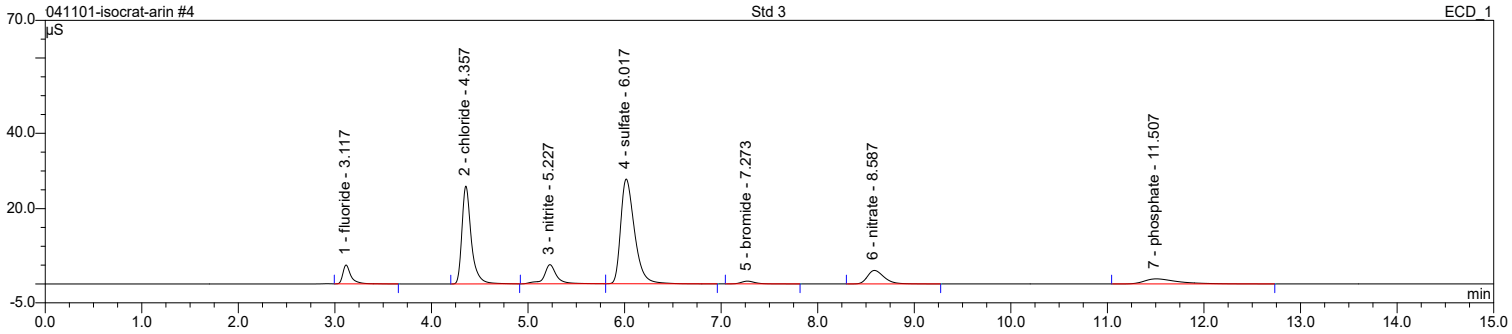
1728 Hazardous Waste Management and Disposal SOP

16. Attachments

Figure 1: Isocratic Anion Standard Separation

Figure 1

Isocratic Anion Standard Separation



Determination of Organochlorine Pesticides By Gas Chromatography/Electron Capture Detection (GC/ECD)

References: Method 8081B Rev. 2, Update IV, Feb 2007, and Method 8000C Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Final Update III, December 1996 (USEPA, Office of Solid Waste and Emergency Response, Washington, DC).

USEPA Contract Laboratory Program Statement of Work for Organics Analysis Multi-Media, Multi-Concentration" August 1991. Document Number OLM03.1 and OLM04.2.

Massachusetts Contingency Plan (MCP) WSC-CAM for SW846 Method 8081 Pesticides by GC/ECD. Section VB Rev4. August 2004 (Massachusetts Department of Environmental Protection Bureau of Waste site cleanup)

1. Scope and Application

Matrices: This method is applicable to the quantification of Organochlorine Pesticides in water, soil, sediment, sludge, product, and tissue (either animal or vegetative).

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the analysis and quantification of sample extracts for Organochlorine Pesticides by gas chromatography/electron capture detector. Target analytes include a wide range of pesticides, a basic list of which is included below. The sensitivity of the method usually depends on the concentration of interferences as well as instrumental limitations. If interferences prevent detection of the analytes, the method may also be performed on samples that have undergone cleanup. Typical reporting limits are based on the lowest level of the initial calibration curve and generally correspond to limits derived from quarterly LOQ studies to verify our MDLs.. Detection limits will vary with the individual sample matrix, sample preparation procedures, instrument calibration range, and volume of sample analyzed.

This method is in substantial conformance with the guidelines established in SW-846 for performing chromatographic analysis as defined in the Method 8000B ("Gas Chromatography), Method 8081B ("Organochlorine Pesticides"), Method 3510C ("Separatory Funnel Liquid - Liquid Extraction"), and Method 3570 Microscale Extraction. The data quality objectives of these methods are met or exceeded by this SOP. The following extraction and cleanup methods may also apply, depending on project specifications:

- Extraction of Water Samples by Separatory Funnel (2156)
- Tissue Preparation and Homogenization (2166)
- Shaker Table Extraction (2261)
- Sulfur Cleanup (2168)
- Microscale Solvent Extraction - 3570 (2172)
- Silica cleanup (2170)
- GPC Cleanup by HPLC (2167)

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory

personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/ECD and in the interpretation of GC/ECD data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Table 2: Target Analytes for Pesticides by GC/ECD

Parameter	CAS
alpha-BHC	319-84-6
beta-BHC	319-85-7
gamma-BHC (Lindane)	58-89-9
delta-BHC	319-86-8
Heptachlor	76-44-8
Aldrin	309-00-2
alpha-Chlordane	5103-71-9
gamma-Chlordane	5103-74-2
Heptachlor epoxide	1024-57-3
Oxychlordane	27304-13-8
Dieldrin	60-57-1
Endrin	72-20-8
Endrin aldehyde	7421-93-4
Endrin ketone	53494-70-5
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
2,4'-DDD	53-19-0
2,4'-DDE	3424-82-6
2,4'-DDT	789-02-6
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan sulfate	1031-07-8
Methoxychlor	72-43-5
Mirex	2385-85-5
Cis-Nonachlor	5103-73-1
Trans-Chlordane	5103-74-2
Hexachlorobenzene	118-74-1
Toxaphene	72-43-5
Chlordane	57-74-9

2. Summary of Method

An aliquot of a well mixed, homogeneous aqueous, solid, or tissue sample is accurately measured or weighed for sample preparation. Generally, 500mL or 1000mL of water sample, 1-5g of tissue sample, or 5g to 30g of sediment/soil sample is extracted. Water, soil/sediment, and tissue samples are spiked with surrogate compounds and then extracted using methylene chloride or a methylene chloride/acetone mixture. The extract is dried and exchanged into hexane during sample concentration. After extraction, cleanup techniques are applied. Generally extracts are cleaned by GPC (for hydrocarbon or lipid removal) and silica cartridge or automated silica column followed by copper (for sulfur removal). The extract is exchanged into hexane and concentrated to the appropriate volume, generally 5mL or 10mL, for analysis. Alternatively, this method can be employed for lower detection limits by decreasing the final volume to 1-5 mL.

After cleanup and re-concentration, the extracts are spiked with internal standards, and analyzed on a gas chromatograph, which is fitted with two capillary columns of differing polarities each employing a separate detector. The target analytes are resolved on each column and detected using an electron capture detector (ECD). Analytes are introduced into the GC/ECD by injecting a known volume of the calibration standards, quality control samples, and sample extracts into the GC that is temperature and flow programmed to separate the compounds of interest. Identification of target analytes is accomplished by confirming a target hit on two dissimilar columns using Retention Time and Pattern Recognition. Concentrations are calculated from the ECD response using internal standard techniques, additionally external standard techniques can also be utilized circumstantially. Concentrations are determined using Mean Relative Response Factor from a multi-level calibration curve. Response factors for target analytes and surrogate compounds are determined relative to the internal standards.

2.1 Method Modifications from Reference

This method exhibits some modification from the reference method. Method 8081B discusses the allowed shelf life for stock solutions as one year and working solutions as six months. The standard expiration protocol for the lab is the same however, if there is a significant amount of working spike or surrogate solution expired after the six-month period the lab will re-QC the solution. If the QC of the solution still passes the lab will extend the expiration date by another month. The method also indicates that a mid level multi-component standard is analyzed with the initial calibration for multi component compounds such as Toxaphene and Chlordane. The current lab procedure is to run the lowest possible level of the multi-component standard and include it with the Initial Calibration so the analyst can determine the lowest level at which the multi-component targets can be detected. If the multi-component analyte is then detected in a sample extract the analyst will then analyze a mid level standard for a single-point quantitation.

3. Reporting Limits

Table 1 lists the routine reporting limits.

4. Interferences

4.1 Contaminants in solvents, reagents, glassware, syringes, and other sample processing hardware may cause inferences that lead to discrete artifacts, biases, and/or elevated baselines. Demonstrate that all of these materials are free from interferences under the conditions of the preparation and analysis by extracting and analyzing a laboratory method blank with each batch of up to 20 samples. Also new solvent lots are to be prescreened and approved by GC/ECD analyst prior to use. All glassware and equipment should be

properly cleaned and solvent rinsed prior to use. Never use any glassware, or other implements such as syringes, on multiple samples within the batch unless there has been thorough cleaning and decontamination between samples.

4.2 Contaminants co-extracted from the sample may cause matrix interferences. The extent of matrix interferences will vary considerably from sample to sample, depending upon the nature of the environment and matrix being investigated. In some cases depending on the severity of the observed interferences, a target hit on one column may be biased high when interfered with. Skilled analyst judgment is imperative in determining if a chromatographic peak appears interfered with. In these cases the non-interfered, non-biased hit can be forced for reporting purposes providing a more suitable value. In extreme cases of interference additional cleanups can be employed, or it may be necessary to analytically re-run the sample at a dilution.

4.3 The ECD is theoretically a halogen specific detector. However, phthalate esters can be a major source of contamination in that they do respond to the ECD and can interfere with accurate determination of the compounds of interest. No forms of plastic or any material containing plasticizers (phthalates) should be used in conjunction with any part of this analysis.

4.4 Elemental sulfur is readily extracted from soil and sediment samples and may cause chromatographic interferences in the determination of pesticides. Sulfur can be removed through the use of Method 3660B.

4.5 Care must be taken to ensure a complete hexane exchange is performed on the extract, because residual methylene chloride would be an interferent in the analysis. Residual methylene chloride can also damage the electron capture detectors.

4.6 The injector system of the gas chromatograph can become contaminated with non-volatile components from samples. This can act to decrease the resolution and responsiveness of the system. The analysis of a DDT/Endrin breakdown standard is the best indicator of the condition of the injector system and the condition of the column. As the percent breakdown increases gradually the system should undergo general maintenance (liner changes, injection port cleaning, etc.) to avoid prolonged down time.

4.7 Aldol condensation products (4-methyl-4-hydroxy-2-pentanone and 4-methyl-3-pentene-2-one) can arise during soil extraction when using methylene chloride: acetone. These products may interfere with detection of analytes and should be monitored.

4.8 Raw GC/ECD data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples should be collected in 500mL, 1L or 2L amber glass bottles and stored without preservative at 4°C. The minimum amount of sample needed to reach the reporting limits on Table 1 in Section 16 for this method for aqueous samples is 500mL. Additional sample is needed (approximately 3X the minimum amount) if MS/MSD analyses are to be performed.

Soil/sediment and Tissue samples should be collected in glass soil jars and stored at 4°C, or if desired, frozen. The minimum amount of sample needed to reach the reporting limits on Table 1 in Section 16 for solid and tissue matrices is 50g. Additional sample is needed (approximately 3X the minimum amount) if MS/MSD analyses are to be performed.

6.2 Sample Preservation

Aqueous samples should be stored without preservative at 4°C.

Soil/sediment and Tissue samples should be stored at 4°C, or if desired, frozen.

6.3 Sample Shipping

Sample Receipt & Login SOP 1559 describes how samples are normally shipped or obtained by the laboratory, precautions to be used in opening sample shipments, and sample storage conditions.

6.4 Sample Handling

The hold time for this method is 7 days for the extraction of aqueous samples and 14 days for the extraction of solid and tissue samples. If a sediment or tissue samples are frozen, this suspends the holding time until removal from the freezer. All extracts must be analyzed within 40 days of the extraction date. Extracts are stored in a refrigerator at 2-6ºC.

7. Equipment and Supplies

7.1 Gas Chromatograph: The instrumentation is a temperature-programmable gas chromatograph with heating range from 40ºC to 325ºC and splitless-type inlet system with electron capture detectors (Hewlett-Packard HP 5890 Series II GC, HP 6890 Plus, or similar).

7.2 Injection Port: Designed for splitless injection onto a capillary column. The injection port utilizes a uniliner for direct injection, to decrease the potential for breakdown of compounds in the injection port and to increase sensitivity. These uniliners are used in place of the standard splitless liners. The 6890 model requires a drilled uniliner due to Electronic Pressure Control (EPC). The injection port will require maintenance on an as needed basis if degradation or contamination is apparent. Please refer to the front of the *Instrument Maintenance Logbook, which* outlines the routine maintenance procedures

7.3 Columns and supplies

7.3.1 Restek's proprietary "RTX-5" Stationary Phase, 60m length x 0.25mm ID, 0.25□m film thickness - Restek Corporation, PN 10226, or equivalent.

7.3.2 Restek's proprietary "RTX-CLPesticide II" Stationary Phase, 60m length x 0.25mm ID, 0.2□m film thickness - Restek Corporation, PN 11326, or equivalent.

7.3.3 Guard column, deactivated fuse silica phenyl-methyl, 5 m x 0.32 mm ID. Restek PN 10027 or equivalent.

7.3.4 flow from the guard column can be split between the two analytical columns using a Siltek MXT-Y connecter Restek PN 20396

7.4 Guard Column: The GC/ECD utilizes one injector with guard column sealed to the uniliner inside the injection port. The guard column is split by connector into the two dissimilar columns listed above. Each column is then attached to a separate detector. The Rtx-5 is always the front detector column and the CLP II is the back detector column.

7.5 Auto sampler: Adapted onto the Gas Chromatograph. Model is HP 6890 series autosampler with a GC autosampler controller and HP 5890 series autosampler with controller, or equivalent.

7.6 Computer: with Windows XP operating software utilizing HP Enviroquant G1701DA Version D.03.00 software, or equivalent.

7.7 Gases: Hydrogen, ultra high purity grade (99.9999% pure) and Nitrogen of equivalent purity. Hydrogen is generally supplied by a Proton Hogen GC 600 Hydrogen generator.

7.8 Syringes: Hamilton Gas tight, various sizes.

7.9 Glass Pasteur Pipettes

7.10 Volumetric Flasks: Class A, various sizes

7.11 Vials: 2ml, 4ml, 10ml

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. See SOP *Reagent, Solvent, and Standard Control* (1816) for additional details regarding solvent purity. All solvent expirations determined as indicated by manufacturer guidelines

Analytical Standards are stored at 2-6ºC, according to manufacturer's recommended procedure. Primary standards are discarded as indicated by the vendor expiration. Stock standards are given 1 year expiration from the preparation date or the expiration of the primary vendor solution, whichever occurs first. Working standards are given a six month expiration from the preparation date or the expiration of the primary solution whichever occurs first. If breakdown of a solution is observed the solution will be discarded. All analytical standards are prepared in Hexane. All prep standards are prepared in Acetone or Hexane.

8.1 Methylene Chloride: ACS approved, Pesticide grade.

8.2 Acetone: ACS approved, Pesticide grade.

8.3 Hexane: ACS approved, Pesticide grade.

8.4 Methanol: Purge and Trap grade.

8.5 Surrogate Spiking Solution: Two surrogates are used for this method, 2,4,5,6-tetrachloro-m-xylene (TMX) and Decachlorobiphenyl (DCB). Alternate surrogates may be employed to avoid co-elutions or when DCB is a target analyte. Example alternate

surrogates would be DBOB (4,4'-Dibromooctafluorobiphenyl) BZ-198 (2,2',3,3',4,5,5',6 Octachlorobiphenyl). Surrogate Spiking solutions are stored in 40ml pre-cleaned amber VOA vials and placed in a standards freezer in the preparatory lab. The Pest/PCB Surrogate is made from the *Pesticides Surrogate Standard Spiking Solution* (catalog # ISM-320) commercially obtained from Ultra at a concentration of 200ug/mL. The spiking solution is the "Pest/PCB Surrogate" spike, which is made by diluting the primary standard appropriately to achieve a concentration of 1000ug/L. All solutions are made volumetrically in Acetone at desired final volume. Generally 200ml of standard is made but this can vary depending on use, need, and preference. The solution must be assayed by analysis before release to the preparation lab. Generally 1ml of surrogate spiking solution is spiked into the samples and QC. Alternative solution concentration and spiking amount can be used.

8.6 LCS and Matrix spiking solution: Two Pest LCS Spiking solutions are generally used in this method. Alternatively, solutions with project specific targets may be used. The LCS spiking solutions are stored in the same manner as surrogate solutions. The Pest LCS solutions are made from a combination of Restek Pest Mix 8081AB#1 cat#32291 at concentration 200ug/ml and Hexachlorobenzene and Additional Pesticide Mix (Quote#041310-632 from Ultra at a concentration of 100ug/ml. The spiking solution is labeled "Pest LCS" spike and is prepared by diluting the appropriate aliquot of each of the two primary solutions to obtain a spike solution concentration of 1000ug/L. All solutions are made volumetrically in Acetone at desired final volume. Generally 100ml of spiking solution standard is made but this amount can vary depending on use, need and preference. Generally 1 mL of LCS spiking solution is added into each laboratory control spike, matrix spike, and spike duplicate QC samples. Alternative solution concentration and spiking amount can be used.

8.7 Internal Standards (IS): PCB congener BZ 192 (2,2',3,4,4',5,6,6'-octachlorobiphenyl This solution is commercially obtained from Ultra at 100ug/mL. The IS solution is prepped by making a volumetric dilution of the primary standard in hexane to achieve a concentration of 2.5ug/ml. For method quantification by internal standard 20ul of internal standard is added into 1ml extracts, to produce a mid-calibration range response on column at 50ng/mL in final sample extract.

8.8 Breakdown Mix: This solution is commercially obtained from Ultra (PN ISM-450) containing Endrin at 1 μg/mL and 4,4'-DDT at 2ug/ml. The primary solution is volumetrically diluted to achieve a working standard concentration of 100ug/L for Endrin and 200 ug/L for 4,4'-DDT.

8.9 Toxaphene Calibration solution: Toxaphene solution is commercially obtained from Ultra (PP-270) at a concentration of 100ug/ml. The stock solution at the concentration of 10,000ug/L is prepared as an intermediate solution. The stock solution is diluted volumetrically to obtain a mixture at the concentration of 25ug/L. The 25ug/L solution is injected as a low standard and run with the calibration curve. If Toxaphene is detected an additional mid level Toxaphene standard is used for the one point quantitation of this compound. The stock solution is appropriately diluted volumetrically to obtain a concentration of 1000 ug/L for the Toxaphene and includes TMX/DCB at 100ug/L.

Toxaphene ICV solution is commercially obtained from NST (111-M) at a concentration of 2000ug/ml. The stock solution at the concentration of 20,000ug/L is prepared as an intermediate solution. The stock solution is appropriately diluted volumetrically to obtain a concentration of 1000 ug/L for the Toxaphene ICV working standard.

8.10 Chlordane Calibration solution: Chlordane solution is commercially obtained from Ultra (PP-151) at a concentration of 100ug/ml. The stock solution at the concentration of 10,000ug/L is prepared as an intermediate solution. The stock solution is diluted

volumetrically in hexane to obtain a mixture at the concentration of 25ug/L. The 25ug/L solution is injected as a low standard and run with the calibration curve. If Chlordane is detected and additional mid level Chlordane standard is utilized for the one point quantitation of this compound. The stock solution is appropriately diluted volumetrically to obtain a concentration of 500ug/L for Chlordane and includes TMX/DCB at 100ug/L

Chlordane ICV solution is commercially obtained from Restek (32021) at a concentration of 1000ug/ml. The stock solution at the concentration of 20,000ug/L is prepared as an intermediate solution. The solution is appropriately diluted volumetrically to obtain a concentration of 500 ug/L for the Toxaphene ICV working standard.

8.11 Independent Calibration Verification (ICV) standard: This is a secondary source standard separate from the calibration curve standards containing target pesticides compounds. The ICV can be made two ways as a combination of "Custom Standard – Pesticide Mix" (Quote#041310-631) and "Custom Standard – Additional Pesticide Mix" (Quote#041310-632) both commercially obtained from Ultra at a concentration of 100ug/ml. Alternatively, use Restek Pest Mix 8081AB#1 cat#32291 at concentration 200ug/ml and Hexachlorobenzene and Additional Pesticide Mix (Quote#041310-632) from Ultra at a concentration of 100ug/ml. Cis-Nonachlor obtained from Ultra at (PP-490) concentration 100ug/ml and Chlorpyriphos obtained from Restek (32212) at concentration 1000ug/ml are added to both mixtures to obtain all targets compounds. The ICV is prepared by diluting the appropriate aliquot of each of the two primary solutions to obtain a spike solution at the concentration of 50ug/L. This standard must be spiked with 20uL of the internal standard as referenced in section 8.7.

8.12 SRM: *1944 – New York/New Jersey Waterway Sediment, and SRM 2974a – Organics in Freeze Dried Mussel Tissue*, from National Institute of Standards & Technology (NIST). Please refer to the individual certifications for the assigned true values. These SRMs may be extracted and analyzed with sample batches as part of the overall QC evaluation, if requested by the client. Other certified SRMs may be used on a project specific basis.

8.13 Pesticide Calibration Standard: Commercially obtained primary solutions are used to volumetrically create stock solutions. All stock solutions are made by adding the appropriate amount of primary standard to a 25 mL volumetric flask to obtain the concentration of 5000 ug/L. The stock solutions are then used in various combinations and serial dilutions to make calibration curve standards (see Table 3).

Calibration standards are always prepared in hexane. The basic calibration curve is made from MCP Pest Stock, Additional Pest Stock, Chloropyrifos/Cis-nonachlor Stock and DBOB/BZ198 Surrogate Stock. MCP Pest Stock is derived from primary *Custom Pesticide Standard (PN # S-9553-SS)* commercially obtained from Accustandard, Additional Pest Stock is made from primary *Custom Pesticide Standard (PN#S-14378-R1)* commercially obtained from Accustandard, and DBOB/BZ198 Surrogate Stock is prepared from primary surrogate solutions commercially obtained from Ultra: *DBOB* – at a concentration of 5000 ug/mL *(catalog # PPS-172) and BZ#198* – at a concentration of 100 ug/mL *(catalog # RPC-075S).* Chloropyrifos/Cis-nonachlor Stock is prepared from primary solutions commercially obtained from Accustandard at a concentration of 100 ug/mL: Chloropyrifos (Dursban) *(catalog # P-094S) and Cis-nonachlor (catalog # P-297S).*

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

9.1.1 Method Blank

9.1.1.1 A method blank must be extracted (spiked with surrogates) and analyzed once per every 20 samples or per extraction batch, whichever is more frequent.

9.1.1.2 Because the analysis of a method blank is used to determine background contamination, an *acceptable* method blank should not contain any individual compound at the concentration of reporting limit, or above. For contaminated blanks all efforts must be made to identify and eliminate the source of contamination. The presence of analytes at concentrations at or above the reporting limit will warrant application of a “B” qualifier to that target compound(s) on all associated report forms, and perhaps re-extraction of all associated samples. The results are qualified with a “B” for any associated sample concentrations that are less then 10x the blank concentration for the analyte. Surrogate and internal standard recoveries must meet the QC limits for the method blank, see Sections 9.7.1 and 9.7.2. Re-extraction must be initiated immediately so that minimum time is wasted before re-extraction can occur - if at all possible, this re-extraction should take place within holding time. Re-extraction *corrective action* that would exceed the sample holding time criteria should be discussed with the client, Laboratory Director, QA Manager, and/or Section Supervisor prior to implementation. Exceptions may be made with approval of the Section Supervisor if the samples associated with an out of control method blank are non-detect for the affected compound(s) or if the concentration of the affected compound(s)in the sample is greater than 10x the blank level. In such cases, the sample results are accepted without corrective action for the high method blank result. The client must be notified, via the project narrative, of any method blank non-compliance associated with the sample results.

9.2 Laboratory Control Sample (LCS)

9.2.1 The laboratory control sample (LCS/LCSD) generally contains all Pesticides listed in Table 1, Section 16 except for Toxaphene & Chlordane. The number of Pesticide compounds in the LCS may vary with client specific requests. The Pesticide list reported to the client on the recovery form depends on the target list requested by the client and may also vary between different projects. The LCS/LCSD is extracted along with the samples and must be extracted and analyzed once per every 20 samples or per extraction batch, whichever is more frequent.

9.2.2 The acceptable recovery QC limits as well as RPD limits are found in Table 2 Section 16 for an aqueous, solid, and tissue LCS/LCSD. All recovery limits should

be monitored and documented in-house through control charts and updated as needed.

9.2.3 Corrective Action: Repeat analysis or check to see if an analytical or spiking error has occurred. If the LCS/LCSD recoveries are still out of control, re-extract and re-analyze the LCS/LCSD and all associated samples. Samples cannot be approved until an acceptable LCS is obtained. Exceptions may be made with approval of the Section Supervisor if the samples associated with the out of control LCS are also associated with a matrix spike and matrix spike duplicate that is in control which demonstrates an isolated problem pertaining to the LCS only. An explanation of this out of control LCS/LCSD recovery must be included in the project narrative to the client and the sample data reported with the acceptable MS/MSD results as batch QC. For specific work plans the project requirements may supersede existing acceptance criteria. E.g., if the LCS/LCSD do not meet quality control criteria for recovery of some of the spike and surrogate compounds, the results for the other samples and quality control samples within the batch must be evaluated to determine if this is an isolated problem for the LCS/LCSD and whether the data should be reported with the affected LCS/LCSD.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.4

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4

9.5 Matrix Spike

9.5.1 Matrix spike and matrix spike duplicate analyses are performed on a project specific basis. If a project calls for a MS/MSD one is performed per 20 samples and per matrix.

9.5.2 The acceptable recovery and RPD QC limits are found in Table 2 for an aqueous, solid, tissue, and product MS/MSDs. All recovery limits should be monitored and documented in-house through control charts and updated as needed.

9.5.3 Corrective Action: Repeat analysis or check to see if an analytical error has occurred. If the % recovery or %RPD still exceeds the control limits and the associated LCS is within control, include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the affected results as evidenced by the matrix spike and matrix spike duplicate exceedance.

9.6 Laboratory Duplicate

9.6.1 Laboratory matrix or sample duplicates are analyzed if requested by the client. The QC limit is 50% RPD for target compounds.

9.6.2 Corrective Action: If the %RPD exceeds the 50% control limit and the associated LCS/LCSD %RPD is within 50%, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results isolated to this sample, as evidenced by the matrix duplicate exceedance and the LCS/LCSD acceptance. If an MS/MSD was also extracted with the batch and the sample/duplicate as well as the MS/MSD exceed the control limits, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results as evidenced by the sample/duplicate and the MS/MSD exceedences.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogate

9.7.1.1 Surrogates are monitored for recovery for all matrices. Two surrogates are used, one that elutes at the beginning and one at the end of GC run. Every sample, blank, and quality control sample (LCS, LCSD, MS, MSD, Dup) must be spiked with the surrogates prior to extraction. The recovery limits are found in Table 2. The recovery limits should be monitored and documented in-house through control charts and updated as necessary.

9.7.1.2 Corrective Action: Check to see if an analytical, spiking, or dilution error occurred and re-calculate. If only one surrogate falls below the recovery limit and all QC samples have acceptable recovery indicating a matrix effect isolated to the sample, the exceedance is noted, with approval of the Section Supervisor, and the results are reported to the client with a notation in the case narrative. If all surrogates are recovered below the limit or there are outliers in any QC, re-extract the sample. If re-extraction occurred beyond the holding time and the re-extract surrogates are within the QC limits, report the re-extract results along with the original results. If the surrogates are recovered below the limit in the re-extract, this confirms suspected matrix interference on the surrogates, and only the original analysis needs to be reported unless a specific QAPP requires otherwise. If the chromatogram shows obvious matrix interference, no re-analysis or re-extraction is necessary. *This decision must be made with approval of the Section Supervisor and/or Data reviewer.* Surrogate outliers and sample re-extracts must be noted in the case narrative to the client.

9.7.2 Internal Standards

9.7.2.1 Internal standards are added to every field sample, QC sample, standard, and method blank. The Daily opening CCV IS areas are compared to the ICAL; for subsequent samples analyzed following CCVs – the IS areas are compared to the nearest opening CCV for each sample. The acceptance limits are -50% to +200% of the internal standard response (or area) of the daily continuing calibration verification standard.

9.7.2.2 Corrective Action: Check to see if an analytical, dilution or spiking error occurred. If the chromatogram shows obvious matrix interference, no re-analysis is necessary. *This decision must be made with approval of the Section Supervisor.* Note the exceedance in the case narrative to the client. If no obvious interference is present, re-analyze the extract. If internal standards are now within the acceptance limits, report only the re-analysis, as long as the re-analysis occurred within the 40-day analytical hold time. If the re-analysis occurred outside of the 40-day analytical hold time, both the original and re-analysis must be reported. If the internal standards again are outside the acceptance limits, after re-analysis, either within or outside of the 40-day hold time, report only the original analysis, and include a narrative to the client that the suspected matrix interference on the internal standards was confirmed by sample re-analysis.

9.7.3 SRMs

9.7.3.1 Standard reference materials (SRMs) are available from the National Institute of Standards and Technology (NIST) and are extracted and analyzed with samples on a project specific basis. These are not used as controls, but to evaluate potential matrix effects in associated samples for the target compounds being evaluated.

9.7.3.2 Acceptance criteria for SRM analysis will vary from project to project depending upon client data quality objectives (DQOs). Generally, 40% - 140% recovery based on the true certified values of the target compounds of interest, serve as advisory acceptance criteria.

9.7.3.3 Corrective Action: Repeat analysis and/or check to see if an analytical error has occurred. If the % recovery still exceeds the control limits and the associated LCS and/or MS/MSD are within control, include a project narrative with the results to client noting that the observed recovered of the SRM are isolated to this sample as evidenced by the LCS and/or MS/MSD acceptance.

9.7.4 Evaluate the percent degradation of 4,4'-DDT (to 4,4'-DDE and 4,4'-DDD) and Endrin (to Endrin Aldehyde and Endrin Ketone) in the Performance evaluation mixture (PEM) to monitor the integrity of the injection system (see section 11.10 for calculation). The breakdown mixture must be analyzed daily to verify that degradation in the system is not excessive. Degradation is not considered to be a problem if the percent degradation of 4,4'-DDT and Endrin are ≤15%. If either compound does exceed 15% breakdown, the analysis must be stopped, the injection port serviced and other maintenance may need to be performed.

9.8 Method Sequence

PEM3060501	(PEM)
C3060501	(CCV at 50ug/L)
Prep Blank ID	Method Blank
LCD ID	LCS
LCSD ID	LCSD
Sample ID	about 10 samples
PEM3060502	(PEM)
C3060502	(CCV at 50ug/L)
Sample ID	about 10 samples
PEM3060503	(PEM)
C3060503	(CCV at 50ug/L)
Sample ID	about 10 samples
C3060504	(Closing CCV at 50ug/L)

10. Procedure

10.1 Equipment Set-up

10.1.1 Prior to the analysis of any standards or samples, the instrument acquisition and process methods must be set up. This includes the GC run parameters and retention time windows. An initial calibration must be analyzed to establish linearity of the instrument. The target analytes are resolved on each column and detected using an electron capture detector (ECD). Calibration standards, quality control samples, and sample extracts are injected into the GC which is temperature and flow programmed to separate the compounds of interest. Identification of target analytes is accomplished by confirming a target hit on two dissimilar columns using Retention time and pattern recognition. Concentrations are calculated from the ECD response using internal standard techniques. Concentrations are determined using Average Response Factor from a multi-level calibration curve. The calibration curves are generated during initial

calibration through the analysis of standards which define the working range of the method. The integrity of the chromatography system must be maintained throughout a run sequence. The chromatographer should ensure that all aspects of the system, from injection through data generation is done in a consistent manner across each batch.

10.1.2 PEM Evaluation

10.1.2.1 Prior to initial calibration and at the start of each run a PEM must be analyzed and evaluated as mentioned above in the quality control section 9.7.4.

10.1.3 GC/ECD Instrumental Conditions

10.1.3.1 Generally a 1 to 3uL aliquot is injected into a uniliner and onto the capillary column of the gas chromatograph at the conditions listed below. Once injected the aliquot moves through the Siltek guard column and into a MXT-Y connector allowing one injection to split onto the two dissimilar columns connected to two separate ECD detectors

GC Parameter	Setting	GC Parameter	Setting
Injector Temp:	250°C	Ramp Rate 3:	5 °C / minute
Initial Oven Temp	125°C	Final Temperature 3:	260 °C
Flow:	33.6 mL/min	Final Hold Time 3:	0 minutes
Initial Hold Time:	0 minutes	Ramp Rate 4:	25 °C / minute
Ramp Rate 1:	25 °C / minute	Final Temperature 4:	280 °C
Final Temperature 1:	197 °C	Final Hold Time 4:	9 minutes
Final Hold Time 1:	2 minutes	Total runtime:	37.68 minutes
Ramp Rate 2:	1 °C / minute	Mode:	Splitless / Ramped Flow
Final Temperature 2:	210 °C	Purge:	25mL / minute – on at 1 minute
Final Hold Time 2:	0 minutes	Detector temp:	300 °C

The settings listed may vary if situation arises requiring an adjustment to the chromatography. *Injection temperature, hold time, flow rate, purge time, and injection volume can affect chromatographic resolution and detection limits.* Only a trained and experienced GC analyst has the authority to change any setting. All standards and samples must be acquired using the same set of parameters. If any parameters are changed, a new initial calibration must be analyzed and accepted before any samples can be analyzed.

10.2 Initial Calibration

10.2.1 Before analysis of sample extracts, establish a multi-point response factor calibration curve showing the linear range of the analysis for all target analytes. At least 5 levels of standards must be analyzed generally the concentrations run are 0.5, 1, 10, 20, 50, 100 and 200. See Section 8.11 for the preparation of the standard solutions for the initial calibration curve.

10.2.2 Construct an analytical sequence using the HP Enviroquant software. ICAL standards are identified in a sequence with an “I” designation followed by the instrument number, the

date, and the sequential number assignment. In the comment section beside each level of the curve add the assigned calibration standard ID. In sequence editor construct a sequence similar to the example listed below:

An example initial calibration sequence, created on 06/05, follows:

Primer1	
PEM3060501	(PEM)
Hexane	
I3060501STD.5	(0.5ng/mL standard)
I3060502STD1	(1ng/mL standard)
I3060504STD10	(10ng/mL standard)
I3060505STD20	(20ng/mL standard)
I3060506STD50	(50ng/mL standard)
I3060507STD100	(100ng/mL standard)
I3060509STD200	(200ng/mL standard)
Hexane	
CQ3060510STD50	ICV@50ng/mL Initial Calibration Verification
Hexane	
I3060511STD25	(Toxaphene @ 25 ng/mL)
I3060512STD25	(Chlordane @ 25 ng/mL)

10.2.3 When the sequence has finished running, the Enviroquant software will generate "Not Reviewed" quantitation reports. All reports must be "Quant Reviewed" before they can become part of the initial calibration acquisition method for sample analysis.

- Enter into the "Environmental Data Analysis"
- Go to "File" and under method, select the appropriate quant method and then select the first data file.
- Go into "Quant" and select "QEdit Quant Results" to process the data files. See Section 11.0 for manual integration details and processing guidelines.
- When processing, peak identification, and retention update is complete for the first standard, "Save" the changes and "Exit." Re-print the re-processed data file by "Generating Quant Report," and save the hard copy for each level of the initial calibration.
- Repeat these steps for all initial calibration standards analyzed within the sequence.
- When all levels have been processed, go into "InitCal," and select "Update Levels," and enter all levels for the initial calibration at the proper concentrations.
- After all responses are entered, "Save" the completed method and print the resulting response factor summary by selecting "Response Factors to Printer" as well as "Response Factors to Forms File".

10.2.3.1 Acceptance Criteria: 20% RSD for all target compounds. All calibration standards must be analyzed within 12 hours.

10.2.3.2 The following *corrective actions* are recommended for failing initial calibrations:

- If a curve has outliers and greater than five calibration solutions were utilized in the initial calibration, standards can be excluded from either extreme to generate a passing calibration. That is, the low-level standard or the high-level standard may be dropped to generate a five-level initial calibration. However the analyst needs to be aware that dropping the low standard can affect the RLs for the project being analyzed. An intermediate-level calibration standard must not be dropped to convert a failing initial calibration curve into a passing five-level initial calibration curve. Reduction in the number of calibration standards must also reduce the linear dynamic range used to quantify analytes in the samples. The resulting average response factor for each target analyte in the initial calibration curve will be used by the computer software to calculate actual sample concentrations. See Section 11.0 for additional calculation details.
- Perform instrument maintenance and repeat the initial calibration

The choice of corrective action must be made in consultation with the Section Supervisor, QA Manager, Project Manager, and/or the client.

10.2.3.3 Alternately, a linear regression model may be employed, provided that the correlation factor (r) is $\geq$0.99. Otherwise, construct a non-linear calibration of no more than a third order equation. Statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approach. A quadratic (second order) model requires six standards, and a third order polynomial requires seven standards. In setting model parameters, do not force the line through the origin. The COD (coefficient of determination) or r^2 must be greater than or equal to 0.99. The experienced analyst must select the regression order, which introduces the least calibration error into the quantitation.

10.2.3.4 Complete the initial calibration by filling out the *Initial Calibration Checklist*. The initial calibration, along with any corresponding continuing calibration data and sample data, is then forwarded for secondary review.

10.2.4 Initial Calibration Verification - ICV (separate source)

10.2.4.1 The analysis of separate source standard must follow the initial calibration curve. All target analytes in this verification standard are at the mid-point concentration. Note: This standard must be re-"Calculated and Generated" in the "QEdit" field, against the completed initial calibration curve method and processed, before it can be reviewed for acceptability.

After final processing, calculate the percent recovery of each target by using the following calculation:

% Recovery = (Found Amount / True Value) x 100

Acceptance Criteria: All recoveries must be +/- 20% of the true values.

10.3 Equipment Operation and Sample Processing

10.3.1 Evaluate the PEM as described in Sections 9.7.4

10.3.2 After successful analysis of the PEM, "Edit" the "Sample Table Log" to include the 50 ug/L CCV standard (C3060501, where "C" is for CCV, "3" is for instrument number 3,

followed by the date, and "01" for the first CCV analyzed this day), and acquire the CCV against the correct initial calibration method. "Save," then "Load and Run" the sequence.

10.3.3 When the sequence has finished running, the Enviroquant software will generate a "Not Reviewed" quantitation report. All reports must be "Quant Reviewed" against the acquisition method for sample analysis.

10.3.4 Samples are processed from "Not Reviewed" data files, to "Quant Reviewed" data files in a similar way the standards were previously processed. See Section 11.0 for details on sample processing. If a CCV fails the criteria outlined in Section 10.4.2, all samples since the last acceptable CCV must be re-analyzed.

10.3.5 If the on-column concentration of any compound exceeds the calibration range of 200 ug/L, the sample must be diluted, re-spiked with the appropriate amount of internal standard and re-analyzed. Assuming all samples are at a 1mL final volume, the following example dilutions would apply. Adjust the volumes accordingly for other sample final volume amounts and other desired dilutions.

1:2 dilution = 500uL of sample: 500uL Hexane and 10uL of IS
1:4 dilution = 250uL of sample: 750uL Hexane and 15uL of IS
1:5 dilution = 200uL of sample: 800uL Hexane and 16uL of IS
1:10 dilution = 100uL of sample: 900uL Hexane and 18uL of IS, etc

10.4 Continuing Calibration

10.4.1 A continuing calibration verification (CCV) standard, at the concentration of the mid-level of the initial calibration curve, must be analyzed at the beginning of every analytical sequence, and every 12 hours within the sequence, to confirm instrument stability, via response factor, for each calibrated pesticide.

10.4.2 Acceptance Criteria: Compare the CCV resulting concentration against the concentration of the compound in the injected solution for each calibrated pesticide, and calculate the % difference (%D). See Section 11.0 for the calculations. The %D for each calibrated pesticide must be below 20%D. Due to the nature of the dual column method, this criterion is applied to each set of column results. If multiple CCVs are analyzed within an analytical sequence, each CCV must be analyzed within 12 hours of the previous CCV and must meet the acceptance criteria. Note: closing CCV is not required as long as all samples are analyzed within 12h and the IS meets the acceptance criteria in section 9.7.2.1.

10.4.3 If the CCV does not meet the criteria for each calibrated analyte, the following *corrective actions* are recommended:

- Perform instrument maintenance and repeat the CCV. If the CCV fails again the initial calibration may need to be repeated
- If CCV is biased high and samples are non-detect the data may be usable
- If there is a CCV outlier for a target compound on one column, the analyst can make a judgment to force hits for that target in associated samples from the opposing acceptable column.
- If within 12 hours and no maintenance or adjustments have been made to the instrument the closing CCV can be re-injected once in case of mis-inject etc.

The choice of corrective action must be made in consultation with the Section Supervisor, QA Manager, Project Manager, and/or the client. The reasoning for choosing the second **option must be documented in the project narrative to the client.**

10.5 Preventive Maintenance

10.5.1 Preventive maintenance may or may not include the following: replacing system uniliner, o-ring, merlin septum, MXTY connector or clipping a length of guard column.

10.5.2 Additionally, preventive or routine maintenance for GC/ECD systems may involve baking out the ECD detector's, replacing guard, RTX-5 or CLPII columns.

11. Data Evaluation, Calculations and Reporting

11.1 Analyze extracts using the same conditions used for the analysis of the calibration standards. After sample analysis, “Not Reviewed” quantitation reports are generated by the software system. It is expected that situations will arise when the automated quantitation procedures of the chromatographic software provide inappropriate quantitations or integrations. Those situations will warrant the analyst perform manual integrations. Due to the especially low levels of the calibration curve, limitations of the software, and nature of the ECD, low level standards may require considerable manual integration. Because of this, a detailed report of both the low level standard and mid level standard will be submitted to QC for integration review and comparison.

11.2 Identification of the Pesticide compounds are based on gas chromatographic relative retention times (RRTs) and pattern recognition from the analysis of initial calibration standards. For these compounds, manual quantitations are performed, if necessary, by integrating the area of the peak. Qualitative identification for pesticides is made when a discrete peak in a sample is observed within the retention time window for a calibrated analyte on both columns. A pesticide hit can only be acceptable if confirmed by identification on the second column. If a Pesticide hit is not accurate on one of the two columns, i.e. outside of the retention window or absent, then the hit should not be quantitated. If manual integration of a candidate peak is necessary a straight-line integration to the baseline, taking into account background noise should be made. Area measurements should all be to baseline unless an unresolved complex mixture (UCM) is observed, in which case, the areas measured should skim the top of the UCM. Manual integrations may also be necessary when there are interferences or co-eluting peaks. Please refer to the manual integration SOP for further information.

Note: Manual integration is not to be used solely to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system.

11.3 Due to the nature of this method requiring confirmation on two dissimilar columns, two quantitations are acquired. Calculate the relative percent difference (RPD) between compound concentrations between both columns. If the calculated RPD is >40%, check the chromatograms to see if an overlapping peak is causing an erroneously high result. If no overlapping peaks are noted, examine baseline parameters established by the data system (or operator) during peak integration.

- If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher result. This approach is conservative relative to the protection of the environment. The disparity between the two columns should be flagged with a “P” on the report.
- If the high RPD is clearly the result of an interference on one of the columns, the lower result is reported with an “IP” qualifier on the report.

11.4 Evaluate all samples according to the following criteria:

- Surrogates and internal standards should meet the acceptance criteria in Table 2.
- Examine the chromatograms for evidence of saturated targets. Re-analyze the sample(s) at the appropriate dilution(s), see Section 10.3.5, as needed for reference.
- Establish daily retention time windows as the retention time of the component in the opening verification standard ± 0.05. It has been found that this window works well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results. The window listed above is used as guidance; however the experience of the analyst weighs heavily in the interpretation of the chromatograms.
- If one of the two results for a target appears to have a biased response due to interference the analyst can force the quantitation from the column providing the more accurate value. Any target hit forced off of the compliant column due to interference on the alternate column will be flagged with an "I" on the report only if the RPD between the two columns is greater than 40%.
- In the instance that a Pesticide co-elutes with another Pesticide or other target compound on one column, the compound will be "C" flagged and reported off the unbiased conformant column (i.e.: on the RTX-5 column Heptachlor Epoxide/oxychlordane co-elute). Studies of independent targets have been conducted for reference of co-eluting pesticides and congeners.

11.5 In instances where manual integrations have been performed, the analyst must identify/acknowledge such edits or manual procedures by initialing and dating the raw quantitation report. The initialed hard copy/printed report, will display a "m" qualifier next to any modified or manually integrated compound(s) and shall be included in the raw data for secondary review. These requirements apply to all QC samples, field samples and blanks. For all samples the signed hard copy will be a detailed report submitted for secondary review. As mentioned above there are guidelines for appropriate manual integrations and analyst initials indicate that they accept the "m" flagged changes as appropriate.

11.6 Qualitative identification of multicomponent pesticides requires pattern matching between the calibration standards and the response observed in the sample on both columns. Retention time windows should be used as a gauge, however, pattern recognition for the multicomponent analytes is most important for samples with Toxaphene or chlordane positively identified on both columns, inject an analytical standard at the mid-level or at a concentration estimated to be similar to the sample amount, and re-inject the sample. Quantitation is generally performed using 3 to 5 major representative peaks.

11.6.1 Multi-component analytes are quantitated on the basis of 3 to 5 major peaks using an internal standard approach to calculate individual response factors for each peak. Separate calibration standards are necessary for each multi-component target analyte (e.g., toxaphene and chlordane). Analysts should evaluate the specific toxaphene standard carefully. Some toxaphene components, particularly the more heavily chlorinated components, are subject to dechlorination reactions. As a result, standards from different vendors may exhibit marked differences which could lead to possible false negative results or to large differences in quantitative results. The relative peaks and number of peaks in the sample should be similar to that observed in the standard; however,

degradation, weathering and interferences may cause the sample pattern to differ from that observed from the standard.

11.7 To calculate the ***Relative Standard Deviation*** (RSD) of all Pesticide targets and surrogate compounds for the initial calibration, use the formula below. See Section 10.2.3 for initial calibration acceptance criteria. Additionally, use the initial multi-point calibration to determine ***Relative Response Factors*** (RRF_Is) at each concentration level, for each Pesticide. Average the RRF_Is from the initial multi-point calibration, to generate mean RRF_Is, for quantification of each Pesticide. Follow the same calculations for each surrogate compound. The RRF_Is are based on the internal standard compounds, and are calculated using the formula below. See Table I, for the listing of target compounds, RLs, and their associated internal standards for quantification.

$$\mathbf{RSD = SD / mean\ RRF_I \times 100}$$

where:

SD = Standard deviation between the five points, for that target analyte.

$$\mathbf{RRF_I = (A_c \times C_{IS}) / (A_{IS} \times C_c)}$$

where:

A_c = Area of the characteristic ion for the standard compound to be measured.
A_{IS} = Area of the characteristic ion for the representative internal standard compound.
C_{IS} = Concentration of the representative internal standard compound (ng/mL).
C_c = Concentration of the standard compound to be measured (ng/mL).

11.8 Based on the mean RRF_Is, calculate the ***Raw Concentration*** (C_a) for each target analyte and surrogate in the sample extracts using the following formula:

$$\mathbf{C_a = (A_a \times C_{IS}) / (A_{IS} \times RRF_I)}$$

where:

C_a = Calculated concentration of the target analyte, from quantitation report (ug/L).
A_a = Area of the target analyte.
A_{IS} = Area of the representative internal standard compound.
C_{IS} =Concentration of the internal standard compound added to each extract (ug/L).

11.9 Calculate the ***Sample Concentration*** (C) for each Pesticide by the following formula:

$$\mathbf{C = (C_a / V_s) \times FV \times DF}$$

where:

C = Concentration in the sample (ug/L liquid or ug/Kg solid).
V_s = Original volume or weight of sample extracted, corrected for % solids, if applicable. (To correct for % solids, multiply the sample weight by the % solid as expressed as a decimal. For example: 15.24g x 0.843 = 12.85, for a sample size of 15.24g at 84.3% solid).
DF = Dilution factor or fraction of the original extract to which internal standard is added.
FV = Sample Final Volume

If the response of any analyte in a sample exceeds the linear response range, as defined by the initial calibration standards in Section 10.2, dilute the extract so that the concentrations of all target compounds fall within the range of the calibration curve.

11.10 The calculation for percentage breakdown for DDT and Endrin are:

$$\%\text{Breakdown DDT} = \frac{(\text{Area DDD} + \text{Area DDE})}{(\text{Area DDD} + \text{Area DDT} + \text{Area DDE})} \times 100$$

$$\%\text{Breakdown Endrin} = \frac{(\text{Area Endrin Ketone} + \text{Area Endrin Aldehyde})}{(\text{Area Endrin} + \text{Area Endrin Ketone} + \text{Area Endrin Aldehyde})} \times 100$$

11.11 Calculate the ***Surrogate Recoveries*** (%R) relative to the internal standards by the following formula:

$$\%R = \frac{C_S \times DF}{C_{Strue}} \times 100$$

where:

C_{Strue} = Actual concentration of the surrogate in the extract (ug/L).
C_S = Calculated concentration of the surrogate, from quantitation report (ug/L).
DF = Dilution factor or fraction of the original extract

11.12 Compare results for each analyte in the ***Continuing Calibration Verification*** (CCV), to the true value by determining the percent difference.

$$\textbf{Percent Difference (\%D)} = ([C_{Ctrue} - C_C] / C_{true}) \times 100$$

where:

C_{Ctrue} = Actual concentration of the compound in the CCV (ug/L).
C_C = Calculated concentration of the compound in the CCV, from quantitation report

11.13 All results must be reported to three significant figures. All solids including soils, sediments, and sludges must be reported on a dry-weight basis. Tissue results may be reported in wet-weight, or "as received," depending upon client request.

11.14 The primary analyst does data entry, or upload of the data, into the LIMS system. The LIMS is "linked" to the instrument, so the analyst must choose the sample(s) to be reported from that instrument's analytical sequence. All associated preparation and instrumental QC samples and dilutions are also chosen. Once the data/samples have been selected and "associated" with the proper QC samples, the batched data set is sent to print.

11.15 The laboratory generates two types of data packages from the LIMS: "Commercial" for routine projects, and "Full Deliverable" or "CLP-like" for fully data validated projects. A Commercial package consists of sample results and the associated method blank and LCS results. A Full Deliverable package includes all sample results, all preparation and instrumental QC results and the associated supporting raw data. Check the "Report Type"

on the project folder to ensure all required deliverables are included. A secondary review is performed on all data.

11.16 Procedures for data and record management must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP, 08-01. All records shall be stored in such a manner as to be safe and accessible for at least 10 years.

11.17 Notebooks: Laboratory notebooks are designed to accommodate the specific analysis. Instrument printouts are used to document run sequences, and each sequence printouts are filed in a three-ring notebook. If a sample requires re-analysis or re-extraction for any reason, a notation is made next to the sample entry on the sequence log. Requests for re-extraction are further documented in the "Request for Re-extraction" logbook. Once a sequence binder if full, the sequence run log is permanently bound, given page numbers and filed accordingly. Such files shall be archived so as to remain available for at least 10 years. All laboratory notebooks must follow the specifications in the *Laboratory Notebook Usage* SOP, G-009, and all record keeping and document control practices.

11.18 Electronic records: All data files from computers, attached to instruments, shall be backed up onto the proper directory on the server. The backups shall be stored so as to be accessible for 10 years. Movement of the data files to the server is the responsibility of the primary analyst. Server backup and storage is the responsibility of the IT department.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 Section 9, Quality Control defines the corrective actions that must be taken in instances where QC outliers exist. If the corrective actions have been followed and the data is still unacceptable.

12.2 Table 2 outlines sample batch QC acceptance criteria. If non-compliant Pesticide results are to be reported, the Section Supervisor and/or the Laboratory Director, and the QA Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may choose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Section Supervisor performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP 1559 Sample Receipt and Login

SOP 1729 Document Control

SOP 1732 MDL/LOD/LOQ Generation

SOP 1739 IDC/DOC Generation

SOP 1797 Hazardous Waste and Disposal

SOP 2165 Extraction of Water Samples by Separatory Funnel

SOP 2166Tissue Preparation and Homogenization

SOP 2167 GPC Cleanup by HPLC

SOP 2168 Sulfur Cleanup

SOP 2261 Shaker Table Extraction

SOP 2267 Silica cleanup

SOP 2172 Microscale Solvent Extraction

16. Attachments

Table 1: Reporting Limits

Table 2: QC Acceptance Criteria

Table 3: 5-8 Level Curve Preparation for Individual Components

Table 1: Reporting Limits (RLs) for Pesticides by GC/ECD

Parameter	Solid Samples (ug/Kg)	Aqueous Samples (ug/L)
alpha-BHC	0.0333	0.0005
beta-BHC	0.0333	0.0005
gamma-BHC (Lindane)	0.0333	0.0005
delta-BHC	0.0333	0.0005
Heptachlor	0.0333	0.0005
Aldrin	0.0333	0.001
Hexachlorobenzene	0.0666	0.001
alpha-Chlordane	0.0333	0.0005
gamma-Chlordane	0.0333	0.0005
Heptachlor epoxide	0.0333	0.0005
Oxychlordane	0.0666	0.001
Cis-Nonachlor	0.0333	0.0005
Trans-Nonachlor	0.0333	0.0005
Dieldrin	0.0333	0.0005
Endrin	0.0333	0.0005
Endrin aldehyde	0.1	0.0005
Endrin ketone	0.0333	0.0005
4,4'-DDD	0.0333	0.0005
4,4'-DDE	0.0333	0.0005
4,4'-DDT	0.0333	0.0005
2,4'-DDD	0.0333	0.0005
2,4'-DDE	0.0333	0.0005
2,4'-DDT	0.0333	0.0005
Endosulfan I	0.0333	0.0005
Endosulfan II	0.0333	0.0005
Endosulfan sulfate	0.0333	0.0005
Methoxychlor	0.333	0.005
Mirex	0.0333	0.0005
Toxaphene	1.67	0.025
Chlordane	1.67	0.025

Note: The reporting limits used for "Solid" refer to the matrices of soil, sediment, tissue, and any other possible non-aqueous matrix. The table above represents an example of the range of reporting limits generally used in this analysis. The RLs for this analysis can vary based on sample matrix, amount extracted, final volume and cleanup schemes. Adjustments to these limits can be made to satisfy specific workplan or data quality objective requirements and specialty matrices.

The quantitation Internal Standard for all the targets is BZ 192.

Table 2: QC Acceptance Criteria

QC Parameter	Acceptance Criteria
Initial Calibration Curve	20% RSD for all target analytes, r is ≥0.99, the COD (coefficient of determination) or r^2 ≥0.99
Independent Check Verification	+/- 20% recovery of the true values
Continuing Calibration Verification	Analyzed every 12 hours, 20% D
Method Blank	No analyte at or above the reporting limit, "B" qualify analyte if detected
Laboratory Control Sample	40-140% (Sporadic Marginal Failure Criteria allowed)
Matrix Spike / Matrix Spike Duplicate	Same as for LCS; 50% RPD between the duplicates.
Sample / Sample Duplicate	50% RPD between the duplicates.
Surrogates	30-150%
Internal Standards	50% - 200% of the daily CCV area for the Internal Standards
SRM	40-140%
%RPD between dual column	+/- 40%, "P" qualify analyste

Table 3: 5-8 Level Curve Preparation for Individual Components

Calibration Level	Volume of Pesticide/Surrogate Stocks added*	Aliquot of High Calibration level** added	Volume of Hexane added
Level 1 – 0.5 ug/L	NA	0.025 ml	10 mL final volume
Level 2 – 1.0 ug/L	NA	0.05 ml	10 mL final volume
Level 3 – 5.0 ug/L	NA	0.25 ml	10 mL final volume
Level 4 – 10 ug/L	NA	0.5 ml	10 mL final volume
Level 5 – 20 ug/L	NA	1.0 ml	10 mL final volume
Level 6 – 50 ug/L	0.25 mL	NA	25 mL final volume
Level 7 – 100ug/L	0.2 mL	NA	10 mL final volume
Level 8 – 150ug/L	0.3 mL	NA	10 mL final volume
Level 9 – 200ug/L	0.4 mL	NA	10 mL final volume

Up to 10 levels may be analyzed. Standards chosen should bracket the linear range of the instrument. A minimum of a 5-level curve must be analyzed, but 6-levels, or more, may be analyzed and evaluated depending upon client specific project detection limits. This is an example of possible curve levels, the curve used may vary based on project and instrument performance.
* Each calibration level is a combination of four stock solutions prepared at the concentration of 5000ug/L added at the indicated amount.
**High calibration level is 200ug/L.

Note: 20uL of the 2.5ug/mL chosen Internal Standard mix is added to each calibration level.

Determination of PCB Homologs and 209 Individual Congeners by GC/MS - SIM

References: **Method 680,** "*Determination of Pesticides and PCBs in Water and Soil/Sediment by Gas Chromatography / Mass Spectrometry*", USEPA, Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, Ohio; November 1985

Method 8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), Revision 4, February 2007, Test Methods for Evaluating Solid Waste, SW-846,

Method 8082A Polychlorinated Biphenyls (PCBs) by Gas Chromatography, Rev. 1, February 2007, Test Methods for Evaluating Solid Waste, SW-846

Determination Of Polychlorinated Biphenyls In Ambient Air Using Low/High Volume Polyurethane Foam (PUF) Sampling Followed By Gas Chromatographic/Multi-Detector Detection (GC/MD), Center for Environmental Research Information, Office of Research and Development, U.S. Environmental Protection Agency, Cincinnati, OH 45268, January1999. EPA/625/R-96/010b.

NOAA Technical Memorandum NMFS-NWFSC-59, Extraction, Cleanup, and Gas Chromatography/Mass Spectrometry Analysis of Sediment and Tissue for Organic Contaminants, March 2004, U.S. of Department of Commerce, National Oceanic and Atmospheric Administration

1. Scope and Application

Matrices: This method is applicable to the quantification of Polychlorinated Biphenyls (PCBs) as Homologs and/ individual Congeners in water, soil, sediment, tissue (either animal or vegetable) and in ambient air using PUF sampling followed by Gas Chromatography/Mass Spectrometry with Selected Ion Monitoring (GC/MS-SIM).

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the analysis and quantification of sample extracts for PCBs as single Congeners and Homologs (isomer groups by the level of chlorination) by Gas Chromatography/Mass Spectrometry with Selected Ion Monitoring (GC/MS-SIM). Additionally, this method can provide a "total" PCB result for a given sample extract. Target analytes include selected PCB congeners from BZ1 to BZ209 and the Homolog groups listed below. Individual congeners and homolog groups are determined and measured in the concentration range of 0.5 to 500 parts per trillion (ng/L) for water samples, 0.033 to 200 parts per billion (ug/Kg) for soil/sediment and tissue samples, and 5-5000 ng/puf for PUF cartridges. These ranges are determined based on the lowest and highest levels of the calibration curve, extracted amount and final volume therefore detection limits will vary with the individual sample matrix, sample preparation procedures, instrument calibration range, and volume of sample analyzed. Detection limits for Homolog groups are equal to the lowest detection limit of the individual congeners detected within that group. In general, analytes detected over these concentration ranges will be diluted and re-analyzed for accurate quantitation.

The following extraction and cleanup methods may apply, prior to sample analysis:

- *Extraction of Water Samples by Separatory Funnel-Method 3510C* (SOP 2165),
- *Tissue Preparation and Homogenization* (SOP 2166),
- *Shaker Table Extraction* (SOP 2261),
- *Sulfur Cleanup with Copper-Method 3660B* (SOP 2168),
- *Gel Permeation Chromatography-GPC (SOP 2167)*

- *Sulfuric Acid Cleanup-Method 3665A* (SOP 2169),
- *Silica Gel Cleanup* (SOP 2170),
- *Microscale Solvent Extraction – Method 3570* (SOP 2172)
- *Soxhlet Extraction – Method 3540C (SOP 2173),*
- *Soxhlet Extraction of PUF Cartridges (SOP 2174)*

The existence of 209 possible PCB congeners makes it impractical to list each potential method analyte and Chemical Abstract Service (CAS) number. Because in some cases, depending upon client request, PCBs are identified and measured as isomer groups, the non-specific CAS number for each level of chlorination is used to describe the method analytes. See below for this listing.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/MS and in the interpretation of GC/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

PCB Homolog Group	Formula	CAS #
Monochlorobiphenyl	$C_{12}H_9Cl$	27323-18-8
Dichlorobiphenyl	$C_{12}H_8Cl_2$	25512-42-9
Trichlorobiphenyl	$C_{12}H_7Cl_3$	25323-68-6
Tetrachlorobiphenyl	$C_{12}H_6Cl_4$	26914-33-0
Pentachlorobiphenyl	$C_{12}H_5Cl_5$	25429-29-2
Hexachlorobiphenyl	$C_{12}H_4Cl_6$	26601-64-9
Heptachlorobiphenyl	$C_{12}H_3Cl_7$	28655-71-2
Octachlorobiphenyl	$C_{12}H_2Cl_8$	31472-83-0
Nonachlorobiphenyl	$C_{12}H_1Cl_9$	53742-07-7
Decachlorobiphenyl	$C_{12}Cl_{10}$	2051-24-3

2. Summary of Method

An aliquot of a well mixed, homogeneous aqueous, solid, or tissue sample is accurately measured or weighed for sample preparation. Generally, 1L of water sample, 1-10g of tissue sample, 5-30g of sediment/soil sample for *Microscale Solvent Extraction - 3570 (*SOP 2172*) and 15-30g* of sediment/soil sample *for Soxhlet Extraction (SOP 2173)*. The PUF cartridge is extracted via Soxhlet, with the appropriate solvent. Water, soil/sediment, and tissue samples as well as PUF cartridges are spiked with surrogate compounds and extracted using methylene chloride,methylene chloride/acetone mixture, or hexane/ether mixture. The extract is dried and exchanged to hexane during sample concentration to a 1-10mL final volume. If necessary, the sample may be copper cleaned to remove sulfur, and/or GPC, silica or acid cleaned to lessen sample matrix interferences, prior to sample analysis.

After cleanup, the extracts are spiked with internal standards, and analyzed by GC/MS-SIM. Analytes are introduced into the GC/MS by injecting a known volume of the calibration standards, quality control samples, and sample extracts into the GC equipped with a narrow-bore capillary column. The GC column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer operating in the selective ion mode (SIM). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of the calibration standards. Concentrations are determined using mean relative response factors from a multi-level calibration curve. Response factors for target analytes and surrogate compounds are determined relative to the internal standards.

2.1 Method Modifications from Reference

This method exhibits some modification from the reference methods.

SIM data acquisition parameters, GC separation/operating conditions and MS sensitivity/calibration ratios for ions, and column type differ from the one described in Method 680 due to changes in technology since 1985.

Different compounds are utilized as Internal Standards and Surrogates then those specified in Method 680.

Different Calibration Congeners are used for Homologs group than the ones specified in Method 680.

Corrections are not made to any data for Homolog groups Cl2 – Cl8 for interferences resulting from M+35 or M+70 ions.

Acceptance criteria for ICAL, ICV, CCV, Method Blank, Surrogates and Internal Standards have been adopted from the guidance in Method 8270D.

The sample preservation procedure has been adopted from Method 8270D.

Different DFTPP criteria then specified in method 8270D. Maximum Sensitivity Criteria are used.

Method Modifications from NOAA Technical Memorandum:

- Different extraction method then specified, MSE versus ASE
- Different requirements for batch quality control samples: method blank and LCS/LCSD versus method blank and SRM
- Different cleanup techniques: silica columns only versus silica/alumina columns and size-exclusion high-performance liquid chromatography
- Different GC settings: PTV/splitless injection versus COC (cool on-column) injection; oven temperature program differs, no guard column
- Different MS settings: electron ionization versus chemical ionization
- Different Calibration model: Average Response Factor obtain from Multi Level Calibration versus Point-to-Point Calibration
- Different Quality Controls and Measurements

3. Reporting Limits

Analytes are determined and measured in the concentration range of 0.5 to 500 parts per trillion (ng/L) for water samples, 0.033 to 200 parts per billion (ug/Kg) for soil/sediment and tissue samples, and 5-5000 ng/puf for PUF cartridges. The detection limit for Homolog groups is equal to the lowest detection limit of the individual congener detected within that group. Detection limits will vary with the individual sample matrix, sample preparation procedures, instrument calibration range, and volume of sample analyzed.

4. Interferences

4.1 Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause interferences that lead to discrete artifacts and/or elevated baselines in the ion current profiles. Demonstrate that all of these materials are free from interferences under the conditions of the preparation and analysis by extracting and analyzing a laboratory method blank with each batch of up to 20 samples.

4.2 Contaminants co-extracted from the sample may cause matrix interferences. The extent of matrix interferences will vary considerably from sample to sample, depending upon the nature of the environment being investigated. An interference, which is unique to SIM techniques, can arise from the presence of co-eluting compounds, which contain the same quantification mass ion, or the same number of chlorine atoms. This event results in a positive interference to the reported value for the compound of interest. This interference is controlled to some degree by acquiring data for a confirmation ion. If the ion ratios between the quantification ion and the confirmation ion are not within the specified limits, then interferences may be present. Quantification and confirmation ion criteria can be found in Table II.

4.3 With the isomer, or Homolog group quantification approach, co-eluting PCBs that contain the same number of chlorines, are identified and measured together. Therefore, co-eluting PCBs are only a problem if they contain a *different* number of chlorine atoms.

4.4 An interference can arise from the presence of co-eluting congeners containing one or two additional chlorines, which will contribute to the quant ion due to the fragmentation that occurs during the analysis. This event results in a positive interference to the reported value for the compound of interest. PCBs listed below are co-eluting with other congeners containing two additional chlorines:
- when analyzed on BNA2/5 for 209 Congeners – BZ#37, BZ#60, BZ#78, BZ#81, BZ#107/123, BZ#105, BZ#127, BZ#126, BZ#167,
The interference from another PCB containing one additional chlorine is small and can be neglected except when measuring the area of a small amount of the congener co-eluting with a large amount of another congener containing one more chlorine.

4.5 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by

whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

***Aqueous* samples:** Collect in 1L or 2L amber glass bottles. The minimum amount of sample needed to reach the reporting limits in Section 3.0 for this method for aqueous samples is 1L. Additional sample is needed (approximately 3X the minimum amount) if MS/MSD analyses are to be performed.

***Soil/sediment* samples:** Collect in glass soil jars. The minimum amount of sample needed to reach the reporting limits in Section 3.0 for this method for solid and tissue matrices is 5g or 30g. Additional sample is needed (approximately 3X the minimum amount) if MS/MSD analyses are to be performed.

***Air Samples*:** Collected with appropriate air sampling techniques described in the Reference Method for collecting PUF cartridge air samples.

6.2 Sample Preservation

***Aqueous* samples:** Store without preservative at 4°C.

***Soil/sediment* samples:** Stored at 4°C, or if desired, frozen.

Air Samples: Stored at 4°C without preservation.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

The hold time for this method is 7 days for the extraction of aqueous samples and 14 days for the extraction of solid and tissue samples. If sediment or tissue samples are frozen, this suspends the holding time until removal from the freezer. Air PUF cartridges must be extracted within 7 days of collection.

All extracts must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph: The instrumentation includes a temperature-programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for splitless injection onto a capillary column. The injection port includes a silanized glass liner containing a plug of silanized glass wool to reduce high-molecular-weight mass discrimination. The model is HP6890 or equivalent. The injection port will require maintenance on an as needed basis if degradation or contamination is apparent.

7.2 Large volume injector, PTV - Gerstel, or equivalent: Temperature and flow programmable and capable of injecting 1 to 50 uL of standards and sample extracts onto the GC column in a split or splitless mode.

7.3 Column: For 209 Congener and Homolog Analysis - Restek 60-m x 0.18 mm ID, 0.18 um film thickness, fused-silica capillary column with Crossbond phase, or equivalent.

7.4 Mass Spectrometer: The mass spectrometer must operate at 70ev (nominal) electron energy in the electron impact ionization mode and be tuned to optimize the sensitivity of the instrument to the maximum in the mass range being monitored (45 - 525 amu). The GC capillary column is fed directly into the ion source of the mass spectrometer. The model is HP5973, or equivalent. The source will require cleaning and/or filament replacement on an as needed basis. Please refer to the instrument hardware manual, located in the laboratory, for detailed procedures.

7.5 Auto sampler: Adapted onto the Gas Chromatograph. The model is HP 6890 series autosampler with a GC autosampler controller, or equivalent.

7.6 Computer: With Windows XP operating software utilizing HP Enviroquant G1701DA Version D.01.02 software; Audit Trail: audit.txt function is used for audit trail purposes.

7.7 Helium: Ultra high purity grade (99.9999% pure) or hydrogen of equivalent purity.

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specification of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. See SOP Reagent, Solvent, and Standard Control (SOP 1816) for additional details regarding solvent purity. All solvent expirations determined as indicated by manufacturer guidelines

Analytical Standards are stored according to manufacturer's recommended procedure. Stock standards, and calibration curve standards are stored in either 10mL or 40mL glass vials and kept in a standards freezer at -10^0 — -20^0C in the GC Instrumentation Lab. Primary standards are discarded as indicated by the vendor expiration. Stock standards are given one year expiration from the preparation date or the expiration of the primary vendor solution, whichever occurs first. Working standards are given six month expiration from the preparation date or the expiration of the primary solution whichever occurs first. If breakdown of a solution is observed the solution will be discarded. All analytical standards are prepared in Hexane. All extraction standards (surrogates, laboratory control spikes and matrix spikes) are prepared in acetone or hexane for Soxhlet extraction - *Method 3540C*. All spiking solutions must be assayed for use by analysis before release to the preparation lab If there is a significant amount of spiking solution remaining after the six-month period, the lab will re-QC the solution. If the QC of the solution still passes the lab will extend the expiration date by another month.

8.1 Methylene Chloride: ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (SOP 1816) for additional details regarding solvent purity.

8.2 Acetone: ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (SOP 1816) for additional details regarding solvent purity.

8.3 Hexane: ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (SOP 1816) for additional details regarding solvent purity.

8.4 Methanol: Purge and Trap grade, see SOP *Reagent, Solvent, and Standard Control* (SOP 1816) for additional details regarding solvent purity.

8.5 Ether, see SOP *Reagent, Solvent, and Standard Control* (SOP 1816) for additional details regarding solvent purity.

8.6 DFTPP (decafluorotriphenylphosphene) tuning solution: Prepare by diluting 0.025mLs of a 2000 ug/mL standard into 10mL of Methylene Chloride for a 5 ug/mL tuning standard. Working solution is further diluted 1:10 prior to analysis.

8.7 Individual Analytes (BZ-1 to BZ-209): Obtained from AccuStandard or equivalent at a concentration of 100 ug/mL.

8.8 209 Congeners Custom Calibration Set: Obtained from Accustandard as 9 separate mixes at the concentration of 10 ug/mL each. The solution consists of 209 Congeners. Prepare the Stock Solution by diluting the calibration mixes to a stock concentration of 1000 ng/mL. See Section 8.16 for calibration preparation information.

8.9 Custom 136 PCB Congeners Set/ Homolog Custom Mix, Retention Time Window / ICV Standard: Obtained from Accustandard as 7 separate mixes at the concentration of 4 ug/mL each. The solution consists of at least one representative PCB congener used for quantitation from each Homolog group and the first and last eluting congener of each Homolog group used to identify the start and stop time of each SIM window. Prepare the Stock Solution by diluting the calibration mixes and the Carbon-labeled Surrogate Stock solution to a 136 Cong/Surr Stock concentration of 400 ng/mL. See Section 8.16 for standard preparation information.

8.10 Surrogates: 4,4'-Dibromooctafluorobiphenyl (DBOB) and BZ 198, obtained from Ultra Scientific or equivalent.

8.10.1 DBOB/BZ198 stock solution: Add 25 uL of the 5000 ug/mL DBOB primary solution and 1250 uL of the 100 ug/mL BZ 198 primary solution to 25 mL volumetric flask and dilute with hexane for 5000 ug/L stock solution.

8.10.2 Pest/Cong Surrogate spiking solution: add 4 mL of DBOB/BZ198 stock solution to 200 mL volumetric flask and dilute with acetone, for a 100 ug/L final concentration. The solution must be assayed for use by analysis before release to the preparation lab. All compounds must be within 20% of their true value. 1mL is spiked into each QC and field sample. The concentrations may be adjusted to meet project specific needs.

8.11 Carbon-labeled Surrogates: BZ 19 and BZ 202, obtained from Cambridge Isotope or equivalent, at a concentration of 40 ug/mL and BZ 101 and BZ 153, obtained from Wellington Labs or equivalent, at a concentration of 50 ug/mL.

8.11.1 Carbon-labeled Surrogates Stock Solution: Prepare a separate solution by taking 1.25mL of each of the 40 ug/mL surrogate solutions and 1.00mL or each

of the 50 ug/mL surrogate solutions, and add to 10mL of hexane for a stock concentration of 5 ug/mL or 5000 ng/mL.

8.11.2 Homolog Surrogate spiking solution: Add 250 uL of each surrogate (BZ 19 and BZ 202) and 200 uL of each surrogate (BZ 101 and BZ 153) to 100 mL volumetric flask and dilute with acetone, for a 100 ug/L final concentration. All compounds must be within 20% of their true value. 1mL is spiked into each QC and field sample. The concentrations and the spiking amount may be adjusted to meet project specific needs.

8.11.3 High Homolog Surrogate spiking solution: Add 1.25 mL of each surrogate (BZ 19 and BZ 202) and 1.00 mL of each surrogate (BZ 101 and BZ 153) to 50 mL volumetric flask and dilute with hexane, for a 1000 ug/L final concentration. The solution must be assayed for use by analysis before release to the preparation lab. All compounds must be within 20% of their true value. 1mL is spiked into each QC and field sample. The concentrations and the spiking amount may be adjusted to meet project specific needs.

NOTE: PCB High Homolog Surrogate spiking solution is prepared in hexane and used for Soxhlet extraction only.

8.12 Carbon-labeled Internal Standards (IS): BZ 15 and BZ 180, obtained from Cambridge Isotope or equivalent, at a concentration of 40 ug/mL. Add 2.5mL of each internal standard to 10mL volumetric flask and dilute with hexane, for a final concentration of 10 ug/mL. 20uL is spiked into each standard, QC sample, and field sample. The resulting on-column concentration is 200 ug/L in a 1mL sample aliquot. The concentrations may be adjusted to meet project specific needs.

8.13 Laboratory Control Sample, Matrix Spike, and Matrix Spike Duplicate (LCS/MS/MSD) Preparation:

8.13.1 Homolog LCS spiking solution: Prepared using Accustandard C-CSQ-SET. Nine ampules containing all 209 congeners at 10ug/mL. Add 1.0 mL of each mix to 100mL volumetric flask and dilute with *acetone,* for a 100 ug/L final concentration. The solution must be assayed for use by analysis before release to the preparation lab. Recoveries of 90% of the analytes must be +/- 20% of the true values.

8.13.2 High Homolog LCS spiking solution: Prepared using Accustandard C-CSQ-SET. Nine ampules containing all 209 congeners at 10ug/mL. Add 1.0 mL of each mix to 25 mL volumetric flask and dilute with hexane, for a 400 ug/L final concentration. The solution must be assayed for use by analysis before release to the preparation lab. Recoveries of 90% of the analytes must be +/- 20% of the true values.

NOTE: HIGH Homolog LCS/MS/MSD spiking solution is prepared in hexane and used for Soxhlet extraction only

8.14 Independent Calibration Verification (ICV) standard:

8.14.1 209 Congener ICV Standard: For the 209 Congener Calibration Curve, a level 5 from the 136 Congeners standard serves as ICV (Accustandard or equivalent)

8.15 SRM 1944/1941b – New York/New Jersey Waterway Sediment, and SRM 1974b – Organics in Mussel Tissue: From National Institute of Standards & Technology (NIST). Please refer to the individual certifications for the assigned true values. These SRMs may be extracted and analyzed with sample batches as part of the overall QC evaluation, if requested by the client. Other certified SRMs may be used on a project specific basis.

8.16 Calibration Preparation Information

Homolog Calibration Congener	**Isomer Group**	**Stock Concentration ng/mL 209Congeners**
BZ 1 (1st Mono)	Cl1	400 1000 ng/mL
BZ 8, BZ 5/8	Cl2	400 1000 ng/mL
BZ 29	Cl3	400 1000 ng/mL
BZ 50	Cl4	400 1000 ng/mL
BZ 87, BZ 87/111	Cl5	400 1000 ng/mL
BZ 154	Cl6	400 1000 ng/mL
BZ 188 (1st Hepta)	Cl7	400 1000 ng/mL
BZ 200, BZ 200/204	Cl8	400 1000 ng/mL
BZ 206 (last Nona)	Cl9	400 1000 ng/mL
BZ 209 (Deca)	Cl10	400 1000 ng/mL
Add'l Ret. Window PCB Congeners	**Isomer Group**	**Stock Conc. ng/mL 209Congeners**
BZ 3 (last Mono)	Cl1	400 1000 ng/mL
BZ 10 (1st Di)	Cl2	400 1000 ng/mL
BZ 15 (last Di)	Cl2	400 1000 ng/mL
BZ 19 (1st Tri)	Cl3	400 1000 ng/mL
BZ 37 (last Tri)	Cl3	400 1000 ng/mL
BZ 54 (1st Tetra)	Cl4	400 1000 ng/mL
BZ 77 (last Tetra)	Cl4	400 1000 ng/mL
BZ 104 (1st Penta)	Cl5	400 1000 ng/mL
BZ 126 (last Penta)	Cl5	400 1000 ng/mL
BZ 155 (1st Hexa)	Cl6	400 1000 ng/mL
BZ 169 (last Hexa)	Cl6	400 1000 ng/mL
BZ 189 (last Hepta)	Cl7	400 1000 ng/mL
BZ 202 (1st Octa)	Cl8	400 1000 ng/mL
BZ 205 (last Octa)	Cl8	400 1000 ng/mL
BZ 208 (1st Nona)	Cl9	400 1000 ng/mL
Surrogates		
DBOB	N/A	5000 ng/mL
BZ 198	Cl8	5000 ng/mL
Carbon-labeled Surrogates		
BZ 19	Cl3	5000 ng/mL
BZ 202	Cl8	5000 ng/mL

Note: Any of the above congeners may be reported as individual congeners, as well as within a Homolog group, as this method is not limited to this congener list. Additional congeners may be analyzed via this method by utilizing the MDL and PQL from a congener of the same Homolog class that has been previously established. Parentheses indicate the first and last eluting PCB congener in each chlorination level, used to establish the selective ion monitoring (SIM) retention time windows.

Suggested ICV Preparation for 209 Congeners

Calibration Level	136 Cong. /Surr Stock 400ng/mL	Final Volume
ICV – 50 ug/L	1.25mL	10 mL

Suggested Curve Preparation for Individual Components (Minimum 5 Levels) for 209 Congeners

Calibration Level	Surr. Stock 5000ng/mL	209 Cong. Stock 1000ng/mL	Calibration L6 (200ug/L)	Final Volume
L1 – 0.5 ug/L	-	-	0.025mL	10 mL
L2 – 1.0 ug/L	-	-	0.05mL	10 mL
L3 – 10 ug/L	-	-	0.5mL	10 mL
L4 – 20 ug/L	-	-	1mL	10 mL
L5 – 50 ug/L	0.1mL	0.5mL	-	10 mL
L6 – 200ug/L	0.4mL	2mL	-	10 mL
L7 – 500 ug/L	1ml	5ml	-	10 mL

Curve Preparation for Individual Components

Up to 10 levels may be analyzed. Standards chosen should bracket the linear range of the instrument. A minimum of a 5-level curve must be analyzed, but 6-levels, or more, may be analyzed and evaluated depending upon client specific project detection limits. This is an example of possible curve levels, the curve used may vary based on project and instrument performance.
* Each calibration level is a combination of three stock solutions prepared at the concentration of 5000ug/L added at the indicated amount.
**High calibration level is 200ug/L.

Note: 20 uL of the 10 ug/mL chosen Internal Standard mix is added to each calibration level for a concentration of 200 ng/mL. *A minimum of a 5-level curve must be analyzed, but 6-levels, or more, may be analyzed and evaluated depending upon client specific project detection limits.*

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Quality Control (QC) samples are necessary to monitor both the sample extraction and instrument analysis procedures. The Quality Control samples described below are considered the method

defaults, and are the minimum requirements, except where noted. Client and Project specific Data Quality Objectives (DQOs) supersede the requirements in this section where applicable. Client or Project specified DQOs shall be included, or referenced, in the final report to the client.

9.1 Blank(s)

A method blank must be extracted (spiked with surrogates and internal standards) and analyzed once per every 20 samples or per extraction batch, whichever is more frequent.

An *acceptable* method blank should not contain any individual compound at the concentration of reporting limit, or above. All efforts must be made to identify and eliminate the source of contamination. The presence of analytes at concentrations at or above the reporting limit will warrant application of a “B” qualifier to that target compound(s) on all associated report forms, and perhaps re-extraction of all associated samples. The results are qualified with a “B” for any associated sample concentrations that are less then 10x the blank concentration for the analyte. Surrogate and internal standard recoveries must meet the QC limits for the method blank, see Sections 9.7.1 and 9.7.2. Re-extraction *corrective action* that would exceed the sample holding time criteria should be discussed with the client, Laboratory Director, QA Manager, and/or Section Supervisor prior to implementation. Exceptions may be made with approval of the Section Supervisor if the samples associated with an out of control method blank are non-detect for the affected compound(s) or if the concentrations of the affected compound(s) are greater than 10x the blank level in the samples. In such cases, the sample results are accepted without corrective action for the high method blank result. The client must be notified, via the project narrative, of any method blank non-compliance associated with the sample results.

9.2 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

The laboratory control sample/laboratory control sample duplicate (LCS/LCSD) contains at least 10 PCB congeners that represent each Homolog group. If only PCB Congeners are being evaluated, and if the list includes more than 20 congeners, at least 16 individual congeners will be spiked. The LCS/LCSD is extracted along with the samples. An LCS/LCSD pair must be extracted and analyzed once per every 20 samples or per extraction batch, whichever is more frequent. The number of PCB congeners in this sample may vary with client specific requests.

The acceptable recovery QC limits are found in Section 12 for an aqueous, solid, tissue and PUF LCS/LCSD.

Corrective Action: Repeat analysis or check to see if an analytical error has occurred. If the LCS recovery is still out of control, re-extract and re-analyze the LCS/LCSD and all associated samples. Samples cannot be analyzed until an acceptable LCS/LCSD is obtained. Exceptions may be made with approval of the Section Supervisor if the samples associated with the out of control LCS/LCSD are also associated with a matrix spike and matrix spike duplicate that is in control which demonstrates an isolated problem pertaining to the LCS and/or LCSD only. An explanation of this out of control LCS and/or LCSD recovery must be included in the project narrative to the client and the sample data reported with the acceptable MS/MSD results as batch QC.

9.3 Initial Calibration Verification (ICV)

Refer to section 10.2.8

9.4 Continuing Calibration Verification (CCV)

Refer to section 10.4

9.5 Matrix Spike / Matrix Spike Duplicate (MS/MSD)

Matrix spike and matrix spike duplicate analyses are performed at the client's request.
The acceptable recovery and RPD QC limits are found in Section 12 for an aqueous, solid, tissue, and PUF MS/MSDs.

Corrective Action: Repeat analysis or check to see if an analytical error has occurred. If the % recovery or %RPD still exceeds the control limits and the associated LCS/LCSD is within control, include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the affected results as evidenced by the matrix spike and matrix spike duplicate exceedance.

9.6 Laboratory Duplicate

Laboratory matrix or sample duplicates are analyzed if requested by the client. The QC limit is 30% RPD for target compounds found above 5 times the reporting limit.

Corrective Action: If the %RPD exceeds the 30% control limit and the associated MS/MSD %RPD is within 30%, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results isolated to this sample, as evidenced by the matrix duplicate exceedance and the MS/MSD acceptance. If both the sample/duplicate and the MS/MSD exceed the control limits, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results as evidenced by the sample/duplicate and the MS/MSD exceedences.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

Surrogates are monitored for recovery for all matrices. The recovery limits are found in Section 12.

Corrective Action: Check to see if an analytical or dilution error occurred and re-calculate. If only one surrogate falls below the recovery limit, but is above 10% recovery, the exceedance is noted, with approval of the Section Supervisor, and the results are reported to the client with a notation in the case narrative. If all surrogates are recovered below the limit, re-extract the sample and report the re-extract results along with the original results, if re-extraction occurred beyond the holding time, and the re-extract surrogates are within the QC limits. If the surrogates are recovered below the limit in the re-extract, this confirms a suspected matrix interference on the surrogates, and only the original analysis needs to be reported. If the chromatogram shows obvious matrix interference, no re-analysis or re-extraction is necessary. *This decision must be made with approval of the Section Supervisor.* Surrogate outliers and sample re-extracts must be noted in the case narrative to the client.

9.7.2 Internal Standards

Internal standards are added to every field sample, QC sample, standard, and method blank. The acceptance limits are -50% to +100% of the internal standard response (or area) of the daily continuing calibration verification standard.

Corrective Action: Check to see if an analytical, dilution, or spiking error occurred. If the chromatogram shows obvious matrix interference, no re-analysis is necessary. *This decision must be made with approval of the Department Manager.* Note the exceedance in the case narrative to the client. If no obvious interference is present, re-analyze the extract. If internal standards are now within the

acceptance limits, report only the re-analysis, as long as the re-analysis occurred within the 40-day analytical hold time. If the re-analysis occurred outside of the 40-day analytical hold time, both the original and re-analysis must be reported. If the internal standards again are outside the acceptance limits, after re-analysis, either within or outside of the 40-day hold time, report only the original analysis, and include a narrative to the client that the suspected matrix interference on the internal standards was confirmed by sample re-analysis.

9.7.3 Standard Reference Materials

Standard reference materials (SRMs) are available from the National Institute of Standards and Technology (NIST) and are extracted and analyzed with samples on a project specific basis. These are not used as controls, but to evaluate potential matrix effects in associated samples for the target compounds being evaluated.

Acceptance criteria for SRM analysis will vary from project to project depending upon client data quality objectives (DQOs). Generally, 40% - 140% recovery of the true certified values of the target compounds of interest, serves as advisory acceptance criteria.

Corrective Action: Repeat analysis and/or check to see if an analytical error has occurred. If the % recovery or %D still exceeds the control limits and the associated LCS/LCSD and/or MS/MSD are within control, include a project narrative with the results to the client noting that the observed recovery exceedences of the SRM are isolated to this sample as evidenced by the LCS/LCSD and/or MS/MSD acceptance.

9.8 Method Sequence

Tune – (5 ug/mL) full scan using DFTPPBNA2/5
CCV – (50 ng/mL) using PCB209BNA2/5.m
Method Blank – (ID from sample preparation batch)
LCS – (ID from sample preparation batch)
LCSD – (ID from sample preparation batch)
Samples (up to 12-18 hours of analytical time)
Tune – (5 ug/mL) full scan using DFTPPBNA2/5
CCV – (50 ng/mL) using PCB209BNA2/5.m
Samples (up to 12-18 hours of analytical time)
CCV – (50 ng/mL) using PCB209BNA2/5.m

10. Procedure

10.1 Equipment Set-up

Prior to the analysis of any standards or samples, the instrument acquisition and processing methods must be set up. This includes the GC run parameters and the SIM mode acquisition ion entries into the different SIM acquisition retention time windows. An initial calibration must be analyzed to establish linearity of the instrument. First, the mass spectrometer must be tuned to the meet the abundance criteria for PFTBA when using maximum sensitivity tuning.

10.1.1 PFTBA Manual Tuning

10.1.1.1 Prior to initial calibration tune the mass spectrometer using PFTBA (Perfluorotributylamine - calibration gas) to maximize the sensitivity of the instrument in the mass range of interest, 45-525 amu. The use of PFTBA for MS tuning maximizes the sensitivity of the analysis within the mass/charge (m/e) range being monitored. *If only the PTFBA tune is required, per client request or project specific DQOs, the DFTPP tune in Section 10.1.2 does not need to be evaluated.*

10.1.1.2 To acquire the PFTBA Tune:

- Click on the “Instrument” icon to open the ChemStation.
- Go into “Instrument Control” in the “GC/MS Top Environmental” screen.
- Go to “View” and select “Manual Tune”.
- Go to “File” and select “Load Tune Values”. Select the ATUNE.U file.
- Go back into “File” and select “Generate Report”. The calibration gas will automatically turn ON, equilibrate for approximately 20 seconds, and generate a report. Evaluate the PFTBA tune against the parameters below.

PFTBA Ion	Relative Abundance
m/e 69	Base Peak with > 150,000 counts
m/e 219	40% to 90% of Base Peak
m/e 502	4% to 10% of Base Peak

If the PFTBA tune meets the criteria, “Save” the tune values, and exit the program.

10.1.1.3 If the PFTBA does not meet the criteria above, an experienced mass spectrometrist may attempt the following corrective actions:

- Adjust the ion focus value up or down while the calibration gas valve is open and continue to scan until the desired abundances are achieved.
- Adjust the entrance lens value up or down while the calibration gas valve is open and continue to scan until the desired abundances are achieved.
- Save the tune parameters under PCBTUNE.U.

10.1.2 DFTPP Tuning: If only DFTPP is required, per client request or project DQOs, PFTBA does not need to be evaluated.

10.1.2.1 Before the analytical standards are analyzed, the mass spectrometer must be evaluated for the proper ion criteria for DFTPP (decafluorotriphenylphosphene), if specifically requested by the client or included in a project specific QAPP. Generally, 1-4uL of a 10x dilution of 50 ug/mL solution is evaluated. A larger volume or lesser concentration may be evaluated if using large volume injections. If the instrument has been adjusted for the maximum sensitivity PFTBA, then the

criteria in Section 10.1.2.5 applies. *DFTPP must be injected under full scan mode.*

10.1.2.2 To acquire the DFTPP tune:

- Click on the "Instrument" icon to open the Chem Station, if not already open.
- *Go into the "GC/MS Top Environmental" screen.*
- *Go into "Sequence."*
- Edit the "Sample Table Log" by entering the "Vial" number starting at position 1, the "Data File ID" the acquisition method, DFTPPBNA2/5., and finally the "Sample Name" (*i.e*., "DFTPP 5 ug/mL") When complete, click "OK".
- Go back into "Sequence" and "Save" the sequence as the date, such as, S2060501.s. The ending "01" indicates the first sequence created on 06/05. The first number comes from instrument ID, i.e. BNA2.
- Go back into "Sequence" and "Load and Run" the sequence that was just saved.

10.1.2.3 After the analysis of the DFTPP, evaluate the tune as follows:

- Enter into the "Environmental Data Analysis" (off-line) screen.
- Go to "File" and select the tune data file.
- Go into "Tuner" and select "Eval DFTPP", then select "AutoFind DFTPP to Screen," to evaluate the tune file, based on the pre-set SW-846 criteria**.** The software will evaluate the tune by selecting three scans of the DFTPP peak and will display the ion intensities on the screen. That is, one scan at the apex, one scan directly preceding the apex and one scan following the apex and averages them, then takes one background subtracted scan, 20 seconds before the beginning of the DFTPP peak. If the criteria below are met, repeat, but select "AutoFind to Printer", for a hardcopy of the tune evaluation for the record.

  Note: the Maximum Sensitivity tune must be evaluated using the correct method to ensure the criteria in Section 10.1.2.5 are met.

10.1.2.4 If the "AutoFind" tune evaluation does not meet the criteria below, manual evaluation of the tune can be performed by attempting either of the options below:

- Blow up the DFTPP peak on the screen and select either one single scan at the apex of the peak, or a scan immediately preceding or following the apex. Go into "Tuner" and select "Evaluate DFTPP to Screen," or "Evaluate DFTPP to Printer," as described above, OR,
- Take the average of the scans across the entire peak. Go into "Tuner" and select "Evaluate DFTPP to Screen," or "Evaluate DFTPP to Printer," as described above.

10.1.2.5 "Maximum Sensitivity" DFTPP is used for the analysis of PCB Congeners by GC/MS. It shows high-end sensitivity for the higher molecular weight ions that are present in, and evaluated for PCBs.

DFTPP KEY MASSES AND ABUNDANCE CRITERIA
(Maximum Sensitivity)

Mass	m/e Abundance criteria
51	N/A
68	N/A
70	N/A
127	30-80 percent of mass 198.
197	Less than 3 percent of mass 198.
198	Greater than 40 percent of mass 442.
199	5-15 percent of mass 198.
275	15-50 percent of mass 198.
365	Greater than 3 percent of mass 198.
441	Present but less than mass 443.
442	Base peak, 100 percent relative abundance.
443	18-30 percent of mass 442.

10.1.2.6 Tune acceptance must be verified at the beginning of every analytical shift, and prior to the analysis of any standards, and again at the beginning of each 12-18 hour tune clock as defined by the injection time of each DFTPP analysis. If the DFTPP tune does not meet the criteria above, the PFTBA must be re-evaluated, and adjustments made by an experienced mass spectrometrist, to obtain an acceptable DFTPP tune, before continuing with any analysis.

10.1.3 GC Instrumental Conditions

10.1.3.1 For 209 Congener and Homolog Analysis inject an aliquot of 1uL to 50uL into the capillary column of the gas chromatograph at the following conditions. Injection volume (using the Large Volume Injector, LVI) amount will be dictated by project specific DQOs.

GC Parameter	*Setting*
Injector Temp:	*70 - 300 °C*
Transfer Line Temp:	*300 °C*
Initial Oven Temp:	*65°C*
Initial Hold Time:	*2.1 minutes*
Ramp Rate 1:	*25 °C / minute*
Final Temperature 1:	*170 °C*
Final Hold Time 1:	*0 minute*
Ramp Rate 2:	*3 °C / minute*
Final Temperature 2:	*290 °C*
Final Hold Time 2:	*15 minutes*
Total runtime:	*61.3 minutes*
Mode:	*Splitless / Constant Pressure or Solvent vent for LVI*
Purge:	*25 mL / minute – on at 2.00 minutes*
MS Temperature:	*250 °C, MS Source, 170 °C, MS Quad*

10.1.4 Mass Spectrometer Conditions

The effluent from the GC capillary column is fed directly into the ion source of the mass spectrometer. The MS is operated in the SIM mode using appropriate retention time windows to include the quantification and confirmation ions for each congener and the interference ions as shown in Table II.

10.1.5 Large Volume Injection (LVI) Parameters

*Gerstel **	*Settings**
Injector Temp:	*70 - 300 °C*
Initial Hold Time:	*30 sec. - 3 minutes*
Flow Rate:	*0.5 - 5.0 mL / minute*
Purge:	*25 mL / minute – on at 2.00 minutes*

Injection Volume:	*1uL - 50uL*

***** = The settings listed may vary from project to project, based on client specific DQOs. *Injection temperature, hold time, flow rate, purge time, and injection volume can affect chromatographic resolution and detection limits.* All parameters listed above can be set within the above setting ranges. Only a trained and experienced mass spectrometrist has the authority to change any setting. All standards and samples must be acquired using the same set of parameters. If any parameters are changed, a new initial calibration must be analyzed and accepted before any samples can be analyzed.

10.1.6 Data Acquisition Parameters

10.1.6.1 SIM Windows must be set up that bracket the expected retention times for each target analyte. These windows include the quantitation (primary) and confirmation ions for each congener Homolog group. To establish the expected retention time window ranges, the mid-level retention time window standard containing the first and last eluting congener in each Homolog group, must be analyzed in full scan mode. The resulting full scan analysis will dictate the windows in which the selected ions will be monitored. Depending upon the length of the analytical GC column, the time each window is selectively monitored may vary. The retention time windows must be shifted accordingly, when instrument maintenance is performed, (*i.e.,* the column is clipped).

10.1.6.2 The “dwell” time for each window should be set close to 20 and the resolution should be set to “high.”

10.2 Initial Calibration

10.2.1 Before analysis of sample extracts, establish a multi-point response factor calibration curve showing the linear range of the analysis for all target analytes in Table II. Use at least 5 levels of standard concentrations at 0.5, 1.0, 10, 20, 50, 200, and 500 or 400 ng/mL to construct the curve. See Section 8.16 for the preparation of the standard solutions for the initial calibration curve.

10.2.2 Construct an analytical sequence using the HP Enviroquant software:

- Click on the “Instrument” icon to open the ChemStation
- *Go into the “GC/MS Top Environmental” screen into “Sequence”*
- Edit the “Sample Table Log” by entering the “Vial” number starting at position 1, the “Data File ID” ,the acquisition method (such as, 209CONGBNA2.m, which indicates the type of method and the instrument ID), and finally the “Sample Name” (such as, “I206051001STD0.5”, for the first standard concentration level, etc.).
- Go back into “Sequence” and “Save” the sequence as the date, such as, S2060501.s. The ending “01” indicates the first sequence created on 06/05. The first number comes from instrument ID, i.e. BNA2.
- Go back in to “Sequence” and “Print” the sequence that was just saved. This will become part of the instrument run log. See Section 11.0 for additional instrument run log details.
- Go back into “Sequence” and “Load and Run” the sequence that was just saved.

10.2.3 When the sequence has finished running, the Enviroquant software will generate “Not Reviewed” quantitation reports. All reports must be “Quant Reviewed” before they can become part of the initial calibration processing method for sample analysis.

- Enter into the “Environmental Data Analysis” (off-line) screen.

- Go to "File" and under method, select the processing method (209Cong0605BNA2.m) that the initial calibration standards will be quantitated with.
- Go into "Quant" and select "QEdit Quant Results" to process the data files. See SOP Manual Integration 08-03 for manual integration details and Section 11.0 for processing of PCB Congener standards.
- When processing is complete for the first standard, "Save" the changes and "Exit." Re-print the re-processed data file by "Generating Quant Report," and save the hard copy for each level of the initial calibration.
- Repeat these steps for all initial calibration standards analyzed within the sequence.
- When the appropriate levels have been processed, go into "IntiCal," and select "Update Levels," and enter all levels for the initial calibration at the proper concentrations. Note: The PCB Homolog *group* responses *must be hand entered* into the calibration curve, or utilizing the RF macro, (*i.e.,* for Mono-, Dichlorobiphenyl, etc.) using the response of the appropriate calibration congener (*i.e.,* BZ1, BZ8, etc.).
- After all responses are entered, "Save" the completed method and print the resulting response factor summary by selecting "Response Factors to Printer."
- Acceptance Criteria: 20% RSD for all target compounds, except 10% of the analytes may be >20% RSD but ≤ 30% RSD. All calibration standards must be analyzed within 12-18 hours.
- Replace the Qion ratio values from the mid-point concentration level of the ICAL, by checking off the appropriate box, then update and "Save" the new method.
- Replace the reference spectra for the method from the spectra in the mid-point concentration level of the ICAL by going into "ConCal" and select "Update Reference Spectrum." Again, "Save" the method.
- Establish retention time window ranges from the first and last eluting congener within each chlorination level. Set the integration window ranges for each Homolog group (*i.e.,* Monochlorobiphenyl, Dichlorobiphenyl, etc.) by using the Easy ID function in the Enviroquant software.

10.2.4 If using greater than five calibration levels in the initial calibration, standards must only be excluded from either extreme. That is, the low-level standard or the high-level standard may be dropped to generate a five-level initial calibration. However, an intermediate-level calibration standard must not be dropped to convert a failing six-level initial calibration curve into a passing five-level initial calibration curve. Reduction in the number of calibration standards must also reduce the linear dynamic range used to quantify analytes in samples. The resulting average response factor for each target analyte in the initial calibration curve will be used by the computer software to calculate actual sample concentrations. See Section 11.0 for additional calculation details.

10.2.5 The following *corrective actions* are recommended for failing initial calibrations:

- Perform instrument maintenance and repeat the initial calibration, OR,
- Qualify all results reported for the analyte failing in the initial calibration, including all Homolog chlorination range(s) quantified using the suspect average response, and any non-detects. If the failure of the suspect average response appears related to a loss in MS sensitivity, instrument maintenance and repeat of the initial calibration curve must be performed.

The choice of corrective action must be made in consultation with the Section Supervisor, QA Manager, Project Manager, and/or the client. The reasoning for choosing the second option must be documented in the project narrative to the client.

10.2.6 Alternately, a linear regression model may be employed, provided that the coefficient of determination (COD or r^2) is ≥0.99. Otherwise, construct a nonlinear calibration of no more than a third order equation. Statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approach. A

quadratic (second order) model requires six standards, and a third order polynomial requires seven standards. In setting model parameters, do not force the line through the origin. The COD or r^2 must be greater than or equal to 0.99. The experienced analyst must select the regression order, which introduces the least calibration error into the quantitation.

10.2.7 Complete the initial calibration by filling out the *Initial Calibration Checklist*. The initial calibration, along with any corresponding continuing calibration data and sample data, is then forwarded for secondary review.

10.2.8 Initial Calibration Verification - ICV (separate source)

The analysis of separate source standard must follow the initial calibration curve.
After final processing, calculate the percent recovery of each congener by using the following calculation:

% Recovery = Found Amount / True Value x 100

Acceptance Criteria: All compounds must agree within +/-30%D with the exception of the partially or fully coeluting congeners that are identified, measured and calibrated together and at least one of the coeluting compounds is not present in the ICV solution. The Compounds listed in section 4.4 should also be excluded from the evaluation due to the described interference.

10.3 Equipment Operation and Sample Processing

10.3.1 Evaluate the PFTBA tune and/or DFTPP tune as described in Sections 10.1.1 and 10.1.2. *The type of tune required will depend upon the client/project DQOs.*

10.3.2 Samples are prioritized for analysis by the Organic Section Supervisor or GC/MS Group Leader based on client due date and sample analytical hold time. Samples are retrieved from the sample storage refrigerator, spiked with 20uL of the chosen internal standard solution per 1mL extract from either Section 8.11 or 8.12, and loaded into the instrument autosampler trays following the generalized sequence below.

- Tune – (5 ug/mL) full scan using DFTPPBNA2/5.m
- CCV – (50 ng/mL) using PCB209BNA2/5.m
- Method Blank – (ID from sample preparation batch)
- LCS – (ID from sample preparation batch)
- SRM – (ID from sample preparation batch) if required
- Samples (up to 12-18 hours of analytical time)
- Tune – (5 ug/mL) full scan using DFTPPBNA2/5.m
- CCV – (50 ng/mL) using PCB209BNA2/5.m
- Samples (up to 12-18 hours of analytical time)
- CCV – (50 ng/mL) using PCB209BNA2/5.m

10.3.3 Samples are processed from “Not Reviewed” data files, to “Quant Reviewed” data files in a similar way the standards were previously processed. See Section 11.0 for details on sample processing. If a CCV fails the criteria outlined in Section 10.4.3, all samples since the last acceptable CCV must be re-analyzed.

10.3.4 If the on-column concentration of any compound exceeds the concentration of the highest calibration standard, the sample must be diluted, re-spiked with the appropriate amount of internal standard and re-analyzed. Assuming all samples are at a 1mL final volume, the following example dilutions would apply. Adjust the volumes accordingly for other sample final volume amounts and other desired dilutions.

- 1:2 dilution = 500uL of sample : 500uL Hexane and 10uL of IS
- 1:4 dilution = 250uL of sample : 750uL Hexane and 15uL of IS
- 1:5 dilution = 200uL of sample : 800uL Hexane and 16uL of IS
- 1:10 dilution = 100uL of sample : 900uL Hexane and 18uL of IS, etc.

10.4 Continuing Calibration

A continuing calibration verification (CCV) standard, at the concentration of the mid-level of the initial calibration curve, must be analyzed at the beginning and end of every analytical sequence, and every 12-18 hours within the sequence, to confirm instrument stability, via response factor, for each calibrated congener.

10.4.1 After successful analysis of the PFTBA or DFTPP tune (Section 10.1.1 or 10.1.2), "Edit" the "Sample Table Log" to include the 50 ng/mL CCV standard and acquire the CCV against the correct initial calibration method. "Save," then "Load and Run" the sequence, as in Section 10.2.3.

10.4.2 When the sequence has finished running, the Enviroquant software will generate a "Not Reviewed" quantitation report. All reports must be "Quant Reviewed" against the processing method for sample analysis.

- Enter into the "Environmental Data Analysis" (off-line) screen.
- Go to "File" and under method, select the method that the CCV was analyzed under, then select the CCV data file.
- Go into "Quant" and select "QEdit Quant Results" to process the CCV file. See SOP Manual Integration 08-03 for manual integration details and Section 11.0 for processing of PCB congener standards.

When processing is complete, go into "ConCal," and select "Evaluate Data File as Continuing Calibration." **Note:** The PCB Homolog group CCV responses may be omitted since the calibration congener associated with the Homolog group, is the quantitation congener for that same Homolog group.

10.4.3 Acceptance Criteria: Compare the CCV resulting response against the average response for the initial calibration for each calibrated congener, and calculate the % difference (%D). See Section 11.0 for the calculations. The %D for each calibrated congener must be below 20%D, except up to 20% of the analytes may be > 20%D but ≤ 30%D. If multiple CCVs are analyzed within an analytical sequence, each CCV must be analyzed within 12-18 hours of the previous CCV, and each CCV, including the ending CCV, must meet the acceptance criteria.

Additional Criteria:

1) The area counts of the internal standards must be within 50-200%of the mid-point standard level from the most recent initial calibration.
2) The retention time of the internal standards must be within 30 seconds of the previous daily standard.

If the CCV meets the acceptance criteria, save the hard copy for each CCV standard and include it with the *Continuing Calibration Checklist*.

Go back into "ConCal" and select "Update Continuing Calibration" and "Save" the method updated to the opening CCV of the day.

10.4.4 If the CCV does not meet the criteria for each calibrated analyte, the following *corrective actions* are recommended:

- Perform instrument maintenance and re-analyze the continuing calibration standard and all affected samples, OR,
- Perform instrument maintenance and repeat the initial calibration, and re-analyze all affected samples, OR,
- If the closing CCV does not meet the criteria and the sample chromatograms show obvious matrix interference, no re-analysis is necessary. *This decision must be made with approval of the Section Supervisor.* CCV outliers and affected samples must be noted in the case narrative to the client.
- If the failure of the suspect response appears related to a loss in MS sensitivity or other instrument related issues, instrument maintenance and repeat analysis of all affected samples and/or the initial calibration curve must be performed.

The choice of corrective action must be made in consultation with the Department Manager, QA Manager, Project Manager, and/or the client. The reasoning for choosing the third option must be documented in the project narrative to the client.

10.5 Preventive Maintenance

10.5.1 Preventive maintenance may include the following: replacing glass liner, ferrules, PTV injection port bottom adapter and/or clipping a length of the analytical column.

10.5.2 Additionally, preventive maintenance for GC-MS system may involve baking out the injection port and the oven, cleaning the ion source, and/or replacing the analytical column.

11. Data Evaluation, Calculations and Reporting

11.1 After sample analysis, "Not Reviewed" quantitation reports are generated by the software system. It is expected that situations will arise when the automated quantitation procedures of the chromatographic software provide inappropriate quantitations or integrations. This normally occurs when there is compound co-elution, baseline noise or matrix interference with the PCB congener compounds. However, with PCB Homolog groups, a range or cluster of peaks is evaluated and manual integration must be performed for each PCB Homolog group or chlorination level cluster. See SOP Manual Integration 08-03 for manual integration details.

11.2 Identification of the PCB congener compounds are based on gas chromatographic relative retention times (RRTs) from the analysis of the mid-level initial calibration standard. For these compounds, manual quantitations are performed, if necessary, by integrating the area of the quantitation ion or peak. For the ten *PCB Homolog groups*, the Homolog groupings (*i.e.,* Dichlorobiphenyl) appear in the extracted ion current profiles (EICPs) as a cluster of congeners with the same degree of chlorination. Establish the pattern of each Homolog group by comparing the primary and secondary ion profiles. Refer to the Manual Integration SOP 1731 for details on Manual Integration. Manually integrate candidate peaks by straight-line integration to the baseline, taking into account background noise in the EICPs for each Homolog group within each determined Homolog-specific retention time window. If a discrete peak, either target or non-target, does not have a confirmation ion, or the experienced analyst judges that the confirmation ion does not meet the ratio criteria, the area for that discrete peak can be measured and subtracted from the total area used to calculate the concentration for that Homolog group. The experienced analyst can also choose not to include the interference in the

manual integration even if the interference appears within the retention time window established by retention time markers for specific Homolog group. If there is interference observed within Homolog-specific retention time window that cannot be excluded or subtracted out, the results for the affected Homolog group will be qualified with the G flag and narrated in the case narrative to the client. See the most recently generated "detailed" PCB reference spectrum hardcopy that is based on the most recent analysis mid-point standard of the ICAL. Table II, in Section 16.0, lists the representative ion(s) used for quantitation and confirmation of each parent congener and PCB Homolog group.

Note: Manual integration is not to be used solely to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system.

11.3 From EICP of the quantification (primary) mass ions and the confirmatory mass ions, identify all target analytes according to the following criteria:

- Surrogates and internal standards should meet the acceptance criteria in Section 12.0.
- Examine the chromatograms for evidence of saturated ions in mass spectra. Re-analyze the sample(s) at the appropriate dilution(s), see Section 10.3.4, as needed.
- The characteristic ions (primary and secondary) of each congener and/or Homolog group of interest should maximize at the same scan, or within one scan of each other.
- The retention time should fall within ± 10 seconds of the retention time of the authentic target compound or PCB Homolog grouping. *Note*: For PCB Homolog groups, the most intense peak within the group may not have the exact retention time of a calibration congener. Analyst judgment and referral to each Homolog groups' retention time window and group-specific pattern is essential for identification. Apply analyst judgment regarding corrective action, as needed, when these criteria are not met.
- The relative peak height of the quantitation ion compared to the confirmation ion for parent analyte should fall within ± 50 percent of the relative intensities of these ions in the reference mass spectrum (*i.e.,* the mid-level standard of the initial calibration curve).

Note: The relative intensities of the quantitation and confirmation ions may vary widely within a given group of PCB Homologs. Thus, the pattern of each PCB Homolog cluster, and the retention time window for the cluster, will be the primary identification criteria for PCB Homologs. In some instances, a parent congener that does not meet secondary ion confirmation criteria may still be determined to be present in a sample after close inspection of the data by the experienced mass spectrometrist. Supportive data includes the presence of the secondary ion, but ratio value greater than ± 50 percent of the primary ion, which may be caused by an interference of the secondary ion. See Section 11.6 for interferences.

11.4 In instances where manual integrations have been performed, they are assigned one of the Manual Integration codes, (Refer to Manual Integration SOP 1731) and can be found on the raw data provided within the data deliverable package The "detailed" report, displaying the manual integration, including an "m" qualifier(s) next to the modified or manually integrated compound(s), shall be provided to the LIMS for secondary review. These requirements apply to all standards, QC samples, field samples and blanks.

11.5 To calculate the ***Relative Standard Deviation*** (RSD) of all PCB congeners, Homolog groups, and surrogate compounds for the initial calibration, use the formula below. See Section 10.2 for initial calibration acceptance criteria. Additionally, use the initial multi-point calibration to determine ***Relative Response Factors*** (RRF_Is) at each concentration level, for each PCB congener. Average the RRF_Is from the initial multi-point calibration, to generate mean RRF_Is, for quantification of each PCB congener. Follow the same calculations for each surrogate

compound. The RRF_I for the quantification of each Homolog group is the RRF_I of the PCB calibration congener assigned to that Homolog group (*i.e.,* Tricholorbiphenyls are quantified using the RRF_I of the PCB calibration congener BZ29, which is associated with the Trichlorobiphenyl group). The RRF_Is are based on the internal standard compounds, and are calculated using the formula below. (The relative response factors for the continuing calibration verifications (RRF_Cs) are calculated using the same formula). See Section 16.0, Table II, for the listing of target compounds and their associated internal standards for quantification.

$$\textbf{RSD} = \textbf{SD / mean } RRF_I \textbf{ x 100}$$

where:

SD = Standard deviation between the five points, for that target analyte.

$$RRF_I = (A_c \times C_{IS}) / (A_{IS} \times C_c)$$

where:

A_c = Area of the characteristic ion for the standard compound to be measured.

A_{IS} = Area of the characteristic ion for the representative internal standard compound.

C_{IS} = Concentration of the representative internal standard compound (ng/mL).

C_c = Concentration of the standard compound to be measured (ng/mL).

Note: Assign the response factor of the calibration congener compound to the Homolog group, (*i.e.,* use BZ 5/8 for the Dichlorobiphenyl group).

11.6 Based on the mean RRF_Is, calculate the ***Sample Extract Amount*** for each PCB congener or Homolog group and surrogate in the sample extracts using the following formula:

$$Q_e = (A_a \times Q_{IS}) / (A_{IS} \times RRF_I)$$

where:

Q_e = Sample extract concentration (ng/mL) of target analyte, from quantitation report.

A_a = Area of the characteristic ion for the target analyte.

A_{IS} = Area of the characteristic ion for the representative internal standard compound.

Q_{IS} = Concentration (ng/mL) of representative internal standard compound, from quantitation report.t.

11.7 Calculate the ***Sample Concentration*** (C) for each PCB congener or Homolog group by the following formula:

$$C = (Q_e / V_s) \times FV \times DF$$

where:

C = Concentration in sample (ng/L water, ug/Kg sediment/tissue or ng/cert for PUF).

V_s = Original volume or weight of sample extracted, corrected for % solids, if applicable. (To correct for % solids, multiply the sample weight by the % solid as expressed as a decimal. For example: 15.24g x 0.843 = 12.85, for a sample size of 15.24g at 84.3% solid).

DF = Dilution factor

FV = Sample Final Volume or Final Effective.

If the response of any analyte in a sample exceeds the linear response range, as defined by the initial calibration standards in Section 10.2, dilute the extract so that the concentration of that analyte falls within the range of the calibration curve.

Note: Homolog groups exceed the calibration level if one single peak in the integration group exceeds the linear response range. If no single peak exceeds the range the total integration range may result above the calibration range. If the target compound in a sample is detected below the reporting limit (RL) but above half of the RL, qualify the reported concentration with a "J". If any target compound is found in the method blank and in the associated sample(s), qualify the reported concentration with a "B" if detected less than 10x the blank concentration for this analyte.

11.8 To calculate ***Total PCBs*** when analyzing for *less than 209 PCB Congeners* use the following:

- Each Homolog group will be identified and final concentration will be calculated using the formulas above.
- Sum all Homolog group concentrations.
- If a Homolog group is non-detect, zero is used in the summation. This minimizes the potential for a high bias result.

To calculate ***Total PCBs*** when analyzing for *all 209 PCB Congeners*, sum all identified congeners.

11.9 Calculate the ***Surrogate Recoveries*** relative to the internal standards by the following formula:

$$\%R_{sc} = (A_{sc} / A_{is}) \times (Q_{is} / Q_{sc}) \times (100 / RRF_{sc}) \times DF$$

where:

Q_{is} = Amount of the representative internal standard (ng).

DF = Dilution factor or fraction of the original extract.

11.10 Compare response factors for each PCB congener in the ***Continuing Calibration Verification*** (CCV), to those of the initial calibration curve by determining the percent difference.

$$\text{Percent Difference (\%D)} = ([RRF_I - RRF_C] / RRF_I) \times 100$$

where:

RRF_I = Mean response factor from initial calibration.

RRF_C = Response factor from CCV.

11.11 All results must be reported to three significant figures. All solids including soils, sediments, and sludge must be reported on a dry-weight basis. Tissue results may be reported on a dry-weight, or "as received," basis depending upon client request. PUF samples are reported "as received".

11.12 The primary analyst does data entry, or upload of the data, into the LIMS system. The LIMS is "linked" to the instrument, so the analyst must choose the sample(s) to be reported from that instrument's analytical sequence. All associated preparation and instrumental QC samples and dilutions are also chosen. Once the data/samples have been selected and "associated" with the proper QC samples, the batched data set is sent to print. In addition to the concentration of each selected PCB congener and Homolog group on the report, the Total PCB concentration is also reported.

11.13 The laboratory generates two types of data packages from the LIMS: "Commercial" or "Standard" for routine projects, and "Full Deliverable" or "CLP-like" for fully data validated projects. A Commercial/Standard package consists of sample results and the associated method blank and LCS/LCSD results. A Full Deliverable/CLP-Like package includes all sample results, all preparation and instrumental QC results and the associated supporting raw data. Check the "Report Type" on the project folder to ensure all required deliverables are included. A secondary review is performed on all data.

11.14 Procedures for data and record management must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP 1729. All records shall be stored in such a manner as to be safe and accessible for at least 10 years.

11.15 Notebooks: Laboratory notebooks are designed to accommodate the specific analysis. Instrument printouts are used to document run sequences, and each daily sequence printout is filed in a three-ring notebook. If a sample requires re-analysis or re-extraction for any reason, a notation is made next to the sample entry on the sequence log. Requests for re-extraction are further documented in the "Request for Re-extraction, Re-clean" logbook. At regular intervals the sequence run log is permanently bound, assigned an internal ID number, and filed accordingly. Such files shall be archived so as to remain available for at least 10 years. All laboratory notebooks must follow the specifications in the *Laboratory Notebook Usage* Work Instructions, WI 108-01, and all record keeping and document control practices.

11.16 Electronic records: All data files from computers, attached to instruments, shall be backed up daily onto the proper directory on the server. The backups shall be stored so as to be accessible for 10 years. Movement of the data files to the server is the responsibility of the primary analyst. Server backup and storage is the responsibility of the IT department.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All PCB congener and Homolog results are reportable without qualification if analytical holding times are met, preservation requirements (including cooler temperatures) are met, and all QC criteria defined in the table below are met. If any of the below QC parameters are not met, all associated samples must be evaluated for re-analysis. See Sections 9.0 and 10.0 for additional QC discussion including corrective actions for any QC outliers.

QC Parameter	Acceptance Criteria
Initial Calibration Curve	20% RSD for all target analytes with exception for 10% of target analytes to be >20%, but ≤ 30%
Independent Check Verification	+/- 30% recovery of the true values
Continuing Calibration Verification	Analyzed every 12-18 hours, 20% D for all target analytes with exception for 20% of target analytes to be >20%, but ≤ 30%
Method Blank	No analyte at or above the reporting limit, The results are qualified with a "B" for any associated sample concentrations that are less than 10x the blank concentration for this analyte.
Laboratory Control Samples (LCS/LCSD)	40-140%; 30% RPD (sporadic marginal failure criteria applies)
Matrix Spike / Matrix Spike Duplicate	Same as for LCS; 30% RPD between the duplicates.
Sample / Sample Duplicate	30% RPD between the duplicates.
Surrogates	50-125%
Internal Standards	50% - 200% of the daily CCV area for the Internal Standards
SRM	40% - 140% recovery

Section 9.0, Quality Control, defines the corrective actions that must be taken in instances where QC outliers exist.

If non-compliant PCB congener or Homolog results are to be reported, the Department Manager, the Laboratory Director, and/or the QA Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP 1732 MDL/LOD/LOQ Generation

SOP 1739 IDC/DOC Generation

SOP 1797 Waste Management and Disposal

SOP 1731 Manual Integration & Compound Rejection

16. Attachments

Table I: PCB Homolog Groups

Table II: PCB Homolog Quantification, Confirmation Ions

Table I: PCB Homolog Groups

Compound	Surrogate and IS Reference 209Cong	Compound	Surrogate and IS Reference
Monochlorobiphenyls	A	Surrogate Compounds	
Dichlorobiphenyls	A	DBOB	A
Trichlorobiphenyls	A	BZ 198	B
Tetrachlorobiphenyls	A	Carbon-labeled Surrogates	
Pentachlorobiphenyls	A	BZ 19	A
Hexachlorobiphenyls	A	BZ 202	B
Heptachlorobiphenyls	B		
Octachlorobiphenyls	B	Carbon-labeled ISs	
Nonachlorobiphenyls	B	BZ 15	A
Decachlorobiphenyl	B	BZ180	B

Note: Individual congeners may also be reported, as needed, depending upon client/project specific DQOs. Each congener depends on the level of chlorination would use the surrogates and internal standards similar to its chlorination grouping.

Table II: PCB Homolog Quantification, Confirmation Ions

Parameter	1^0 Ion	2^0 Ion	CASRN	Acceptance Ratio
Monochlorobiphenyls	188	190	27323-18-8	2.5-3.5
Dichlorobiphenyls	222	224	25512-42-9	1.3-1.7
Trichlorobiphenyls	256	258	25323-68-6	0.8-1.2
Tetrachlorobiphenyls	292	290	26914-33-0	1.1-1.5
Pentachlorobiphenyls	326	324	25429-29-2	1.4-1.8
Hexachlorobiphenyls	360	362	26601-64-9	1.0-1.4
Heptachlorobiphenyls	394	396	28655-71-2	0.8-1.2
Octachlorobiphenyls	428	430	31472-83-0	0.8-1.1
Nonachlorobiphenyls	464	466	53742-07-7	1.1-1.5
Decachlorobiphenyl	498	500	2051-24-3	0.9-1.3

Pace ANALYTICAL SERVICES	**ENV-SOP-ASHE-0009 v03_Sulfur by ASTM D4239**
	Effective Date: 04/10/2023

Management Approval:
Daniel Naylon Approved on 4/10/2023 10:33:17 AM
Eric Setzer Approved on 3/31/2023 9:14:55 AM
Felicia Grogan Approved on 3/30/2023 4:50:33 PM
Michael Washington Approved on 4/4/2023 4:43:58 PM
Jacob Cottrell Approved on 4/10/2023 10:36:02 AM

1.0 SCOPE AND APPLICATION

This SOP documents the procedure used by PASI – Asheville This adaptation of ASTM Method 4239 describes the determination of sulfur in waste materials using the LECO SC632 Sulfur Analyzer.

1.1 Target Analyte List and Limits of Quantitation (LOQ)

The PRL is 0.02%

1.2 Parameters:

1.2.1 Every sample is subjected to an oxidative-reduction process that first breaks down all sulfur bearing compounds, and then converts all sulfur to sulfur dioxides, which is detected by an infrared detector. These instruments are effective in the analysis of a wide variety of organic or inorganic waste materials.

1.2.2 Test results from this method are used primarily to show compliance with various EPA and State Environmental Regulations. These results may also be used by various manufacturing and service divisions to assist in process improvement.

1.2.3 This method is not included under the Laboratory NELAC Accreditation

1.3 Applicable Matrices

1.3.1 The results of this analysis are used to establish suitability and feed rates for burning of waste materials.

2.0 SUMMARY OF METHOD

2.1 The LECO Sulfur Analyzers are non-dispersive infrared instruments, which is software controlled and is calibrated to measure both high levels (>3%), and low levels (0 to 3%) of sulfur. Samples are weighed and placed in a sample boat, then pushed into a pure oxygen combustion chamber maintained at a temperature of 1350°C. All sulfur in the sample is then converted to SO2 gas in the high heat and swept through a series of filtering and drying tubes filled with anhydrous magnesium perchlorate, through a flow controller, which sets the sample gas flow to 3.5 L/min, and finally through one of the two IR detection cells. The detection cells are set at the wavelength specific to SO2. The detector measures the absorption in the cell, which is directly proportional to the concentration of sulfur in the sample. The instruments have a long cell for low concentrations and a short cell for high concentrations. A three coal standard series, at a sulfur concentration at the upper end of the detector cell range, with weights varying from 0.1 to 0.35 grams, is used to produce a calibration curve.

3.0 INTERFERENCES

The distribution of printed or electronic copies of this document outside of the PAS electronic document management system (eDMS) is not controlled or tracked. PAS employees must ensure any copy used for daily work, if not accessed directly from PAS eDMS, is the current version of the document.

3.1 Special Environmental Operating Conditions: The Sulfur Analyzers are computer controlled and may experience data system problems at ambient temperatures above 85°F.

3.2 Standard Contamination: If calibration standards are contaminated when an attempt is made to calibrate the instrument, it can affect the linearity of the calibration curve. Always discard a standard when 5 grams or less remain in the bottom of the bottle. Standards tend to become contaminated when they reach this level in the container.

3.3 For an extensive list of additional sources of error for this method and suggestions for eliminating them, refer to the troubleshooting matrix in Appendix 3.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (HSE) policies and procedures specified in this SOP and in the Pace Chemical Hygiene / Safety Manual.

Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. Use these acids in a fume hood whenever possible with additional PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. Any processes that emit large volumes of solvents (evaporation/concentration processes) must be in a hood or apparatus that prevents employee exposure.

Contact your supervisor or local HSE coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

Samples: Take precautions when handling samples. Samples are always to be treated as potentially hazardous “unknowns”. The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

The Sulfur Analyzers operate at very high temperatures. Handle sample boats with extreme caution when moving them in and out of the combustion chamber. Avoid contact with skin, clothing or other combustible materials.

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Infrared can be damaging to the eyes. Do not look into the combustion chamber when the door is open. Keep the analyzer shut until a sample is to be analyzed.

Magnesium perchlorate is a strong oxidizer. Do not dispose of it with other chemicals. Consult the Safety Data Sheet for full hazard details.

Must be disposed of in water to neutralize. Do not throw in garbage.

Use adequate ventilation when working with glass wool. It is a respiratory hazard.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

The laboratory does not perform sample collection for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with laboratory, *Bottle Preparation* (most current revision or replacement).

Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.

General Requirements

Matrix	Routine Container	Minimum Sample Amount[1]	Preservation	Holding Time
Solids	Glass bottle – cleaned and rinsed with reagent water	4/8 oz	Above zero to 6.0 ºC	There are no holding time requirements for this method.

Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory SOP, *Sample Management* (most current revision or replacement).

After receipt, samples are stored at ≤6°C until sample preparation (but not allowed to freeze).

After analysis, unless otherwise specified in the analytical services contract, samples are retained for 45 days from receipt and then disposed of in accordance with Federal, State, and Local regulations.

Note: Samples are to be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

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7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment and Supplies

Supplies*	Manufacturer* / Vendor	Model/ Version	Description /Catalog #/Comments*
Analyzer	Leco	SC 632	
Analyzer	Leco	SC S832DR	
Drying Oven	Precision	9606-003	103-105°C
	Thermo Fisher	41729011	≤100°C
Disposable Pipette	Fisher or equivalent	N/A	N/A
Balance	Sartorius	SECURA 124-1S	Analytical
Sample boats	N/A	N/A	N/A

*Or Equivalent

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent	Concentration/ Description	Requirements
Coal Calibration Check Standards	LECO, approximately 2 %. LECO part number 501-437	Reagents and standards are stored in desiccator.
Coal QC Check Standard	LECO, approximately 1 %. LECO part number 501-435	Reagents and standards are stored in desiccator.
Magnesium Perchlorate	(LECO Anhydrone, part number 501-171	Reagents and standards are stored in desiccator.
Magnesium Sulfate, 26.64% sulfur	26.64% sulfur, Aldrich, part number 24697-2	Reagents and standards are stored at room temperature.
Sulfur, 100%	Aldrich, part number 21392-2	

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Zinc Sulfide, 32.84% sulfur, or any certified standard at the 20-30% level for the high calibration standard.		Reagents and standards are stored at room temperature.
Compressed oxygen	medical grade	Reagents and standards are stored at room temperature.

9.0 PROCEDURE

9.1 Calibration and Standardization

Calibration Metric	Parameter / Frequency	Criteria	Comments
Initial Calibration Verification (ICV) (~1%)	Immediately after each initial calibration	% Diff ±10%	Second source single standard. Reanalyze once. If it still fails, recalibrate.
Continuing Calibration Verification (CCV) (~2%)	After every 10 samples and at the end of the analytical sequence.	% Diff ±10%	Second source single standard. Reanalyze once. If it still fails, recalibrate and reanalyze all samples after last passing CCV.

9.1.1.1 **NOTE: Calibration is typically done monthly unless the instrument fails standardization.**

9.1.2 All calibration standards that are to be used must be moisture free. To accomplish this, place standards in a drying oven at 104°C to 110°C for two hours prior to use, and then store them in desiccators.

9.1.3 The instrument remains on at all times, therefore the correct method settings and calibration is always loaded and ready for analysis.

9.1.4 Ensure that the instrument has been optimized and is functioning properly by clicking on diagnostics. Refer to attachment 1and 2.

9.1.5 Ensure that the appropriate method has been loaded. To change, click on cell in the “Methods” column to bring up the methods box.

9.1.6 On a balance that has been checked with reference weights, weigh the standard and analyze quickly to avoid moisture accumulation.

9.1.7 The instrument may need to be conditioned before attempting to calibrate by using a sample that has approximately 2 to 3% sulfur, labeled as “COND” in the sample file.

9.1.8 To enter “COND” standard to be analyzed, click “Add Sample” and put the cursor on the down pointing arrow at the bottom of the list of samples that have been run, and left click the mouse. Highlight “COND” standard that is to be run and hit enter.

9.1.9 Obtain a sample boat and place it on the balance and tare it. Add 0.25 g – 0.30 g of 2 3% sulfur.

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9.1.10 Ensure that there is not another sample boat already in the combustion tube.

9.1.11 Click on the “Analyze” button on the tool bar. The SC632 system will only respond with the "Push boat into furnace" message.

9.1.12 After the "Push boat into furnace" message, open the oven door and gently push the boat into the furnace and all the way to the back of the tube with the boat puller. Immediately close the door and hit “OK”. Specific to 632

9.1.13 Repeat steps 9.1.7 -9.1.11 three times to properly condition the instrument.

9.1.14 The instrument has two sulfur cells that must be calibrated individually. The low cell which includes values below approximately 3.0% sulfur, and the high cell which includes values from 3.0% to 100%. However, it is not necessary to have a high-level calibration in place in order to use the low-level cell. The low-level cell is adequate for most samples. A low cell calibration is accomplished by analyzing an appropriate standard, at a concentration as close as possible to the top of the cell range, at 3 different weights (0.1 g - 0.3 g), creating a calibration for 0 to 3.0% sulfur. It is not necessary to calibrate the high cell unless it is needed for concentrations above 3% sulfur.

9.1.15 To ensure that the appropriate standards are entered into the software, click on “Standards” on the tool bar to bring up the standards dialogue box. Check to see that the standards being used are listed. If they are, click “OK” and exit. If they are not, click “New”. Enter the name of the standard and known sulfur percentage and hit “enter”.

9.1.16 When using the SC632 instrument, simply click on the next open box at the end of the column and enter the sample name.

9.1.17 To enter the correct standard to be analyzed, put the cursor on the down pointing arrow at the bottom of the list of samples that have been run, and left click the mouse. Highlight the standard that is to be run and hit “enter”. This will list that standard as the next one to be run, and automatically move the cursor to the sample “Weight” column.

9.1.18 Obtain a sample boat and place it on the balance and tare it.

9.1.18.1 For the 632 – for the first calibration point Weigh the appropriate amount of the NIST calibration coal standard (weighed to the nearest mg)(0.10-0.15g) and enter the weight in the weight column.

9.1.19 Note: The system operates better, and thus produces a better calibration, if the sample is distributed evenly in the sample boat before being placed into the combustion tube.

9.1.20 Ensure that there is not another sample boat already in the combustion tube.

9.1.21 Click on the “Analyze” button on the tool bar. The SC 632 system will only respond with the "Push boat into furnace" message.

9.1.22 After the "Push boat into furnace" message on the SC 632, open the oven door and gently push the boat into the furnace and all the way to the back of the tube with the boat puller. Immediately close the door and hit “OK”.

9.1.23 Repeat this process with approximate weights of 0.1 g, 0.2 g, and 0.3g to complete a run of three standards for the low-level cell calibration.

9.1.24 This calibration run data can now be used to calibrate the system.

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9.1.25 Select the three low concentration standards, all the way left on the number column, by putting the cursor on the first one and clicking and dragging all the way down until all five standards are highlighted.

9.1.26 Click on the “Calibrate” button. The system will display a graph representing the low sulfur calibration curve and below the graph will be a table that indicates which samples it used on the graph and information about each point. Ensure that “Low Sulfur” is displayed and highlighted in the cell window. The curve type window displays “Linear, Fixed at Origin”, and the weighted window display “normal”.

9.1.27 Click on the “OK” button and a “save calibration” window will be displayed with “low sulfur” highlighted. Click “OK”.

9.1.28 The low concentration cell is now calibrated.

9.1.29 These calibrations can then be verified daily by analyzing the appropriate level of sulfur QC check standards (low level sulfur) or another appropriate level standard certified to be in the necessary range for a high-level calibration check.

9.1.30 A calibration check standard must also be analyzed prior to running any samples to verify the calibration.

9.1.31 To calibrate the high-level concentration cell, use a high-level sulfur standard (magnesium sulfate, zinc sulfide, cystene, or any other certified compound at the 20 to 30% sulfur range) following the same sequence as the low-level but highlight “High Sulfur” in the calibration dialog box. It is not necessary to calibrate the high-level cell to 100% sulfur unless a sample is expected to run higher than the set calibration will accommodate.

9.1.32 If a 100% sulfur calibration is required, use a standard certified to be 100% sulfur, and follow the procedure described above using weights in the range of 0.01 to 0.05 g. The weights must be smaller because the detector cell will saturate if larger weights are used.

9.2 Sample Procedure

9.2.1 Prior to the analysis of any samples on a given day, it may be necessary to condition the instrument. Analyze three “warm up” samples made from any other standard in the 2% to 3% sulfur range. Title these samples as “COND” so that they can be identified as conditioning samples.

9.2.2 Analysis of Daily Quality Check Standards - Use a certified standard at a percent sulfur level that is between zero and the calibration level. A quality check standard must be analyzed at the beginning and end of every set of ten samples.

9.2.3 The established limits of the certified coal standards are taken from the manufacturer’s stated precision limits printed on the bottle label. The ± number on the label is equal to 2 standard deviations. The limits of the daily standard are found by first dividing the label standard deviation by 2 then multiplying that number by 3. Plus or minus this number are the limits of that standard.

9.2.4 If a final quality check standard of a sample series is out of established limits, then the sample results, prior to that standard, back to the last check standard, are suspect and must be repeated.

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9.2.5 Troubleshoot and service the system to correct the problem. See attachment 2 and 3. A couple of conditioning runs may be required afterwards.

9.2.6 Reanalyze the quality check standard. If the result is within limits, proceed to reanalyze the samples. Analyze final quality check standard after the last waste sample.

9.2.7 If the standard result still does not fall within limits, even after servicing, check the parameters in Appendix 1.

9.2.8 If all systems check out and are within limits, recalibrate the instrument.

9.2.9 Analysis of a Waste Sample - Place a sample boat on the analytical balance and tare it. Add 0.25 to 0.30 grams of the waste sample. On the data terminal click on the next open cell in the column for the SC 632. Then click again in the “Sample” box to type in sample identification. Click “enter.”

9.2.10 Click on “Weight” and type in the sample weight or use the weight transfer key on the balance to electronically transfer the weight.

9.2.11 At this point, ensure that there is not another boat already in the furnace.

9.2.12 Click on “Analyze.” The SC632 system will only respond with the "Push boat into furnace" message. When the "Push boat into furnace" prompt appears, open the oven door and gently push the boat into the furnace and all the way to the back of the tube with the boat puller. Immediately close the door and hit OK on the screen.

9.2.13 It will take approximately two minutes for the sample to be analyzed and the results will be displayed in the “Low Sulfur” column, in the “Sulfur, (%S)” column, and on the graph that appears at the bottom of the screen.

9.2.14 Most samples that are analyzed on the SC 632 will be low level samples and the result can be taken directly from the % sulfur column on the main screen, which will match the result in the low-level sulfur column. However, some samples will cause the low-level cell to become saturated and will be analyzed using the high-level cell. The result in the % sulfur column will then match the result found in the high sulfur column only if the high cell has been calibrated. It is necessary to view and evaluate the individual cell data confirm or rule out saturation.

9.2.15 To view and evaluate individual cell data, click on “sample” at the top of the screen, and then click on “properties.”

9.2.16 Check the name and weight to ensure it is the sample of interest.

9.2.17 If not, exit that window, and highlight the appropriate sample in the sample column to the left side of the screen then reenter the cell information window.

9.2.18 If the sample is correct, click on the “Low sulfur” tab. The low cell has saturated when the saturation indicator has a check mark in it. The sample weight can be reduced to 0.10 g to prevent low-level cell saturation and the need to calibrate the high-level cell.

9.2.19 If the sample cannot be analyzed at 0.10 g without saturating the low-level cell, then the high-level cell must be calibrated, and the sample must be reanalyzed and bracketed with high-level standards.

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9.2.20 To ensure that the high-level cell is not saturated, hit the “High sulfur” tab and observe the saturation indicator for a highlight.

9.2.21 If a sample saturates the high-level cell, then the sample weight must be reduced, the high-level cell will have to be calibrated and the sample will have to be bracketed by high-level standards.

9.2.22 If the level of sulfur is so high that a 100% sulfur calibration must be used, use sample weights in the 0.01 to 0.05-gram range, just like the calibration weights.

9.2.23 When all samples have been analyzed (10 or fewer), analyze the final quality check standard.

9.2.24 If the final quality check standard is within limits, report the percent sulfur on the LIMS worksheet, then enter the results into LIMS.

9.2.25 For SC632 samples are manually posted at this time.

9.2.26 If a final quality check standard is out of limits, the sample data must be reanalyzed back to the last valid quality check standard.

9.2.27 Troubleshoot and service the instrument, including the reagent tubes. It may be necessary to analyze a couple of conditioning samples afterwards. Troubleshooting and service activity must be recorded in the instrument maintenance logbook including the problem, corrective action and results from recalibration or quality checks as well as the date and signature or initials of the analyst making the entry.

9.2.28 Reanalyze the quality check standard. If it meets acceptance criteria, reanalyze the remaining samples. If it still does not meet the acceptance criteria, then the system will have to be recalibrated.

9.2.29 Periodic Instrument Stabilization and Calibration – On a periodic basis (daily if the instrument is used daily), the stability of the instrument and its calibration must be verified by analyzing a sample of the certified coal standard that was used for calibration. The value determined for this standard must be within three standard deviations of the certified value.

9.2.30 The standard will be listed in the standards dialog box. Highlight “Verification” then follow steps 9.2.26-9.2.29.

9.2.31 If the system fails to meet this criterion, the system must be recalibrated and a verification check and quality check standard must meet requirements before analysis can proceed.

9.3 Data Reduction

9.3.1 Evaluate the calibration curve by plotting the instrument response against the standard concentration. Compute sample concentration by comparing sample response with the standard curve. Multiply answer by appropriate dilution factor.

9.3.2 Report only those values that fall between the lowest and the highest calibration standards. Samples that exceed the highest standard must be diluted and reanalyzed.

9.4 Instrument / Equipment Maintenance

9.4.1 Refer to instrument manual.

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9.4.2 The analyst is responsible for performing daily and routine maintenance on all instrumentation and recording all information.

9.5 Troubleshooting

9.5.1 Refer to instrument manual as needed.

10.0 DATA ANALYSIS AND CALCULATIONS

See the Laboratory Quality Assurance Manual for equations for common calculations.

10.1 All calculations are completed by the data system for reportable data.

10.2 Data is rounded to two significant figures.

10.3 The established limits of the certified coal standards are taken from the manufacturer's stated precision limits printed on the bottle label. The ± number on the label is equal to 2 standard deviations. The limits of the daily standard are found by first dividing the label standard deviation by 2 then multiplying that number by 3. Plus or minus this number are the limits of that standard. Acceptable standards must fall in this range.

11.0 QUALITY CONTROL

The following QC samples are prepared and analyzed with each batch of samples.

Calibration Metric	Parameter / Frequency	Criteria	Comments
Initial Calibration Verification (ICV) (~1%)	Daily before any samples have been analyzed. Immediately after each initial calibration	% Diff ±10%	Second source single standard. Reanalyze once. If it still fails, recalibrate.
Continuing Calibration Verification (CCV) (~2%)	After every 10 samples and at the end of the analytical sequence.	% Diff ±10%	Second source single standard. Reanalyze once. If it still fails, recalibrate and reanalyze all samples after last passing CCV.

11.1 Batch:

11.1.1 Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates), which are analyzed together as a group.

11.2 Method Performance

11.2.1 Method Validation

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11.2.1.1 **Detection Limits Not applicable**

11.2.2 Analyst Qualifications and Training

11.2.2.1 Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP *Orientation and Training Procedures* (most current revision or replacement) for more information.

12.0 DATA REVIEW AND CORRECTIVE ACTION

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP *Data Review Process* (most current revision or replacement) for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

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Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's SOP *Waste Handling and Management* (most current revision or replacement).

14.0 MODIFICATIONS

A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

Not applicable to this SOP.

15.0 RESPONSIBILITIES

Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

16.1 Attachment I - Maintenance for Leco Analyzer

16.2 Attachment II - Troubleshooting Matrix for Leco Analyzer

16.3 Attachment III - Instrument Parameter for Leco SC Analyzer

17.0 REFERENCES

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17.1 Pace Quality Assurance Manual- most current version.

17.2 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

17.3 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

17.4 American Standard Test Method D4239, Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods (Method C)", Annual Book of ASTM Standards_2002.

17.5 LECO SC632 Instruction Manual, 2007 LECO Corporation

18.0 REVISION HISTORY

18.1 This Version: ENV-SOP-ASHE-0009-Rev.02

Section	Description of Change
7.0	Added new analyzer, LECO SC832DR
9.1	Reworded balance check process
11.0	Added ICV requirement "Daily before samples are analyzed."
All	Updated to newer formatting

This Document Supersedes the following documents(s):

Document Number	Title	Version
ENV-SOP-ASHE-0009	Determination of Sulfur in Waste Materials	02

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Attachment 1

Maintenance for LECO Analyzer

1. If the reagent tubes material becomes moist during analysis, it will form a cake or crust on the surface, causing the gas flow through them to become severely hampered, causing poor instrument performance. A typical symptom of this is when the low concentration cell begins to saturate at concentration levels that previously did not saturate. The material in the tube needs to be changed. Also, if the reagent tube packing has become severely blackened and fouled by high carbon samples, the system response can be affected.

    1.1. Procedure: Slightly lift the reagent tube and remove it by pulling it out to the side. If the magnesium perchlorate packing material has moisture, caking or black soot accumulation, dump it out in an appropriate waste container.

    1.2. Inspect the O-rings on their metal seats for any obvious signs of damage.

    1.3. Use a small screwdriver or other tool to gently remove the O-rings from their seats. Do not damage them.

    1.4. Inspect for cuts or gouges. If they have no damage, they can be cleaned and reused. If they are damaged, they must be discarded and replaced.

    1.5. Use acetone and a cotton swab to clean the metal seats. Also use acetone and a cloth to clean the O-rings.

    1.6. Apply a thin coat of vacuum grease to the O-rings before placing them back in their seats.

    1.7. Remove the glass wool at the bottom of the reagent tube. Proceed to clean the reagent tube with acetone and a cloth.

    1.8. Replace the glass wool with approximately 0.5 inches of glass wool. Fill the space but do not pack it tightly. (The glass wool is at the bottom of the reagent tube)

    1.9. Refill the reagent tube with anhydrous magnesium perchlorate and reinstall the reagent tube, being careful not to get reagent dust under the O-ring and cause a gas leak.

2. A fouled disposable filter can become clogged, causing poor gas flow. Replace the filter when it turns gray, or the flow has become restricted.

    2.1. A fouled disposable filter can cause restricted flow.

    2.2. Unscrew the adapter.

    2.3. Ream out the adapter with the adapter brush and reinstall the reagent tube.

If a detector cell is not functioning properly, or a temperature is not at its set point, poor instrument performance can result. Ensure that the following parameters are at their set points by entering, “Tools” and “Diagnostics”. See Appendix 1 for Parameters.

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Attachment:2

Troubleshooting Matrix for LECO Analyzer

Error	Source	Problem	1.1. Action
Gas flow	Parameters	Low gas flow	Check flow rates and adjust if necessary.
	Tanks	No gas flow	Check gauges and replace tanks (PSI 60).
	Adaptor	Low gas flow	Remove and clean with adapter brush.
	Reagent Tube	Low gas flow: Moisture	Replace reagent tube materials.
	Disposable filter	Low gas flow	Check filter, if clogged (gray) replace.
Low Recovery Broad Tail	Outer Combustion Tube (OCT)	Leak: cracked tube Front O-ring	Replace Outer Combustion Tube. Reconnect or Replace.
	Inner Combustion Tube (ICT)	Leak: cracked tube ICT O-ring	Replace Inner Combustion Tube. Reconnect or Replace.
	Reagent Tubes	Leak	Replace 0-rings.
	Parameters	Poor Performance	Check Diagnostics reset if needed.
	Saturated IR Cell	Loss of Sulfur Dioxide	Reduce sample weight and reanalyze.
	Balance	Improper technique	Calibrations of scales.
High Recovery	Operator	Improper technique Contamination Concentrated samples	Proper use of scales. Use clean boat and clean standards. Run quality check standards frequently.
	Reagent Tube	Contamination	Clean reagent tubes and replace reagent tube materials.

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Attachment: 3

Instrument Parameters for LECO SC Analyzer

1. Oxygen inlet pressure: 35 psi
2. Oxygen flow: 3.5 lpm
3. Analyze flow: 2.5 lpm
4. Lance flow: 1.0 lpm
5. Low IR cell (gain): >1.5 V
6. High IR Cell (gain): >1.5 V
7. Furnace temperature: 1350° C (±25)
8. Oven temperature: 48° C (±2)

Note: Do not attempt to obtain a calibration if one or more of these voltages have drifted out of range. The check standards will not meet limits. The instrument software will alarm if these voltages drift out of tolerable range.

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Document Information

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Document Title: Calorific Value (BTU) or Waste Materials

Department(s): Wet Chemistry

Date Information

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Notes

Document Notes:

All Dates and Times are listed in: Central Time Zone

Signature Manifest

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ENV-SOP-ASHE-0015 Calorific Value (BTU) or Waste Materials

QM Approval

Name/Signature	Title	Date	Meaning/Reason
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Management Approval

Name/Signature	Title	Date	Meaning/Reason
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TEST METHOD STANDARD OPERATING PROCEDURE

TITLE:	DETERMINATION OF CALORIFIC VALUE (BTU/LB) OF WASTE MATERIALS USING A PARR 6300/6400 OXYGEN BOMB CALORIMETER
TEST METHOD	ASTM Method D 5468-95, Standard Test Method for Gross Calorific and Ash Value of Waste Materials.
ISSUER:	Pace ENV – Asheville– ASHE

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1.0 SCOPE AND APPLICATION

1.1 Target Analyte List and Limits of Quantitation (LOQ)

No Method Detection Limit (MDL) is performed for this test.

LOQ are established in accordance with Pace policy and SOPs for method validation and for the determination of detection limits (DL) and quantitation limits (LOQ). DL and LOQ are routinely verified and updated when needed.

The reporting limit (RL) is the value to which analytes are reported as detected or not detected in the final report. When the RL is less than the lower limit of quantitation (LLOQ), all detects and non-detects at the RL are qualitative. The LLOQ is the lowest point of the calibration curve used for each target analyte.

DL, LOQ, and RL are always adjusted to account for actual amounts used and for dilution.

Practical Quantitation Limit (PQL) = 50 BTU/lb (British Thermal Units)

1.2 The purpose of this method is to determine calorific value of waste materials.

1.2.1 Personnel: The policies and procedures contained in this SOP are applicable to all personnel involved in the analytical method.

1.2.2 Parameters: This adaptation of ASTM Method D 5468-95 describes the determination of the gross calorific value of waste materials by isoperibol (isothermal jacket) bomb calorimetry using the Parr 6300 /6400 bomb calorimeter.

1.2.3 The results from this analysis are used to establish suitability and feed rates for burning waste materials.

1.2.4 This method is not included under the Laboratory NELAC Accreditation

1.3 Applicable Matrices

1.3.1 The purpose of this method is to determine calorific value of waste materials.

2.0 SUMMARY OF METHOD

2.1 Calorific value is determined in this method by burning a weighed sample in oxygen under controlled conditions, in a calibrated calorimeter. The calorimeter is the isoperibol type and is calibrated by burning a specified amount of benzoic acid. The calorific value of the test sample is computed from temperature observations made before, during and after combustion and making proper allowances for heat contributions from other processes.

3.0 INTERFERENCES

Any printed copy of this SOP and all copies of this SOP outside of Pace are uncontrolled copies.
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3.1 The balance must be capable of weighing to the nearest 0.0001 g and must be checked for accuracy at the beginning of each day that calorimetric tests are to be conducted.

3.2 Weight determination in this analysis is critical. All crucibles must be clean and dry before weighing the sample(s).

3.3 Contamination by residual materials on the crucible, or within the bomb, can cause error in the Btu value.

3.4 For reproducibility of results, the sample needs to be homogeneous.

3.5 Ensure that the correct heat of combustion is used for all combustion aid spikes.

3.6 Any standard that is used must not absorb water, or it is stored with a desiccant. Error can occur from weight fluctuations and/or varying BTU values.

3.7 All fusing must be sized according to manufacturer specifications and have the correct Btu value for its length and weight. This correction factor is permanently set into the instrument, and any deviation from that amount of fuse will cause error in the BTU result.

3.8 The formation of nitric acid during an analysis produces an appreciable amount of heat, which can cause error. This heat is produced in every analysis because of the nitrogen present. The corresponding Btu can be set as a correction factor in the instrument and is consistent in every analysis.

3.9 Care must be taken to ensure that complete combustion has occurred. If not, large error can occur in the BTU value. This analysis will tolerate as much as 20% moisture in a sample and still permit complete combustion. If the sample does not burn completely, it will require a catalyst to aid in combustion.

3.10 All instrument sensors, if not functioning properly, can cause error, particularly the temperature sensors. If the water temperature in the reservoir is not maintained properly then error will occur. The instrument software is designed to monitor these parameters and flag them if an error occurs.

3.11 First, check the oxygen supply pressure (see that the regulator is set to appropriate pressure of 450 psi.) and replace a cylinder if required. Check your oxygen supply pressure is ok.

3.12 Some samples can be corrosive to the rubber seals in the bomb, so check the condition of, and replace if necessary, the quad-ring or the bomb release piston and seal O-rings.

3.13 If the daily calibration verification benzoic acid standard fails to meet limits, the most common reason is a bad seal at the bomb lid quad ring. Accumulation of residual waste at this seal can create a leak that will not show as a low oxygen error message, but it will prevent the instrument from passing the daily calibration verification standard analysis limits.

3.13.1 Remove the bomb lid and remove the rubber quad ring seal from its polished brass seat at the top of the bomb.

3.13.2 Replace the ring with a new one.

3.13.3 Clean the brass surface at the top of the open bomb, with acetone to remove the heavy residual accumulation. Use Crocus cloth to polish the brass surface. Scrub the entire surface with the cloth until the entire surface has a shiny luster.

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3.14 All water needs to be wiped off the firing wire and electrodes before every analysis. If not, the cotton fuse will fail to ignite, and the analysis will fail. Also, for consistent results, the bomb and lid need to be dried before each analysis.

3.15 The bomb calorimeter is intended for use indoors between 20 – 30℃ with relative humidity between 20%-80%.

3.16 Avoid making any changes to the oxygen supply pressure after the instrument has been calibrated. This will change the pressure of the oxygen in the bomb during analysis, and will void the calibration. The pressure of the oxygen will not exceed 450 psi.

3.17 "Fuse misfire" error message is given in the display when the firing did not occur during analysis. Some main causes of this error are:

3.18 Sample has water in it (This will require a catalyst to aid in combustion).

3.19 Fuse thread has become unattached (replace with new one).

3.20 Fuse thread has moisture on it (replace with new one).

3.21 Fuse wire is burnt in two & needs to be replaced.

3.22 The "water diffuser screw" located underneath the bracket holder is loose and will need to be tightened.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

5.0 HEALTH AND SAFETY

Standards and Reagents: The toxicity and carcinogenicity of standards and reagents used in this method have not been fully defined. Each chemical compound is treated as a potential health hazard. Reduce exposure by the use of gloves, lab coats and safety glasses. Safety Data Sheets (SDSs) are on file in the laboratory and available to all personnel. Standard solutions are prepared in a hood whenever possible.

Samples: Take precautions when handling samples. Samples are always treated as potentially hazardous "unknowns". The use of personal protective equipment (gloves, lab coats and safety glasses) is required when handling samples. In the event a sample container must be opened, it is recommended to perform this in a hood whenever possible.

Manufacturer operating instructions and guidelines for the Parr calorimeter shall be followed at all times.

The maximum mass of sample allowed for combustion is 1.5 grams. The bomb chamber could possibly explode if this mass is exceeded.

The pressure of the oxygen supply shall not exceed 450 psi. This pressure allows for a consistent oxygen pressure in the bomb chamber of 30 atm per analysis.

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Exercise caution when working with and around the oxygen supply line. It is made of plastic and could pose a hazard when pinched or cut. (The line is rated to 1000 psi.).

Inspect the bomb parts carefully after each use. All O-rings are to be replaced according to manufacturer specifications after 500 firings. After 500 firings the instrument software will give an alarm of "Exceeds bomb firing Count". Once routine maintenance is performed you will need to reset bomb firing counter.

Oxygen supply cylinders are to be equipped with approved regulators (300 to 500 psi discharge pressure).

To avoid damage to the instrument or bodily injury, do not fire loose fluffy material, or powdered samples unless thoroughly mixed with a combustion aid (mineral oil).

During ignition of a sample, the operator must not permit any portion of the operator's body to extend over the calorimeter.

Use extreme caution not to exceed the bomb manufacturer's recommended calorie limit of 8000 calories when using combustion aids, to avoid damage to the instrument or bodily injury.

Normal laboratory safety practices are to be observed at all times. Always wear proper PPE.

The Parr 6400/6300 is equipped with a bomb washings reservoir, which has to be emptied. This waste and all other sample waste are to be disposed of in accordance with laboratory policy.

6.0 SAMPLE COLLECTION, PRESERVATION, HOLDING TIME, AND STORAGE

6.1 The laboratory does not perform sample collection for this test method. To assure sample collection and field checks and treatment are performed in accordance with applicable regulations Pace project managers will inform the client of these requirements at the time of request for analytical services when the request for testing is received prior to sample collection. If samples were already collected, the laboratory will record any nonconformance to these requirements in the laboratory's sample receipt record when sufficient information about sample collection is provided with the samples.

6.2 The laboratory will provide containers for the collection of samples upon client request for analytical services. Bottle kits are prepared in accordance with laboratory, *Bottle Preparation* (most current revision or replacement).

6.3 Requirements for container type, preservation, and field quality control (QC) for the common list of test methods offered by Pace are included in the laboratory's quality manual.

General Requirements

Matrix	Routine Container	Minimum Sample Amount	Preservation	Holding Time
Solids	Glass bottle – cleaned and rinsed with reagent water	4/8 oz	above zero to 6.0 ºC	There are no holding time requirements for this method.

6.3.1 Thermal preservation is checked and recorded on receipt in the laboratory in accordance with laboratory SOP, *Sample Management* (most current revision or replacement).

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6.3.2 After receipt, samples are stored at ≤6°C until sample preparation (but not allowed to freeze).

6.3.3 After analysis, unless otherwise specified in the analytical services contract, samples are retained for 45 days from receipt and then disposed of in accordance with Federal, State, and Local regulations.

6.3.4 Note: Samples are to be stored separately from all standards, reagents, and highly contaminated samples. To avoid contamination, no food or drink products can be located near samples.

6.3.5 Care must be taken to eliminate the possibility of contamination during collection, transport, storage and analysis of samples. Samples stored in the lab must be kept separate from standards.

6.3.6 Sample Storage – Before transferring the portion of the sample to be ashed, to the evaporating crucible, take particular care to ensure that the portion taken is truly representative of the larger portion. Vigorous shaking could be necessary.

7.0 EQUIPMENT AND SUPPLIES

7.1 Equipment and Supplies

Supplies*	Manufacturer*/Vendor	Model/Version	Catalog#	Description/Comments*
Isoperibol (Isothermal-Jacket) Bomb Calorimeter	Parr	Parr 6400 and 6300	N/A	meets all criteria in ASTM Method D5468- 95
Sample Combustion Crucible	Parr	Fused silica	N/A	
Sample Combustion Crucible	Parr	Metal	N/A	Corrosion resistant Pt-Rh.
Disposable Pipette	Fisher	N/A	N/A	N/A
Balance	Sartorius	SECURA 124-1S	N/A	Analytical capable of weighing to 0.0001 gram
Desiccator	N/A	N/A	N/A	Minimum 14”x20”x12”

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Supplies*	Manufacturer*/Vendor	Model/Version	Catalog#	Description/Comments*
Pipette Volumetric	N/A	Fisher	N/A	Checked Quarterly
Nitrogen Supply System	Airgas	N/A	N/A	A Nitrogen Supply system ca psi supply pressure.
Ignition Thread	PARR	PARR	845DD2	4”

*Or Equivalent

8.0 REAGENTS AND STANDARDS

8.1 Reagents

Reagent	Conc.	Purity	Manufacturer	Vendor	Catalog #	Expiration / Other
Water (H2O)	Unless otherwise indicated, references to water will mean deionized water.	N/A	N/A	N/A	N/A	N/A
Benzoic Acid Standard (C6H5COOH) :	Pellets of approximately 1 gram with certified heat of combustion.		Parr	Fisher	23-279045	Used for calibration &monthly calibration check.
Benzoic Acid Standard (C6H5COOH) :	Pellets of approximately 1 gram, with certified value for heat of combustion		Leco	Leco	774-208-150	Used for daily calibration check.

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Oxygen (O2)		medical grade	Airgas	Airgas		
Mineral Oil						Commercially available
3MNon-Halogenated Cellulose Tape	Parr, #517A					
Crocus cloth	9 x 11 sheets 001K, Crystal Bay Crocus Cloth.					

9.0 PROCEDURE

9.1 Calibration and Standardization – N/A

9.1.1 NOTE: The "Clear Entry" key can be used to clear error messages due to incorrect operations; do not press "Reset" with a sample loaded in the bomb calorimeter. This procedure is used in the setup of a new instrument or if a major change has been made to the instrument. Use "Parr benzoic acid tablets" to calibrate.

9.1.2 To "calibrate" the bomb calorimeter is to determine the heat capacity of the bomb and its components by burning a standard material with a known heat of combustion. This "heat capacity" or "energy equivalent" of the instrument is used in the calculation of the heat of combustion of all samples. Benzoic acid is used for this purpose.

9.1.2.1 Determine the initial heat capacity of a new bomb as the average of a minimum of 6 individual runs made over a period of not less than 3 days or more than 5 days. To be acceptable for high precision work, the relative standard deviation of the series shall be 0.15% or less. Any individual test may be discarded only if there is evidence of incomplete combustion. If this limit is not met, the entire series shall be repeated until a series is obtained with a relative standard deviation below the acceptable limit.

9.1.3 All crucibles made of base-metal alloys must be thoroughly heat treated before using for analysis, by baking in an oven for 4 hours at 500oC. If metal crucibles are not thoroughly oxidized, error in BTU measurement can occur.

9.1.4 The Parr cotton fuse must be prepared before any calorimeter analyses can be carried out.

9.1.4.1 Use precut thread.

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9.1.5 Each tablet of benzoic acid is to be weighed, to the nearest 0.0001 g, in the crucible in which it will be burned, on each day of use.

9.1.6 The calorimeter is the isoperibol type, which means that the calorimeter jacket temperature is maintained constant throughout the analysis. The instrument software calculates a temperature correction for the system to allow for any heat lost by the jacket.

9.1.6.1 The calculation for the corrected temperature rise is as follows:

$$t = t_c - t_a + C_r$$

Where: t = corrected temperature rise, °C

ta = initial temperature reading at time of firing

tc = final temperature reading

Cr = temperature correction

9.1.6.2 The calculations and Thermochemical corrections used by the instrument software, for the heat capacity of the bomb, are as follows:

$$E = [(H_c m) + e_1 + e_2] / t$$

Where: E = calorimeter heat capacity or energy equivalent

Hc = known heat of combustion of benzoic acid, cal/g.

M = mass of benzoic acid, g.

e1 = correction for heat of formation of nitric acid, 10 cal.

e2 = correction for heat of combustion for the fuse, 50 cal.

T = corrected temperature rise, °C.

9.1.7 The software keeps 10 heat capacity values in memory and uses the average of them in the calculation.

9.1.7.1 From “Main Menu”, arrow to “Calorimeter Operation” and press “Enter”. “Bomb Installed/EE”, is the current bomb configuration and current energy equivalent average being used by the calorimeter. The bomb configuration (#1) stays the same all the time since the instrument is recalibrated if a change is made with the bomb. The minimum number of average values that may be used is 10.

9.1.8 It is necessary to have all Thermochemical corrections in place before attempting to calibrate the instrument. The heats of combustion for the fuse (50 cal/g) and the formation of nitric acid (10 cal/g) must be entered in the system; however, a correction for the formation of sulfuric acid is not required here. Benzoic acid has no sulfur. This may be required later for a sample that has been found to contain a significant amount of sulfur.

9.1.8.1 To enter the Thermochemical corrections into the instrument, observe the instrument command screen.

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9.1.8.1.1 At "Main Menu", arrow down to "Thermochemical Corrections" and press "Enter".

9.1.8.1.2 All selections on this page are to be set "ON", except "Calculate Net Heat of Combustion". This selection is turned "OFF".

9.1.8.1.3 Enter the values for "Fixed Fuse Correction" and "Fixed Acid Correction" (50 cal/g and 10 cal/g, respectively) for both Standardization and Determination sections. Use the arrow keys to move through the table. When the value to be changed is highlighted, press "Skip". Type in the new value and press "Enter". Once these values have been entered into the system, they will default and won't need to be entered again.

9.1.9 "Escape" back to "Main Menu.

9.1.10 Arrow to "Calorimeter Operations" and press "Enter".

9.1.11 Operation Mode is set on "STD" by pressing the "Yes" button.

9.1.12 Arrow to "Heater and Pump". Turn "On" by pressing "Yes".

9.1.13 Allow instrument to warm up approximately 10-15 min. The Jacket temperature will be 30°C ± 1.05.

9.1.14 Press "Start the pretest." This initiates an instrument self-test, in which the instrument will go through the entire analysis process with the exception of actually firing. This ensures that all parameters are correct, and that the system is ready for use. When "Idle" is displayed analysis can proceed.

9.1.15 Place bomb head on rack.

9.1.16 Remove crucible from balance and place in crucible holder of the bomb head.

9.1.17 Attach a fuse thread to the firing wire. Using tweezers, lift benzoic acid tablet and place thread underneath to insure proper ignition.

9.1.18 Place the bomb head in the bucket turning clockwise.

9.1.19 Close lid.

9.1.20 When the analysis has finished the software will generate a heat capacity for the bomb (EE).

9.1.21 Press "Start"

9.1.22 "Bomb ID" will appear on screen this defaults to 1 or 2 depending on the bomb you are using, press "Enter".

9.1.23 "Sample ID" will appear on screen. This defaults to the next sequence number. You may press "Enter" at this point or type in your own 4-digit ID.

9.1.24 The instrument will prompt for sample weight. At this point, place a clean, dry sample crucible on scales. Zero. Add a Parr benzoic acid tablet

9.1.25 Press "Enter" to automatically enter weight or press "Skip" to enter weight manually.

9.1.26 The instrument will prompt to enter a spike weight. Press "Skip" to bypass this option and "Enter"

9.1.27 Open instrument lid. Remove bomb head by turning counterclockwise and lifting.

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9.1.28 A total of ten benzoic acid tablet analysis runs must be done within a 3-5-day period. These runs will be averaged by the instrument software. The RSD for the runs must not exceed 0.15% difference. If this limit is not met, the cause of the high standard deviation in the calibration series must be found and corrected and the calibration procedure repeated.

9.1.29 If a major component of the bomb or calorimeter is changed, the above process will need to be repeated to ensure appropriate calibration.

9.1.30 The calibration is checked once per month by running a second source benzoic acid tablet (Parr). This check must be within 1% of true value.

9.1.31 To clear Memory:

9.1.31.1 Press “CLEAR MEM.” Key

9.1.31.2 At the “From ID” prompt, enter the beginning ID number of the data to be cleared and press “Enter”.

9.1.31.3 At the “To ID” prompt, enter the ending ID number of the data to be cleared and press “Enter”. (To clear only one data point ID, enter the same number at both prompts.)

9.1.31.4 Instrument will indicate the data has been cleared.

9.1.32 After a proper calibration is in place, a study must be conducted to establish heat combustion for all spiking materials.

9.1.33 Analyze mineral oil as a sample, using 0.5-0.7g. Ensure that at least 6000 calories are generated. If the total calorie count is less than 6000, increase the sample size and repeat the analysis until the 6000-calorie minimum is achieved.

9.1.34 Analyze a total of 10 and average the Btu/lb values to obtain the heat of combustion for the spike.

9.2 Sample Preparation

9.2.1 Batch samples in groups of 20 or less using the associated A-code in EpicPro.

9.2.2 After selecting the samples, click the “Build Batch” button. This will put the sample in a unique Batch with its own Batch number and default QC samples with their own QC number IDs. If necessary, add QC by using the “Add QC” button. The cursor must be on a particular sample to add a DUP.

9.2.3 Deleting QC or samples is done by putting the cursor on what you want to delete and clicking the red “Delete Record” button in the Toolbar Menu. Press “F10” (save) when all is done. Print the newly created Batch Work list report using the Print button in the EpicPro toolbar. Select “All” for all pages to print.

9.2.4 Print the Batch Compound report for the analyst by going “System” > “Submit Job” > “F9” > “Batch Compound List” > “Enter” or “OK” > Select “WETA” as Queue and enter the Batch number > “F10”. Print using the Print command in the EpicPro toolbar, select “All” > “OK”. This will give the analyst a report of analytes to report per sample.

9.2.5 Pull samples from storage refrigerator.

9.3 Sample Procedure

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9.3.1 A daily Benzoic acid standard must be analyzed as a calibration verification at the beginning and end of each run. Calibration verification must also be run every 10 runs.

9.3.1.1 Use a Parr tablet for the daily calibration

9.3.1.2 Use a LECO table for the monthly calibration

9.3.2 The following procedure can be used to run the daily calibration verification as well as samples.

9.3.2.1 Arrow to “Calorimeter Operations” and press “Enter.

9.3.2.2 Operation Mode is set on “DETR” by pressing the “Yes” or “No” buttons.

9.3.2.3 Arrow to “Heater and Pump”. Turn “On” by pressing “Yes”.

9.3.2.4 Allow instrument to warm up approximately 10-15 min. The Jacket temperature is 30 °C ± 1.05.

9.3.3 Press “Start the pretest”.

9.3.3.1 This initiates an instrument self-test, in which the instrument will go through the entire analysis process with the exception of actually firing. This ensures that all parameters are correct, and that the system is ready for use. When “Idle” is displayed, analysis can proceed.

9.3.4 Analysis

9.3.4.1 Press “Start”.

9.3.4.2 Enter the Spike weight

9.3.4.3 Weight the sample and place it in the bomb head.

9.3.4.4 Enter the information into the instrument and follow the steps below

9.3.4.5 Press "Enter” to automatically enter weight or press “Skip” to enter weight manually.

9.3.4.6 The instrument will prompt for a spike weight. Press “Skip” to bypass this option or, if adding a spike, zero the balance. Add appropriate amount of spike to the sample and press “Enter”.

9.3.4.7 Open instrument lid. Remove bomb head by turning counterclockwise and lifting.

9.3.4.8 Place bomb head on rack.

9.3.4.9 Remove crucible from balance and place in crucible holder of the bomb head.

9.3.4.10 Attach a fuse thread to the firing wire. Make sure fuse thread is touching the sample to ensure proper ignition.

9.3.4.11 Place the bomb head in the bucket turning clockwise.

9.3.4.12 Close lid. The analysis will start automatically.

9.3.4.13 When analysis has finished, the software will generate a heat of capacity for the bomb (EE).

9.3.5 Two criteria determine whether the results of an analysis can be accepted.

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9.3.5.1 The sample must have completely combusted, leaving nothing but moisture and in some cases, a small amount of dark ash.

9.3.5.2 The sample must also have produced a temperature rise of at so that the calorie count is in the acceptable range of the instrument calibration.

9.3.5.2.1 Temp rise limits

9.3.5.2.1.1 6300 is 7.5-11°C

9.3.5.2.1.2 6400 is 5.1-8.5°C

9.3.5.3 If the above criteria are met, then the Btu/lb value of the sample can be reported as valid.

9.3.5.4 If not, either the sample must be reanalyzed using a larger sample size or the sample must be spiked. Use the following equations to evaluate the next analysis procedure.

9.3.6 Once you are finished using the instrument for the day, press the “No” key to turn off the heater and pump.

9.3.7 Check the waste reservoir underneath the cabinet and if it needs to be emptied, do so at this time.

9.4 Instrument / Equipment Maintenance

9.4.1 Refer to instrument manual.

9.4.2 The analyst is responsible for performing daily and routine maintenance on all instrumentation and recording all information.

9.4.3 Daily

9.4.3.1 Clean Small Quad Ring

9.4.3.2 Clean Electrode pins

9.4.3.3 Clean Bomb head seal

9.4.3.4 Verify glycerin correction factor and record on bench sheet.

9.4.4 Monthly

9.4.4.1 Clean, remove, and inspect Cylinder, and Sealing area in Cylinder

9.4.4.2 Wet the hole in the center of the head

9.4.4.3 Refill rinse tank

9.4.4.4 Clean electrode contact points

9.4.4.5 Inspect critical bomb head O-rings

9.4.4.6 ICV monthly QC Check (LECO -Benzoic Acid Tablet)

9.4.4.7 Inspect all tubing and wiring

9.4.4.8 Replace ignition wire

9.4.4.9 500 Count Maintenance

9.5 Troubleshooting

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9.5.1 Refer to instrument manual as needed and attachment II.

10.0 DATA ANALYSIS AND CALCULATIONS

10.1 See the Laboratory Quality Assurance Manual for equations for common calculations.

10.2 The following calculation can be used to help in determining the amount of spike needed for spiking a sample.

10.2.1 Calculate Calories/gram = (BTU/lb) x (0.56).

10.2.2 Calculate Total Calories = (Calories/gram) x (g sample).

10.2.3 Estimate the calorie output if the sample size was increased to the limit of 1.1 grams: Calories = (Calories/gram) x (1.1 g sample).

10.2.4 If it is estimated that this weight would give 6000 calories or more, proceed to prepare another sample at this weight and reanalyze.

10.2.5 If increasing the sample size is not expected to produce the necessary calorie count, the sample will have to be spiked with a combustion aid (mineral oil).

10.2.6 After determining the total calories from 0.7 g of sample, estimate the amount of mineral oil spike, which will have to be added to the sample to obtain the necessary 6000 calories using the following equations.

10.2.6.1 Calories required from spike = 6000 – Total Calories from sample.

10.2.6.2 Spike weight = 0.00009 x Calories required from spike.

10.2.6.3 Use caution when calculating the spike weight. Do not exceed 1.1g total weight limit or the calorie limit of 8000 calories.

10.2.7 Repeat the analysis procedure starting at Section 9.3.4.

10.2.8 If the acceptance criteria stated in Section 9.3.5 are met, the Btu/lb value can be reported. If not, repeat the analysis with a slightly higher spike weight until the criteria is met, being careful not to exceed the 1.1g total weight!

10.2.8.1 Calculation Example: 8842.1074 (BTU value) x 0.56 = 4912 x 0.7052 (sample weight) = 3464 (number of calories produced).

10.2.8.2 6000 (calories needed) – 3464 (calories produced) = 2536 (how many more calories are needed). 2536 x 0.00009 = (gram of spike needed).

10.3 The calculation used by the instrument software to obtain the heat of combustion of a sample is as follows:

10.3.1 Qg = Gross calorific value (heat of combustion) in BTU/lb.

10.3.2 Corrected temperature change in calorimeter degrees C.

10.3.3 Heat capacity or energy equivalent of the calorimeter in cal / degrees C.

10.3.4 Thermochemical correction for the formation of nitric acid, (10 cal/g) x (g HNO3); this correction is considered a constant value for every analysis.

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10.3.5 Thermochemical correction for the combustion of the fuse thread, (50 cal/g) x (g fuse thread); this correction is considered a constant value for every analysis.

10.3.6 Thermochemical correction for use of spiking material, (heat of combustion of spiking material) x (g spiking material).

11.0 QUALITY CONTROL

11.1 QC sample, components, frequency, acceptance criteria and corrective action.

QC Sample	Components	Frequency	Acceptance Criteria	Corrective Action
Sample Duplicate	Sample	Two per batch of 20 samples.	RPD ≤10%	Report results with appropriate footnote.
Continuing Calibration Verification (CCV)	Benzoic Acid Tablet	At start of run, every 10 runs (excluding misfires), and after last sample.	99% to 101% of True Value. TV = 11373	Rerun once. If rerun does not pass. Perform maintenance and recalibrate.
Initial Calibration Verification (ICV)	Benzoic acid table from second source.	Monthly	99% to 101% of True Value. TV = 11373	Rerun once. If rerun does not pass. Perform maintenance and recalibrate.

11.2 Batch:

11.2.1 Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same NELAC-defined matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates), which are analyzed together as a group.

11.3 Sample Concentration:

11.3.1 Not applicable

11.4 Method Performance

11.4.1 Method Validation

11.4.1.1 **Detection Limits Not applicable**

11.4.2 Analyst Qualifications and Training

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Employees that perform any step of this procedure must have a completed Read and Acknowledgment Statement for this version of the SOP in their training record. In addition, prior to unsupervised (independent) work on any client sample, analysts that prepare or analyze samples must have successful initial demonstration of capability (IDOC) and must successfully demonstrate on-going proficiency on an annual basis. Successful means the initial and on-going DOC met criteria, documentation of the DOC is complete, and the DOC record is in the employee's training file. Refer to laboratory SOP *Orientation and Training Procedures* (most current revision or replacement) for more information.

12.0 DATA REVIEW AND CORRECTIVE ACTION

12.1 Data Review

Pace's data review process includes a series of checks performed at different stages of the analytical process by different people to ensure that SOPs were followed, the analytical record is complete and properly documented, proper corrective actions were taken for QC failure and other nonconformance(s), and that test results are reported with proper qualification.

The review steps and checks that occur as employee's complete tasks and review their own work is called primary review.

All data and results are also reviewed by an experienced peer or supervisor. Secondary review is performed to verify SOPs were followed, that calibration, instrument performance, and QC criteria were met and/or proper corrective actions were taken, qualitative ID and quantitative measurement is accurate, all manual integrations are justified and documented in accordance with the Pace ENV's SOP for manual integration, calculations are correct, the analytical record is complete and traceable, and that results are properly qualified.

A third-level review, called a completeness check, is performed by reporting or project management staff to verify the data report is not missing information and project specifications were met.

Refer to laboratory SOP *Data Review Process* (most current revision or replacement) for specific instructions and requirements for each step of the data review process.

12.2 Corrective Action

Corrective action is expected any time QC or sample results are not within acceptance criteria. If corrective action is not taken or was not successful, the decision/outcome must be documented in the analytical record. The primary analyst has primary responsibility for taking corrective action when QA/QC criteria are not met. Secondary data reviewers must verify that appropriate action was taken and/or that results reported with QC failure are properly qualified.

Corrective action is also required when carryover is suspected and when results are over range.

Samples analyzed after a high concentration sample must be checked for carryover and reanalyzed if carryover is suspected. Carryover is usually indicated by low concentration detects of the analyte in successive samples analyzed after the high concentration sample.

Sample results at concentrations above the upper limit of quantitation must be diluted and reanalyzed. The result in the diluted samples should be within the upper half of the calibration range. Results less than the mid-range of the calibration indicate the sample was over diluted and analysis should be repeated with a lower level of dilution. If dilution is not performed, any

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result reported above the upper range is considered a qualitative measurement and must be qualified as an estimated value.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

13.1 Pace proactively seeks ways to minimize waste generated during our work processes. Some examples of pollution prevention include but are not limited to reduced solvent extraction, solvent capture, use of reusable cycletainers for solvent management, and real-time purchasing.

13.2 The EPA requires that laboratory waste management practice to be conducted consistent with all applicable federal and state laws and regulations. Excess reagents, samples and method process wastes must be characterized and disposed of in an acceptable manner in accordance with Pace's SOP *Waste Handling and Management* (most current revision or replacement).

14.0 MODIFICATIONS

14.1 A modification is a change to a reference test method made by the laboratory. For example, changes in stoichiometry, technology, quantitation ions, reagent or solvent volumes, reducing digestion or extraction times, instrument runtimes, etc. are all examples of modifications. Refer to Pace ENV corporate SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification* for the conditions under which the procedures in test method SOPs may be modified and for the procedure and document requirements.

14.2 Not applicable to this SOP.

15.0 RESPONSIBILITIES

15.1 Pace ENV employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement in their training file for this version of the SOP. The employee is responsible for following the procedures in this SOP and handling temporary departures from this SOP in accordance with Pace's policy for temporary departure.

15.2 Pace supervisors/managers are responsible for training employees on the procedures in this SOP and monitoring the implementation of this SOP in their work area.

16.0 ATTACHMENTS

16.1 Attachment I: Routine Alarms and Troubleshooting

17.0 REFERENCES

17.1 Pace Quality Assurance Manual- most current version.

17.2 National Environmental Laboratory Accreditation Conference (NELAC), Chapter 5, "Quality Systems"- most current version.

17.3 The NELAC Institute (TNI); Volume 1, Module 2, "Quality Systems"- most current version.

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17.4 ASTM Method D 5468-95, Standard Test Method for Gross Calorific and Ash Value of Waste Materials.

18.0 REVISION HISTORY

18.1 This Version: ENV-SOP-ASHE-0015-Rev.03

Section	Description of Change
3.5 and 9.1.41	Removed Reference to Cellulose Tape
3.13	Subordinated Steps following this.
7.1	Clarified crucible composition
8.1	Adjusted formatting
9.1.30-35	Removed calibration and calibration check that did not follow current practice.
9.3.1	Added statement that calibration verification must be run every 10 runs.
11.1	Added QC table
Attachment I	Removed Attachment I: Running Volatile Samples

18.2 This document supersedes the following document(s):

Document Number	Title	Version
S-ASV-I-066	Determination of BTU in Waste Materials	00
S-ASV-I-066	Determination of BTU in Waste Materials	01
S-ASV-I-066	Determination of BTU in Waste Materials	02
S-ASV-I-066	Determination of BTU in Waste Materials	03
S-ASV-I-066	Determination of BTU in Waste Materials	04
ENV-SOP-ASHE-0015	Determination of BTU in Waste Materials	00
ENV-SOP-ASHE-0015	Determination of BTU in Waste Materials	01
ENV-SOP-ASHE-0015	Determination of BTU in Waste Materials	02

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Pace Analytical®

TEST METHOD STANDARD OPERATING PROCEDURE

TITLE:	DETERMINATION OF CALORIFIC VALUE (BTU/LB) OF WASTE MATERIALS USING A PARR 6300/6400 OXYGEN BOMB CALORIMETER
TEST METHOD	ASTM Method D 5468-95, Standard Test Method for Gross Calorific and Ash Value of Waste Materials.
ISSUER:	Pace ENV – Asheville– ASHE

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Attachment I

Routine Alarms and Troubleshooting

1. Temperature Rise must be 7.5-11°C for 6300 and 5.1-8.5°C for 6400

2. If the Temperature Rise exceeds 8.5 the instrument software will give an "Exceeds Temperature Rise" error. The analysis is to be repeated using a smaller sample weight or if the sample was spiked, use a smaller spike weight.

3. For analyzing glass: crush the glass into small pieces. Weigh up around 0.7g of glass and spike with 0.6g mineral oil. NOTE: a negative value will always print for the analysis of glass.

4. Never let your spike weight be greater than your sample weight. This is an invalid result and analysis should be repeated.

5. For samples that ran >5% sulfur, a thermo chemical correction must be entered before analyzing the Btu/lb value. Proceed with the following steps to do so:

- At the main menu, press "Thermo Chemical Corrections" and "Enter".
- Highlight "Fixed Sulfur Correction" (item #6) found under the "Determination" section and press "Skip".
- Type in the percent sulfur in the sample and press "Enter".
- Once the Btu/lb analysis has finished, the sulfur correction factor is to be changed back to zero.

Troubleshooting

1.Firing pin may be out of place

"Misfire-Fuse Intact" – Thread burned without igniting sample. Replace thread, being careful not to touch the sides of the crucible.

- Thread was wet, replace.
- Fuse wire burned into, replace.

2. "Refill Bomb Rinse Tank" – The rinse tank needs refilling.

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3. "Lid Failed to Lock" – Press lid firmly. If this doesn't work, you probably have a bad sensor.

4. "Temperature Rise Exceeds Limit" – The temperature rise was exceeded. Repeat analysis using a small amount of sample. If sample was spiked, use a smaller amount of spike.

5. "Bomb Firing Count Exceeds Limit" – The calorimeter count has reached its limit of 500 counts. Do routine maintenance. Once this has been done, refer to Section for testing bomb firing counter.

6. "02 Low Pressure" – Check pressure gauge, if below 450 psi, change cylinder. If pressure on the gauge is adequate, check the piston in the bottom of the bomb bucket to see that the o-ring is not worn. Replace if needed.

7. "Open Fuse" – Check and/or replace the bomb heating wire. Check the "water diffuser screw," tighten if needed.

Filling Rinse Tank:

1. The instrument will give you a "Refill Bomb Rinse Tank" alarm when the tank needs refilling. 750 washings is the limit.

2. Turn off nitrogen supply pressure (both valves) behind the hood marked "1".

3. Open cabinet door. Set rinse tank outside cabinet.

4. Open pressure relief valve.

5. Once pressure has bled off, open the lid to the rinse tank

6. Using only deionized water. Fill the rinse tank as close to the top without running over.

7. Replace lid. Close pressure relief valve.

8. Set rinse tank back in the cabinet.

9. Turn on nitrogen supply pressure

10. From the instrument software go to "Operating Controls" and "Enter"

11. Arrow to "Bomb rinse tank Controls" (#4). "Enter".

12. Arrow to "Reset rinse tank Counter" (#3). "Enter".

13. This will reset the counter to 750 washings.

14. Press "Escape" to get back to "Calorimeter Operation".

Reset Bomb Firing Counter:

1. Go to "Main Menu". "Enter"

2. Go to "Calibration data and Controls". "Enter".

3. Go to "Bomb Service Interval"- this is to be reading 500. NOTE: An entry will not have to be made at this point unless the number on the display is reading something other than 500. If so, change the display reading to 500 and continue with next step

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4. Use arrow keys to select either bomb #1 or bomb # 2 (depending on the instrument being used.) and “Enter”.

5. Arrow to “Bomb Firing Count”. Enter “0”. This resets your counter.

6. Escape back to Main menu.

Determination of Selected Perfluorinated Alkyl Substances by Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry Isotope Dilution (LC/MS/MS)

References: EPA Method 537.1, Version 2, March 2020, EPA Document #: EPA/600/R-20/006

EPA Method 533, November 2019, EPA Document #: 815-B-19-020

ISO 25101, First Edition, March 2009, Reference #: ISO 25101:2009(E)

Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.4, 2021

1. Scope and Application

Matrices: Drinking water, Non-potable Water, Tissues, Biosolids and Soil Matrices
(Drinking water is applicable for specific state regulatory requirements for this method)
Definitions: Refer to Alpha Analytical Quality Manual.

1.1. This is a liquid chromatography/tandem mass spectrometry (LC/MS/MS) method for the determination of selected perfluorinated alkyl substances (PFAS) in Non-Drinking Water and soil Matrices. Accuracy and precision data have been generated in reagent water, finished ground and surface waters, and soils for the compounds listed in Table 1.

1.2. The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

1.3. This method is restricted to use by or under the supervision of analysts experienced in the operation of the LC/MS/MS and in the interpretation of LC/MS/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of the Method

2.1. A 250-mL water sample is fortified with extracted internal standards (EIS) and passed through a solid phase extraction (WAX) cartridge containing a mixed mode, Weak Anion Exchange, reversed phase, water-wettable polymer to extract the method analytes and isotopically labeled compounds. The compounds are eluted from the solid phase in two fractions with methanol followed by a small amount of 2% ammonium hydroxide in methanol solution. The extract is concentrated with nitrogen in a heated water bath and then adjusted to a 1-mL volume with 80:20% (vol/vol) methanol:water.

2.2. A 0.5 to 4 gram soil, solid, tissue, or biosolid sample is fortified with extracted internal standards (EIS), diluted in methanol, and agitated rigorously. An aliquot of the methanol is passed across an SPE-based clean-up cartridge and the eluate is collected. The extract is concentrated with

nitrogen in a heated water bath and then adjusted to a 1-mL volume with 80:20% (vol/vol) methanol:water.

2.3. A sample extract is injected into an LC equipped with a C18 column that is interfaced with an MS/MS. The analytes are separated and identified by comparing the acquired mass spectra and retention times to reference spectra and retention times for calibration standards acquired under identical LC/MS/MS conditions. The concentration of each analyte is determined by using the isotope dilution technique. Extracted Internal Standards (EIS) analytes are used to monitor the extraction efficiency of the method analytes.

2.4. Method Modifications from Reference

2.4.1. None.

Table 1

Parameter	Acronym	CAS
PERFLUOROALKYL ETHER CARBOXYLIC ACIDS (PFECAs)		
Tetrafluoro-2-(heptafluoropropoxy)propanoic acid	HFPO-DA	13252-13-6
4,8-dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4
PERFLUOROALKYLCARBOXILIC ACIDS (PFCAs)		
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnA	2058-94-8
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluorotetradecanoic acid	PFTA	376-06-7
Perfluorohexadecanoic acid	PFHxDA	67905-19-5
Perfluorooctadecanoic acid	PFODA	16517-11-6
PERFLUOROALKYL SULFONIC ACIDS (PFASs)		
Perfluoropropanesulfonic acid	PFPrS	423-41-6
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoS	79780-39-5

Table 1 Cont.

Parameter	Acronym	CAS
CHLORO-PERFLUOROALKYLSULFONATE		
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid	9Cl-PF3ONS	756426-58-1
PERFLUOROOCTANESULFONAMIDES (FOSAs)		
Perfluorooctanesulfonamide	PFOSA	754-91-6
N-methylperfluoro-1-octanesulfonamide	NMeFOSA	31506-32-8
N-ethylperfluoro-1-octanesulfonamide	NEtFOSA	4151-50-2
TELOMER SULFONIC ACIDS		
1H,1H,2H,2H-perfluorohexanesulfonic acid (4:2)	4:2FTS	757124-72-4
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2)	6:2FTS	27619-97-2
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2)	8:2FTS	39108-34-4
1H,1H,2H,2H-perfluorododecanesulfonic acid (10:2)	10:2FTS	120226-60-0
PERFLUOROOCTANESULFONAMIDOACETIC ACIDS		
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
NATIVE PERFLUOROOCTANESULFONAMIDOETHANOLS (FOSEs)		
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	NMeFOSE	24448-09-7
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	NEtFOSE	1691-99-2
PERFLUOROETHER AND POLYETHER CARBOXYLIC ACIDS		
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6

3. Reporting Limits

3.1. The reporting limit for PFAS's is 2 ng/L for aqueous samples (20 ng/L for HFPO-DA) and 0.25 - 1 ng/g (2.5 ng/g for HFPO-DA) for soil samples.

4. Interferences

4.1. PFAS standards, extracts and samples should not come into contact with any glass containers or pipettes as these analytes can potentially adsorb to glass surfaces. PFAS analyte and EIS standards commercially purchased in glass ampoules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene containers.

4.2. Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that can lead to discrete artifacts and/or elevated baselines in the chromatograms. The method analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, SPE sample transfer lines, etc. All items such as these must be demonstrated to be free from interferences (less than 1/3 the RL for each method analyte) under the conditions of the analysis by analyzing laboratory reagent blanks whenever a new vendor or reagent is brought online as described in Section 9.1,

and by continuous evaluation of method blanks and/or instrument blanks. **Subtracting blank values from sample results is not permitted.**

4.3. Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the water. Humic and/or fulvic material can be co-extracted during SPE and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent. Total organic carbon (TOC) is a good indicator of humic content of the sample.

4.4. SPE cartridges can be a source of interferences. The analysis of field and laboratory reagent blanks can provide important information regarding the presence or absence of such interferences. Brands and lots of SPE devices should be tested to ensure that contamination does not preclude analyte identification and quantitation.

5. Health and Safety

5.1. The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

5.2. All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.3. PFOA has been described as “likely to be carcinogenic to humans.” Pure standard materials and stock standard solutions of these method analytes should be handled with suitable protection to skin and eyes, and care should be taken not to breathe the vapors or ingest the materials.

6. Sample Collection, Preservation, Shipping and Handling

6.1. Sample Collection for Aqueous Samples

6.1.1. Samples must be collected in two (2) 250-mL high density polyethylene (HDPE) container with an unlined plastic screw cap.

6.1.2. The sample handler must wash their hands before sampling and wear nitrile gloves while filling and sealing the sample bottles. PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

6.1.3. Open the tap and allow the system to flush until the water temperature has stabilized (approximately 3 to 5 min). Collect samples from the flowing system.

6.1.4. Fill sample bottles. Samples do not need to be collected headspace free.

6.1.5. After collecting the sample and cap the bottle. Keep the sample sealed from time of collection until extraction.

6.1.6. Field Reagent Blank (FRB)

6.1.6.1. An FRB must be handled along with each sample set. The sample set is composed of samples collected from the same sample site and at the same time. At the laboratory, fill the field blank sample bottle with reagent water and preservatives, seal, and ship to the sampling site along with the sample bottles. For each FRB shipped, an empty sample bottle (no preservatives) must also be shipped. At the sampling site, the sampler must open the shipped FRB and pour the reagent water into the empty shipped sample bottle, seal and label this bottle as the FRB. The FRB is shipped back to the laboratory along with the samples and analyzed to ensure that PFAS's were not introduced into the sample during sample collection/handling.

6.1.6.2. The reagent water used for the FRBs must be initially analyzed for method analytes as a MB and must meet the MB criteria in Section 9.1.1 prior to use. This requirement will ensure samples are not being discarded due to contaminated reagent water rather than contamination during sampling.

6.2. Sample Collection for Soil and Sediment samples

6.2.1. Grab samples are collected in polypropylene containers. Sample containers and contact surfaces containing PTFE shall be avoided.

6.3. Sample Preservation

6.3.1. Not applicable.

6.4. Sample Shipping

6.4.1. Samples must be chilled during shipment and must not exceed 10 °C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 10 °C when the samples are received at the laboratory. Samples stored in the lab must be held at or below 6 °C until extraction but should not be frozen.

6.4.2. **NOTE:** Samples that are significantly above 10° C, at the time of collection, may need to be iced or refrigerated for a period of time, in order to chill them prior to shipping. This will allow them to be shipped with sufficient ice to meet the above requirements.

6.5. Sample Handling

6.5.1. Holding Times

6.5.1.1. Aqueous samples should be extracted as soon as possible but must be extracted within 28 days and stored at 0° - 6° C in the dark. Solid and tissue matrices should be extracted within 90 days and stored at 0° - 6° C in the dark. Extracts are stored at <10° C and analyzed within 90 days after extraction.

6.5.1.2. The holding time for extraction for all matrices reporting to the State of Maine is 14 days stored at 0-6°C or -20°C; analytical holding time is 28 days for extracts stored at 0-4°C.

7. Equipment and Supplies

7.1. SAMPLE CONTAINERS – 250-mL high density polyethylene (HDPE) bottles fitted with unlined screw caps. Sample bottles must be discarded after use.

7.2. SAMPLE JARS – 8-ounce wide mouth high density polyethylene (HDPE) bottles fitted with unlined screw caps. Sample bottles must be discarded after use.

7.3. POLYPROPYLENE BOTTLES – 4-mL narrow-mouth polypropylene bottles.

7.4. CENTRIFUGE TUBES – 50-mL conical polypropylene tubes with polypropylene screw caps for storing standard solutions and for collection of the extracts.

7.5. AUTOSAMPLER VIALS – Polypropylene 0.7-mL autosampler vials with polypropylene caps.

7.6. NOTE: Polypropylene vials and caps are necessary to prevent contamination of the sample from PTFE coated septa. However, polypropylene caps do not reseal, so evaporation occurs after injection. Thus, multiple injections from the same vial are not possible.

7.7. POLYPROPYLENE GRADUATED CYLINDERS – Suggested sizes include 25, 50, 100 and 1000-mL cylinders.

7.8. Auto Pipets – Suggested sizes include 5, 10, 25, 50, 100, 250, 500, 1000, 5000 and 10,000-µls.

7.9. PLASTIC PIPETS – Polypropylene or polyethylene disposable pipets.

7.10. ANALYTICAL BALANCE – Capable of weighing to the nearest 0.0001 g.

7.11. ANALYTICAL BALANCE – Capable of weighing to the nearest 0.1 g.

7.12. SOLID PHASE EXTRACTION (SPE) APPARATUS FOR USING CARTRIDGES

7.13. SPE CARTRIDGES – 0.5 g SPE cartridges containing a reverse phase copolymer characterized by a weak anion exchanger (WAX) sorbent phase.

7.14. VACUUM EXTRACTION MANIFOLD – A manual vacuum manifold with large volume sampler for cartridge extractions, or an automatic/robotic sample preparation system designed for use with SPE cartridges, may be used if all QC requirements discussed in Section 9 are met. Extraction and/or elution steps may not be changed or omitted to accommodate the use of an automated system. Care must be taken with automated SPE systems to ensure the PTFE commonly used in these systems does not contribute to unacceptable analyte concentrations in the MB (Sect. 9.1.1).

7.15. SAMPLE DELIVERY SYSTEM – Use of a polypropylene transfer tube system, which transfers the sample directly from the sample container to the SPE cartridge, is recommended, but not mandatory. Standard extraction manifolds come equipped with PTFE transfer tube systems. These can be replaced with 1/8" O.D. x 1/16" I.D. polypropylene or polyethylene tubing cut to an appropriate length to ensure no sample contamination from the sample transfer lines. Other types of non-PTFE tubing may be used provided it meets the MB (Sect. 9.1.1) and LCS (Sect. 9.2) QC requirements.

7.16. Extract Clean-up Cartridge – 250 mg 6mL SPE Cartridge containing graphitized polymer carbon.

7.17. EXTRACT CONCENTRATION SYSTEM – Extracts are concentrated by evaporation with nitrogen using a water bath set no higher than 65 °C.

7.18. LABORATORY OR ASPIRATOR VACUUM SYSTEM – Sufficient capacity to maintain a vacuum of approximately 10 to 15 inches of mercury for extraction cartridges.

7.19. LIQUID CHROMATOGRAPHY (LC)/TANDEM MASS SPECTROMETER (MS/MS) WITH DATA SYSTEM

7.20. LC SYSTEM – Instrument capable of reproducibly injecting up to 10-µL aliquots and performing binary linear gradients at a constant flow rate near the flow rate used for development of this method (0.4 mL/min). The LC must be capable of pumping the water/methanol mobile phase

without the use of a degasser which pulls vacuum on the mobile phase bottle (other types of degassers are acceptable). Degassers which pull vacuum on the mobile phase bottle will volatilize the ammonium acetate mobile phase causing the analyte peaks to shift to earlier retention times over the course of the analysis batch. The usage of a column heater is optional.

7.21. LC/TANDEM MASS SPECTROMETER – The LC/MS/MS must be capable of negative ion electrospray ionization (ESI) near the suggested LC flow rate of 0.4 mL/min. The system must be capable of performing MS/MS to produce unique product ions for the method analytes within specified retention time segments. A minimum of 10 scans across the chromatographic peak is required to ensure adequate precision.

7.22. DATA SYSTEM – An interfaced data system is required to acquire, store, reduce, and output mass spectral data. The computer software should have the capability of processing stored LC/MS/MS data by recognizing an LC peak within any given retention time window. The software must allow integration of the ion abundance of any specific ion within specified time or scan number limits. The software must be able to calculate relative response factors, construct linear regressions or quadratic calibration curves, and calculate analyte concentrations.

7.23. ANALYTICAL COLUMN – An LC BEH C18 column (2.1 x 50 mm) packed with 1.7 µm dp C18 solid phase particles was used. Any column that provides adequate resolution, peak shape, capacity, accuracy, and precision (Sect. 9) may be used.

8. Reagents and Standards

8.1. GASES, REAGENTS, AND SOLVENTS – Reagent grade or better chemicals must be used.

8.1.1. REAGENT WATER – Purified water which does not contain any measurable quantities of any method analytes or interfering compounds greater than 1/3 the RL for each method analyte of interest. Prior to daily use, at least 3 L of reagent water should be flushed from the purification system to rinse out any build-up of analytes in the system's tubing.

8.1.2. METHANOL (CH_3OH, CAS#: 67-56-1) – High purity, demonstrated to be free of analytes and interferences.

8.1.3. AMMONIUM ACETATE ($NH4C2H3O2$, CAS#: 631-61-8) – High purity, demonstrated to be free of analytes and interferences.

8.1.4. ACETIC ACID (H_3CCOOH, CAS#: 64-19-7) - High purity, demonstrated to be free of analytes and interferences.

8.1.5. 1M AMMONIUM ACETATE/REAGENT WATER – High purity, demonstrated to be free of analytes and interferences.

8.1.6. 2mM AMMONIUM ACETATE/METHANOL:WATER (5:95) – To prepare, mix 2 mL of 1M AMMONIUM ACETATE,1 mL ACETIC ACID and 50 mL METHANOL into I Liter of REAGENT WATER.

8.1.7. Methanol/Water (80:20) – To prepare a 1 Liter bottle, mix 200 mL of REAGENT WATER with 800 mL of METHANOL.

8.1.8. AMMONIUM HYDROXIDE (NH_3, CAS#: 1336-21-6) – High purity, demonstrated to be free of analytes and interferences.

8.1.9. Sodium Acetate ($NaOOCCH_3$, CAS#: 127-09-3) – High purity, demonstrated to be free of analytes and interferences.

8.1.10. 25 mM Sodium Acetate Buffer – To prepare 250 mLs, dissolve .625 grams of sodium acetate into 100 mLs of reagent water. Add 4 mLs Acetic Acid and adjust the final volume to 250 mLs with reagent water.

8.1.11. NITROGEN – Used for the following purposes: Nitrogen aids in aerosol generation of the ESI liquid spray and is used as collision gas in some MS/MS instruments. The nitrogen used should meet or exceed instrument manufacturer's specifications. In addition, Nitrogen is used to concentrate sample extracts (Ultra High Purity or equivalent).

8.1.12. ARGON – Used as collision gas in MS/MS instruments. Argon should meet or exceed instrument manufacturer's specifications. Nitrogen gas may be used as the collision gas provided sufficient sensitivity (product ion formation) is achieved.

8.2. STANDARD SOLUTIONS – When a compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. PFAS analyte and IS standards commercially purchased in glass ampoules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene containers. Standards for sample fortification generally should be prepared in the smallest volume that can be accurately measured to minimize the addition of excess organic solvent to aqueous samples.

NOTE: Stock standards and diluted stock standards are stored at ≤4 °C.

8.2.1. ISOTOPE DILUTION Extracted Internal Standard (ID EIS) STOCK SOLUTIONS - ID EIS stock standard solutions are stable for at least 6 months when stored at 4 °C. The stock solution is purchased at a concentration of 1000 ng/mL.

8.2.2. ISOTOPE DILUTION Extracted Internal Standard PRIMARY DILUTION STANDARD (ID EIS PDS) – Prepare the ID EIS PDS at a concentration of 500 ng/mL. The ID PDS is prepared in methanol. The ID PDS is stable for 1 year when stored at ≤4 °C (table 2a).

Table 2a

Isotope Labeled Standard	Conc. of EIS Stock (ng/mL)	Vol. of EIS Stock (mL)	Final Vol. of EIS PDS (mL)	Final Conc. of EIS PDS (ng/mL)
M4PFBA	1000	1.0	2.0	500
M5PFPeA	1000	1.0	2.0	500
M5PFHxA	1000	1.0	2.0	500
M4PFHpA	1000	1.0	2.0	500
M8PFOA	1000	1.0	2.0	500
M9PFNA	1000	1.0	2.0	500
M6PFDA	1000	1.0	2.0	500
M7PFUdA	1000	1.0	2.0	500
MPFDoA	1000	1.0	2.0	500
M2PFTeDA	1000	1.0	2.0	500
M2PFHxDA	50,000	.02	2.0	500
M8FOSA	1000	1.0	2.0	500
d3-N-MeFOSAA	1000	1.0	2.0	500
d5-N-EtFOSAA	1000	1.0	2.0	500
M3PFBS	929	1.0	2.0	464.5
M3PFHxS	946	1.0	2.0	473
M8PFOS	957	1.0	2.0	478.5
M2-4:2FTS	935	1.0	2.0	467.5
M2-6:2FTS	949	1.0	2.0	474.5
M2-8:2FTS	958	1.0	2.0	479
M2,D4-10:2FTS	50,000	.04	2.0	1000
M3HFPO-DA	50,000	.4	2.0	10,000

Table 2b

Isotope Labeled Standard	Conc. of EIS Stock (ng/mL)	Vol. of EIS Stock (mL)	Final Vol. of EIS PDS (mL)	Final Conc. of EIS PDS (ng/mL)
d3-N-MeFOSA	50,000	.2	2.0	5000
d5-N-EtFOSA	50,000	.2	2.0	5000
d7-N-MeFOSE	50,000	.2	2.0	5000
d9-N-EtFOSE	50,000	.2	2.0	5000

8.2.3. ANALYTE STOCK STANDARD SOLUTION – Analyte stock standards are stable for 1 year when stored at 4 °C. When using these stock standards to prepare a PDS, care must be taken to ensure that these standards are at room temperature and adequately vortexed.

8.2.4. ANALYTE PRIMARY SPIKING STANDARD – Prepare the spiking standard at a concentration of 500 ng/mL in methanol. The spiking standard is stable for at least two months when stored in polypropylene centrifuge tubes at room temperature.

8.2.5. Analyte Secondary Spiking Stock (SS): Prepare the spiking solution of additional add on components for project specific requirements only.

Table 3: Purchased PFAC-30PAR

Analyte	Conc. of Stock (ng/mL)	Vol. of Stock (mL)	Final Vol. of PDS (mL)	Final Conc. of PDS (ng/mL)
PFBA	1000	1	2	500
PFPeA	1000	1	2	500
PFHxA	1000	1	2	500
PFHpA	1000	1	2	500
PFOA	1000	1	2	500
PFNA	1000	1	2	500
PFDA	1000	1	2	500
PFUnA	1000	1	2	500
PFDoA	1000	1	2	500
PFTrDA	1000	1	2	500
PFTA	1000	1	2	500
FOSA	1000	1	2	500
Br-NMeFOSAA	240	1	2	120
L-NMeFOSAA	760	1	2	380
Br-NEtFOSAA	225	1	2	112.5
L-NEtFOSAA	775	1	2	387.5
L-PFBS	887	1	2	443.5
L-PFPeS	941	1	2	470.5
L-PFHxSK	741	1	2	370.5
Br-PFHxSK	173	1	2	86.5
L-PFHpS	953	1	2	476.5
L-PFOSK	732	1	2	366
Br-PFOSK	196	1	2	98
L-PFNS	962	1	2	481
L-PFDS	965	1	2	482.5
4:2FTS	937	1	2	468.5
6:2FTS	951	1	2	475.5
8:2FTS	960	1	2	480
9ClPF3ONS	933	1	2	466.5
11ClPF3OUdS	943	1	2	471.5
ADONA	945	1	2	472.5
HFPO-DA	1000	1	2	500

Table 4: Secondary Stock (SS)

Analyte	Conc. of Stock (ng/mL)	Vol. of Stock (mL)	Final Vol. of SS (mL)	Final Conc. of SS(ng/mL)
PFHxDA	50,000	0.4	10	2000
PFODA	50,000	0.4	10	2000
HFPO-DA	50,000	3.9	10	19,500
10:2-FTS	48,300	0.4	10	1932
PFDoS	48,400	0.4	10	1936
PFPrS	45,800	0.4	10	1832
PFMPA	50,000	0.4	10	2000
PFMBA	50,000	0.4	10	2000
PFEESA	44,500	0.4	10	1780
NFDHA	50,000	0.4	10	2000
NMeFOSA	50,000	0.4	10	2000
NMeFOSE	50,000	0.4	10	2000
NEtFOSA	50,000	0.4	10	2000
NEtFOSE	50,000	0.4	10	2000

Table 5: Final PDS solution

Stock	Conc of Stock	Vol of Stock (mL)	Final Vol of PDS (mL)	Final conc (ng/mL)
PFAC30PAR	**Varies (see Table 3)**	**2**	**4**	**500 (varies)**
Secondary Stock	**Varies (see Table 4)**	**1**	**4**	**500 (varies)**

8.2.6. LOW, MEDIUM, AND HIGH LEVEL LCS – The LCS's will be prepared at the following concentrations and rotated per batch; 2 ng/L, 40 ng/L, 500 ng/l for drinking water. The analyte PDS contains all the method analytes of interest at various concentrations in methanol. The analyte PDS has been shown to be stable for six months when stored at ≤ 4 °C.

8.2.7. Isotope Dilution Labeled Recovery Stock Solutions (ID REC) – ID REC Stock solutions are stable for at least 1 year when stored at 4 °C. The stock solution is purchased at a concentration of 2000 ng/mL.

8.2.8. Isotope Dilution Labeled Recovery Primary Dilution Standard (ID REC PDS) - Prepare the ID REC PDS at a concentration of 500 ng/mL. The ID REC PDS is prepared in methanol. The ID REC PDS is stable for at least 1 year when stored in polypropylene centrifuge tubes at ≤4 °C.

Table 5

Analyte	Conc. of REC Stock (ng/mL)	Vol. of REC Stock (mL)	Final Vol. of REC PDS (mL)	Final Conc. of REC PDS (ng/mL)
M2PFOA	2000	1	4	500
M2PFDA	2000	1	4	500
M3PFBA	2000	1	4	500
M4PFOS	2000	1	4	500

8.2.9. CALIBRATION STANDARDS (CAL)

Current Concentrations (ng/mL): 0.25 (optional), 0.5, 1.0, 5.0, 10.0, 25, 50.0, 100, 250.

Prepare the CAL standards over the concentration range of interest from dilutions of the analyte PDS in methanol containing 20% reagent water. 20 µl of the EIS PDS and REC PDS are added to the CAL standards to give a constant concentration of 10 ng/mL. The lowest concentration CAL standard must be at or below the RL (2 ng/L), which may depend on system sensitivity. The CAL standards may also be used as CCVs (Sect. 9.8).

Table 6

Calibration Standard Concentration	Final Aqueous Cal STD Concentration	Final Soil Cal STD Concentration	30 Compound Stock Added (uL)	Individual Analyte Stocks Added (uL)	500 ng/mL Dilution Added (uL)	Final Volume in MeOH/H2O (80:20)
.5 ng/mL	2 ng/L	.25 ng/g	6.25		25	25 mLs
1 ng/mL	4 ng/L	.5 ng/g	5		20	10 mLs
5 ng/mL	20 ng/L	1 ng/g	25		100	10 mLs
10 ng/mL	40 ng/L	5 ng/g	125	5		25 mLs
50 ng/mL	200 ng/L	25 ng/g	250	10		10 mLs
125 ng/mL	500 ng/L	62.5 ng/g	625	25		10 mLs
150 ng/mL	600 ng/L	75 ng/g	750	30		10 mLs
250 ng/mL	1000 ng/L	125 ng/g	625			5 mLs
500 ng/mL	2000 ng/L	250 ng/g	1250			5 mLs

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1. Blanks

9.1.1. METHOD BLANK (MB) - A Method Blank (MB) is required with each extraction batch to confirm that potential background contaminants are not interfering with the identification or quantitation of method analytes. Prep and analyze a MB for every 20 samples. If the MB produces a peak within the retention time window of any analyte that would prevent the determination of that analyte, determine the source of contamination, and eliminate the interference before processing samples. Background contamination must be reduced to an acceptable level before proceeding. Background from method analytes or other contaminants that interfere with the measurement of method analytes must be below the RL. If the method analytes are detected in the MB at concentrations equal to or greater than this level, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch. Because background contamination is a significant problem for several method analytes, it is highly recommended that the analyst maintains a historical record of MB data.

9.1.1.1. Corrective Action: Samples should not be analyzed until an acceptable method blank analysis is obtained. The results are qualified with a “B” flag for any associated sample concentrations that are less than 10x the blank concentration for the analyte. Sample results are qualified with a “B” flag for analytes observed in the blank > RL. Exceptions may be made if the samples associated with the out-of-control method blank

are non-detect for the compound of interest, or if sample concentrations are greater than 10x the blank levels. In such cases when the sample results are accepted, the client is notified in a project narrative associated with the sample results.

9.1.2. **FIELD REAGENT BLANK (FRB)** - The purpose of the FRB is to ensure that PFAS's measured in the Field Samples were not inadvertently introduced into the sample during sample collection/handling. Analysis of the FRB is performed on a project specific basis. The FRB is processed, extracted, and analyzed in exactly the same manner as a Field Sample.

9.2. Laboratory Control Sample (LCS) and Laboratory Control Sample Duplicates (LCSD)

9.2.1. An LCS is required with each extraction batch. The fortified concentration of the LCS may be rotated between low, medium, and high concentrations from batch to batch. Default limits of 50-150% (60 – 140% for New Jersey programs) of the true value may be used for analytes until sufficient replicates have been analyzed to generate proper control limits. Calculate the percent recovery (%*R*) for each analyte using the equation:

$$\%R = \frac{A \times 100}{B}$$

Where:

A = measured concentration in the fortified sample
B =fortification concentration.

9.2.2. Where applicable, in the absence of additional sample volume required to perform matrix specific QC, LCSD's are to be extracted and analyzed. The concentration and analyte recovery criteria for the LCSD must be the same as the batch LCS. The RSD's must fall within ≤30% of the true value for medium and high-level replicates, and ≤50% for low level replicates. Calculate the relative percent difference (RPD) for duplicate MSs (MS and MSD) using the equation:

$$RPD = \frac{|LCS - LCSD|}{(LCS + LCSD) / 2} \times 100$$

9.2.3. If the LCS and or LCSD results do not meet these criteria for method analytes, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch.

9.2.4. Corrective Action: All compounds should be recovered within the specified limits. If the LCS recovery is still out of control, re-extract and/or re-analyze the LCS and all associated samples. Samples cannot be reported until an acceptable LCS is obtained. Exceptions may be made in some cases for documented difficult/poor performing compounds such as PFODA, FOSA, and NFDHA. An explanation of any out-of-control LCS recovery must be included in the project narrative to the client.

9.3. Labeled Recovery Standards (REC)

9.3.1. The analyst must monitor the peak areas of the REC(s) in all injections during each analysis day.

9.4. Extracted Internal Standards (EIS)

9.4.1. The EIS standard is fortified into all samples, CCVs, MBs, LCSs, MSs, MSDs, FD, and FRB prior to extraction. It is also added to the CAL standards. The EIS is a means of assessing method performance from extraction to final chromatographic measurement. Calculate the recovery (%R) for the EIS using the following equation:

$$\%R = (A / B) \times 100$$

Where:

A = calculated EIS concentration for the QC or Field Sample

B = fortified concentration of the EIS.

9.4.2. EIS recoveries are evaluated according to their matrix specific control limits in the LIMS. Any samples with EIS recoveries < 5% (< 10 % for New York) must be re-extracted. Any data associated with EIS recoveries <2% cannot be reported and the project manager needs to be notified. A low or high percent recovery for a sample, blank, or CCV does not require discarding the analytical data, but it may indicate a potential problem with future analytical data. When EIS recovery from a sample, blank, or CCV are outside control limits, check 1) calculations to locate possible errors, 2) standard solutions for degradation, 3) contamination, and 4) instrument performance. For CCVs and QC elements spiked with all target analytes, if the recovery of the corresponding target analytes meet the acceptance criteria but the EIS is outside criteria, the data can be used. If the associated target analytes do not meet the acceptance criteria, the data must be reanalyzed.

9.5. Matrix Spike (MS)

9.5.1. Analysis of an MS is required in each extraction batch and is used to determine that the sample matrix does not adversely affect method accuracy. Assessment of method precision is accomplished by analysis of a Field Duplicate (FD) (Sect. 9.6); however, infrequent occurrence of method analytes would hinder this assessment. If the occurrence of method analytes in the samples is infrequent, or if historical trends are unavailable, a second MS, or MSD, must be prepared, extracted, and analyzed from a duplicate of the Field Sample. Extraction batches that contain MSDs will not require the extraction of a field sample duplicate. If a variety of different sample matrices are analyzed regularly, for example, drinking water from groundwater and surface water sources, method performance should be established for each. Over time, MS data should be documented by the laboratory for all routine sample sources.

9.5.2. Within each extraction batch, a minimum of one Field Sample is fortified as an MS for every 20 Field Samples analyzed. The MS is prepared by spiking a sample with an appropriate amount of the Analyte Stock Standard (Sect. 8.2.3). Use historical data and rotate through the low, mid and high concentrations when selecting a fortifying concentration. Calculate the percent recovery (%*R*) for each analyte using the equation:

$$\%R = \frac{(A - B)}{C} \times 100$$

Where:

A = measured concentration in the fortified sample
B = measured concentration in the unfortified sample
C = fortification concentration.

9.5.3. Analyte recoveries may exhibit matrix bias. For samples fortified at or above their native concentration, recoveries should range between 50-150%. If the accuracy of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the LCS, the recovery is judged to be matrix biased. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.6. Laboratory Duplicate

9.6.1. FIELD DUPLICATE OR LABORATORY FORTIFIED SAMPLE MATRIX DUPLICATE (FD or MSD) – Within each extraction batch (not to exceed 20 Field Samples), a minimum of one FD or MSD must be analyzed. Duplicates check the precision associated with sample collection, preservation, storage, and laboratory procedures. If method analytes are not routinely observed in Field Samples, an MSD should be analyzed rather than an FD.

9.6.1.1. Calculate the relative percent difference (*RPD*) for duplicate measurements (*FD1*

and *FD2*) using the equation:

$$RPD = \frac{|FD1 - FD2|}{(FD1 + FD2) / 2} \times 100$$

9.6.2. RPDs for FDs should be ≤30%. Greater variability may be observed when FDs have analyte concentrations that are within a factor of 2 of the RL. At these concentrations, FDs should have RPDs that are ≤50%. If the RPD of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the CCV, the recovery is judged to be matrix biased. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.6.3. If an MSD is analyzed instead of a FD, calculate the relative percent difference (RPD) for duplicate MSs (MS and MSD) using the equation:

$$RPD = \frac{|MS - MSD|}{(MS + MSD) / 2} \times 100$$

9.6.4. RPDs for duplicate MSs should be ≤30% for samples fortified at or above their native concentration. Greater variability may be observed when MSs are fortified at analyte concentrations that are within a factor of 2 of the RL. MSs fortified at these concentrations should have RPDs that are ≤50% for samples fortified at or above their native concentration. If the RPD of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the LCSD where applicable, the result is judged to be matrix biased.

9.7. Initial Calibration Verification (ICV)

9.7.1. After each ICAL, analyze a QCS sample from a source different from the source of the CAL standards. If a second vendor is not available, then a different lot of the standard should be used. The QCS should be prepared and analyzed just like a CCV. Acceptance criteria for the QCS are identical to the CCVs; the calculated amount for each analyte must be ± 30% of the expected value. If measured analyte concentrations are not of acceptable accuracy, check the entire analytical procedure to locate and correct the problem.

9.8. Continuing Calibration Verification (CCV)

9.9. CCV Standards are analyzed at the beginning of each analysis batch, after every 10 Field Samples, and at the end of the analysis batch. See Section 10.11 for concentration requirements and acceptance criteria.

9.10. Method-specific Quality Control Samples

9.10.1. None

9.11. Method Sequence

- CCV-LOW
- MB
- LCS
- LCSD (where applicable)
- MS
- Duplicate or MSD
- Field Samples (1-10)
- CCV-MID
- Field Samples (11-20)
- CCV-LOW

10. Procedure

10.1. Equipment Set-up

10.1.1. This procedure may be performed manually or in an automated mode using a robotic or automatic sample preparation device. If an automated system is used to prepare samples, follow the manufacturer's operating instructions, but all extraction and elution steps must be the same as in the manual procedure. Extraction and/or elution steps may not be changed or omitted to accommodate the use of an automated system. If an automated system is used, the MBs should be rotated among the ports to ensure that all the valves and tubing meet the MB requirements (Sect. 9.1).

10.1.2. Some of the PFAS's adsorb to surfaces, including polypropylene. Therefore, the aqueous sample bottles must be rinsed with the elution solvent (Sect 10.3.4) whether extractions are performed manually or by automation. The bottle rinse is passed through the cartridge to elute the method analytes and is then collected (Sect. 10.3.4).

10.1.3. NOTE: The SPE cartridges and sample bottles described in this section are designed as single use items and should be discarded after use. They may not be refurbished for reuse in subsequent analyses.

10.2. Sample Preparation and Extraction of Aqueous Samples

10.2.1. Samples are preserved, collected, and stored as presented in Section 6. The entire sample that is received must be sent through the SPE cartridge. In addition, the bottle must be solvent rinsed and this rinse must be sent through the SPE cartridge as well. The method blank (MB) and laboratory control sample (LCS) must be extracted in the same manner (i.e., must include the bottle solvent rinse). It should be noted that a water rinse alone is not sufficient. This does not apply to samples with high concentrations of PFAS that are prepared using serial dilution and not SPE.

10.2.2. Determine sample volume. Weigh all samples to the nearest 1g. If visible sediment is present, centrifuge and decant into a new 250mL HDPE bottle and record the weight of the new container.

NOTE: Some of the PFAS's adsorb to surfaces, thus the sample volume may NOT be transferred to a graduated cylinder for volume measurement.

10.2.3. The MB, LCS and FRB may be prepared by measuring 250 mL of reagent water with a polypropylene graduated cylinder or filling a 250-mL sample bottle to near the top.

10.2.4. Adjust the QC and sample pH to 3 by adding acetic acid in water dropwise.

10.2.5. Add 20 µL of the EIS PDS (Sect. 8.2.2) to each sample and QC, cap and invert to mix.

10.2.6. If the sample is an LCS, LCSD, MS, or MSD, add the necessary amount of analyte PDS (Sect. 8.2.4 and Sect 8.2.5). Cap and invert each sample to mix.

10.3. Cartridge SPE Procedure

10.3.1. CARTRIDGE CLEAN-UP AND CONDITIONING – DO NOT allow cartridge packing material to go dry during any of the conditioning steps. Rinse each cartridge with 3 X 5 mL of 2% ammonium hydroxide in methanol, followed by 5mLs of methanol. Next, rinse each cartridge with 5 mLs of the 25 mM acetate buffer, followed by 15 mL of reagent water, without allowing the water to drop below the top edge of the packing. If the cartridge goes dry during the conditioning phase, the conditioning must be started over. Add 4-5 mL of reagent water to

each cartridge, attach the sample transfer tubes (Sect. 7.11.3), turn on the vacuum, and begin adding sample to the cartridge.

10.3.2. SAMPLE EXTRACTON – Adjust the vacuum so that the approximate flow rate is approximately 4 mL/min. Do not allow the cartridge to go dry before all the sample has passed through.

10.3.3. SAMPLE BOTTLE AND CARTRIDGE RINSE – After the entire sample has passed through the cartridge, rinse the sample bottles with 4 mL reagent water followed by 4 mL 25 mM acetate buffer at pH 4 and draw the aliquot through the sample transfer tubes and the cartridges. Draw air or nitrogen through the cartridge for 5-10 min at high vacuum (10-15 in. Hg). NOTE: If empty plastic reservoirs are used in place of the sample transfer tubes to pass the samples through the cartridges, these reservoirs must be treated like the transfer tubes. After the entire sample has passed through the cartridge, the reservoirs must be rinsed to waste with reagent water.

10.3.4. SAMPLE BOTTLE AND CARTRIDGE ELUTION, Fraction 1 – Turn off and release the vacuum. Lift the extraction manifold top and insert a rack with collection tubes into the extraction tank to collect the extracts as they are eluted from the cartridges. Rinse the sample bottles with 12 mLs of methanol and draw the aliquot through the sample transfer tubes and cartridges. Use a low vacuum such that the solvent exits the cartridge in a dropwise fashion.

10.3.5. NOTE: Due to the possible volatility and suspect degradation of sulfonamides and sulfonamide ethanols when exposed to heat, a portion of the methanol fraction may be retained and analyzed independently with no evaporation for these analytes.

10.3.6. SAMPLE BOTTLE AND CARTRIDGE ELUTION, Fraction 2 In a separate collection vial, rinse the sample bottles with 12 mL of 2% ammonium hydroxide in methanol and elute the analytes from the cartridges by pulling the 4 mL of methanol through the sample transfer tubes and the cartridges. Use a low vacuum such that the solvent exits the cartridge in a dropwise fashion.

10.3.7. NOTE: If empty plastic reservoirs are used in place of the sample transfer tubes to pass the samples through the cartridges, these reservoirs must be treated like the transfer tubes. After the reservoirs have been rinsed in Section 10.3.3, the elution solvent used to rinse the sample bottles must be swirled down the sides of the reservoirs while eluting the cartridge to ensure that any method analytes on the surface of the reservoirs are transferred to the extract.

10.3.8. CLEAN-UP CARTRIDGE ELUTION, Elute the clean-up cartridge with 8 additional mLs of methanol and draw the aliquot through the cartridge. Use a low vacuum such that the solvent exits the cartridge in a dropwise fashion.

10.3.9. Fractions 1 and 2 are to be combined during the concentration stage (section10.8).

10.4. Sample Prep and Extraction Protocol for Soils, Solids and Sediments.

10.4.1. Homogenize and weigh 4 grams of sample (measured to the nearest hundredth of a gram) into a 50 mL polypropylene centrifuge tube. For laboratory control blanks and spikes, 4 grams of clean sand is used.

10.4.2. Add 40 µL of the EIS PDS (Sect. 8.2.2) to each sample and QC.

10.4.3. If the sample is an LCS, LCSD, MS, or MSD, add the necessary amount of analyte PDS (Sect. 8.2.6). Cap and invert each sample to mix.

10.4.4. To all samples, add 10 mLs of methanol, cap, vortex for 25 seconds at 2500 RPM.

10.4.5. Following mixing, sonicate each sample for 30 minutes and let samples sit overnight (at least 2 hours is required for RUSH samples).

10.4.6. Centrifuge each sample at 3500RPM for 10 minutes.

10.4.7. Remove 5mL of supernatant, and reserve for clean-up.

10.5. Sample Prep and Extraction Protocol for Tissues, Oils and Biosolids.

10.5.1. Homogenize and weigh 2-8 grams of sample (measured to the nearest hundredth of a gram) into a 50 mL polypropylene centrifuge tube. For laboratory control blanks and spikes, 4 grams of clean sand is used.

10.5.2. Add 40 µL of the EIS PDS (Sect. 8.2.2) to each sample and QC.

10.5.3. If the sample is an LCS, LCSD, MS, or MSD, add the necessary amount of analyte PDS (Sect. 8.2.6). Cap and invert each sample to mix.

10.5.4. Add 100 ul of Ammonium Hydroxide.

10.5.5. To all samples, add 10 mLs of methanol, cap, vortex for 25-30 seconds at 2500 RPM.

10.5.6. Following mixing, sonicate each sample for 30 minutes and let samples sit for 2 hours.

10.5.7. Centrifuge each sample at 3500RPM for 10 minutes.

10.5.8. Remove 5 mLs of the supernatant, and reserve for clean-up.

10.6. Extract Clean-up: Soils, Solids and Sediment Matrices

10.6.1. CARTRIDGE CLEAN-UP AND CONDITIONING –. Rinse each cartridge with 15 mL of methanol and discard. If the cartridge goes dry during the conditioning phase, the conditioning must be started over. Attach the sample transfer tubes (Sect. 7.11.3), turn on the vacuum, and begin adding sample to the cartridge. For Soils extracts, transfer 5 mLs of the MeOH eluate to the cartridge. Samples should be allowed to pass through the cartridge by gravity feed at a dropwise rate to ensure adequate contact time with the cartridge sorbent. Vacuum is only applied if the flow of solvent through the cartridge stops.

10.6.2. Adjust the vacuum so that the approximate flow rate is 1-2 mL/min. Do not allow the cartridge to go dry before all the sample has passed through.

10.6.3. SAMPLE BOTTLE AND CARTRIDGE RINSE – After the entire sample has passed through the cartridge, rinse the sample collection vial with two 4-mL aliquots of methanol and draw each aliquot through the cartridges. Draw air or nitrogen through the cartridge for 5 min at high vacuum (10-15 in. Hg).

10.6.4. If extracts are not to be immediately evaporated, cover collection tubes and store at ambient temperature till concentration.

NOTE: Due to the possible volatility and suspect degradation of sulfonamides and sulfonamide ethanols when exposed to heat, a portion of the eluate may be retained and analyzed independently with no evaporation for these analytes.

10.7. Extract Clean-up: Tissues, Oils and Biosolids

10.7.1. CARTRIDGE CLEAN-UP AND CONDITIONING –. Stack a 500 mg WAX cartridge onto a 250 mg GCB cartridge. Rinse each cartridge set with 10 mL of 2% NH_4OH and discard. Immediately rinse each cartridge stack with 15 mLs MeOH and discard, If the cartridge goes

dry during the conditioning phase, the conditioning must be started over. Attach the sample transfer tubes (Sect. 7.11.3), turn on the vacuum.

10.7.2. Adjust the vacuum so that the approximate flow rate is 1-2 mL/min. Do not allow the cartridge to go dry before all the sample has passed through.

10.7.3. SAMPLE elution AND CARTRIDGE RINSE – Load 5 mLs of the MeOH sample extract to the cartridge. After the entire sample has passed through the cartridge, rinse the cartridges with 5-mLs of methanol and draw through the cartridges. Immediately add and elute 2 5mL aliquots of 2% NH_4OH to the cartridges, collecting the eluate with the MeOH eluate.

10.7.4. If extracts are not to be immediately evaporated, cover collection tubes and store at ambient temperature till concentration.

10.7.5. NOTE: Due to the possible volatility and suspect degradation of sulfonamides and sulfonamide ethanols when exposed to heat, a portion of the eluate may be retained and analyzed independently with no evaporation for these analytes.

10.8. Extract Concentration

10.8.1. Concentrate the extract to dryness under a gentle stream of nitrogen in a heated water bath (60-65 °C) to remove all the water/methanol mix. Add the appropriate amount of 80:20% (vol/vol) methanol:water solution and 20 µl of the ID REC PDS (Sect. 8.2.8) to the collection vial to bring the volume to 1 mL and vortex. Transfer two aliquots with a plastic pipet (Sect. 7.8) into 2 polypropylene autosampler vials.

10.8.2. NOTE: It is recommended that the entire 1-mL aliquot not be transferred to the autosampler vial because the polypropylene autosampler caps do not reseal after injection. Therefore, do not store the extracts in the autosampler vials as evaporation losses can occur occasionally in these autosampler vials. Extracts can be split between 2 X 700 µl vials (Sect. 7.5).

10.9. Sample Volume Determination

10.9.1. If the level of the sample was marked on the sample bottle, use a graduated cylinder to measure the volume of water required to fill the original sample bottle to the mark made prior to extraction. Determine to the nearest 10 mL.

10.9.2. If using weight to determine volume, weigh the empty bottle to the nearest 10 g and determine the sample weight by subtraction of the empty bottle weight from the original sample weight (Sect. 10.2.2). Assume a sample density of 1.0 g/mL. In either case, the sample volume will be used in the final calculations of the analyte concentration (Sect. 11.2).

10.10. Initial Calibration - Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed. After the initial calibration is successful, a CCV is required at the beginning and end of each period in which analyses are performed, and after every tenth Field Sample.

10.10.1. ESI-MS/MS TUNE

10.10.1.1. Calibrate the mass scale of the MS with the calibration compounds and procedures prescribed by the manufacturer.

10.10.1.2. Optimize the [M-H]- for each method analyte by infusing approximately 0.5-1.0 µg/mL of each analyte (prepared in the initial mobile phase conditions) directly into the MS at the chosen LC mobile phase flow rate (approximately 0.4 mL/min). This tune can be done on a mix of method analytes. The MS parameters (voltages, temperatures, gas flows, etc.) are

varied until optimal analyte responses are determined. The method analytes may have different optima requiring some compromise between the optima.

10.10.1.3. Optimize the product ion for each analyte by infusing approximately 0.5-1.0 µg/mL of each analyte (prepared in the initial mobile phase conditions) directly into the MS at the chosen LC mobile phase flow rate (approximately 0.4 mL/min). This tune can be done on a mix of method analytes. The MS/MS parameters (collision gas pressure, collision energy, etc.) are varied until optimal analyte responses are determined. Typically, carboxylic acids have very similar MS/MS conditions and sulfonic acids have similar MS/MS conditions.

10.10.2. Establish LC operating parameters that optimize resolution and peak shape. Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted.

10.10.2.1. Cautions: LC system components, as well as the mobile phase constituents, contain many of the method analytes in this method. Thus, these PFAS's will build up on the head of the LC column during mobile phase equilibration. To minimize the background PFAS peaks and to keep background levels constant, the time the LC column sits at initial conditions must be kept constant and as short as possible (while ensuring reproducible retention times). In addition, prior to daily use, flush the column with 100% methanol for at least 20 min before initiating a sequence. It may be necessary on some systems to flush other LC components such as wash syringes, sample needles or any other system components before daily use.

10.10.3. Inject (2µl for Sciex systems, 3µl for Waters systems, 20µl for MeOH fractions) a mid-level CAL standard under LC/MS conditions to obtain the retention times of each method analyte. If analyzing for PFTA, ensure that the LC conditions are adequate to prevent co-elution of PFTA and the mobile phase interferants. These interferants have the same precursor and products ions as PFTA, and under faster LC conditions may co-elute with PFTA. Divide the chromatogram into retention time windows each of which contains one or more chromatographic peaks. During MS/MS analysis, fragment a small number of selected precursor ions ([M-H]-) for the analytes in each window and choose the most abundant product ion. For maximum sensitivity, small mass windows of ±0.5 daltons around the product ion mass were used for quantitation.

10.10.4. Inject a mid-level CAL standard under optimized LC/MS/MS conditions to ensure that each method analyte is observed in its MS/MS window and that there are at least 10 scans across the peak for optimum precision.

10.10.4.1. NOTE: PFHxS, PFOS, NMeFOSAA, and NEtFOSAA have multiple chromatographic peaks using the LC conditions in Table 7 due to chromatographic resolution of the linear and branched isomers of these compounds. Most PFAS's are produced by two different processes. One process gives rise to linear PFAS's only while the other process produces both linear and branched isomers. Thus, both branched and linear PFAS's can potentially be found in the environment. For the aforementioned compounds that give rise to more than one peak, all the chromatographic peaks observed in the standard must be integrated and the areas totaled. Chromatographic peaks in a sample must be integrated in the same way as the CAL standard.

10.10.5. Prepare a set of CAL standards as described in Section 8.2.9. The lowest concentration CAL standard must be at or below the RL (2 ng/L), which may depend on system sensitivity.

10.10.6. The LC/MS/MS system is calibrated using the isotope dilution technique. Target analytes are quantitated against their isotopically labeled analog (Extracted Internal Standard) where commercially available. If a labeled analog is not commercially available, the extracted internal standard with the closest retention time and /or closest chemical similarity is to be used. Use the LC/MS/MS data system software to generate a linear regression or quadratic calibration curve for each of the analytes. This curve must always be forced through zero and may be concentration weighted, if necessary. Forcing zero allows for a better estimate of the background levels of method analytes. A minimum of 5 levels are required for a linear calibration model and a minimum of 6 levels are required for a quadratic calibration model.

10.10.7. CALIBRATION ACCEPTANCE CRITERIA – A linear or quadratic fit is acceptable if the coefficient of determination (r^2) is greater than 0.990. All curves must force through zero (some of the SCIEX software versions do not allow forcing through zero for quadratic curves, therefore an additional standard at approximately half the RL must be analyzed). Any target analyte calibrated using average response must have a relative standard deviation (RSD) of ≤20%. When quantitated using the initial calibration curve, each calibration point, except the lowest point, for each analyte must calculate to be within 70-130% of its true value. Any CAL point within 2x the RL must calculate to be within 50-150% of its true value. If these criteria cannot be met, the analyst will have difficulty meeting ongoing QC criteria. It is recommended that corrective action is taken to reanalyze the CAL standards, restrict the range of calibration, or select an alternate method of calibration (forcing the curve through zero is still required). EIS compounds must have an RSD of ≤20%; the fluorotelomer EIS compounds must have an RSD ≤30%.

10.10.7.1. **CAUTION:** When acquiring MS/MS data, LC operating conditions must be carefully reproduced for each analysis to provide reproducible retention times. If this is not done, the correct ions will not be monitored at the appropriate times. As a precautionary measure, the chromatographic peaks in each window must not elute too close to the edge of the segment time window.

10.10.8. CONTINUING CALIBRATION CHECK (CCV) – Minimum daily calibration verification is as follows. Verify the initial calibration at the beginning and end of each group of analyses, and after every tenth sample during analyses. In this context, a “sample” is considered to be a Field Sample. MBs, CCVs, LCSs, MSs, FDs FRBs and MSDs are not counted as samples. The beginning CCV of each analysis batch must be at or below the RL in order to verify instrument sensitivity prior to any analyses. If standards have been prepared such that all low CAL points are not in the same CAL solution, it may be necessary to analyze two CAL standards to meet this requirement. Alternatively, the analyte concentrations in the analyte PDS may be customized to meet these criteria.

10.10.8.1. Inject an aliquot of the appropriate concentration CAL standard and analyze with the same conditions used during the initial calibration.

10.10.8.2. Calculate the concentration of each analyte and EIS in the CCV. The calculated amount for each analyte for medium level CCVs must be within 70 – 130% of the true value with an allowance of 10% of the reported analytes to be less than 70% (minimum recovery is 10%) for compounds without their own labelled EIS. For CCVs, if the recovery of the corresponding target analytes meets the acceptance criteria but the EIS is outside criteria, the data can be used. The calculated amount for the lowest calibration point for each analyte must be within 50-150%. If these conditions do not exist, then all data for the problem analyte must be considered invalid, and remedial action should be taken which may require recalibration. Any Field or QC Samples that have been analyzed since the last acceptable calibration verification should be reanalyzed after adequate calibration has been restored, with the following exception. If the CCV fails

because the calculated concentration is greater than 130% (150% for the low-level CCV) for a particular method analyte, and Field Sample extracts show no detection for that method analyte, non-detects may be reported without re-analysis.

10.10.8.3. REMEDIAL ACTION – Failure to meet CCV QC performance criteria may require remedial action. Major maintenance, such as cleaning the electrospray probe, atmospheric pressure ionization source, cleaning the mass analyzer, replacing the LC column, etc., requires recalibration (Sect 10.10) and verification of sensitivity by analyzing a CCV at or below the RL (Sect 10.11).

10.10.9. EXTRACT ANALYSIS

10.10.9.1. Establish operating conditions equivalent to those summarized in Tables 7-9 of Section 16.

10.10.9.2. Establish an appropriate retention time window for each analyte. This should be based on measurements of actual retention time variation for each method analyte in CAL standard solutions analyzed on the LC over the course of time. A value of plus or minus three times the standard deviation of the retention time obtained for each method analyte while establishing the initial calibration can be used to calculate a suggested window size. However, the experience of the analyst should weigh heavily on the determination of the appropriate retention window size.

10.10.9.3. Calibrate the system by either the analysis of a calibration curve (Sect. 10.10) or by confirming the initial calibration is still valid by analyzing a CCV as described in Section 10.11.

10.10.9.4. Begin analyzing Field Samples, including QC samples, at their appropriate frequency by injecting the same size aliquots under the same conditions used to analyze the CAL standards.

10.10.9.5. At the conclusion of data acquisition, use the same software that was used in the calibration procedure to identify peaks of interest in predetermined retention time windows. Use the data system software to examine the ion abundances of the peaks in the chromatogram. Identify an analyte by comparison of its retention time with that of the corresponding method analyte peak in a reference standard.

10.10.9.6. The analyst must not extrapolate beyond the established calibration range. If an analyte peak area exceeds the range of the initial calibration curve, the sample should be re-extracted with a reduced sample volume in order to bring the out of range target analytes into the calibration range. If a smaller sample size would not be representative of the entire sample, the following options are recommended. Re-extract an additional aliquot of sufficient size to insure that it is representative of the entire sample. Spike it with a higher concentration of internal standard. Prior to LC/MS analysis, dilute the sample so that it has a concentration of internal standard equivalent to that present in the calibration standard. Then, analyze the diluted extract.

11. Data Evaluation, Calculations and Reporting.

11.1. Complete chromatographic resolution is not necessary for accurate and precise measurements of analyte concentrations using MS/MS. In validating this method, concentrations were calculated by measuring the product ions listed in Table 9.

11.2. Calculate analyte concentrations using the multipoint calibration established in Section 10.9. Do not use daily calibration verification data to quantitate analytes in samples. Adjust final analyte concentrations to reflect the actual sample volume determined in Section 10.8

Cex = (Area of target analyte * Concentration of Labeled analog) / (area of labeled analog * CF)

C_s = (Cex / sample volume in mL) * 1000

Cex = The concentration of the analyte in the extract
CF = calibration factor from calibration.

11.3. Prior to reporting the data, the chromatogram should be reviewed for any incorrect peak identification or poor integration.

11.4. PFHxS, PFOS, PFOA, NMeFOSAA, and NEtFOSAA have multiple chromatographic peaks using the LC conditions in Table 7 due to the linear and branch isomers of these compounds (Sect. 10.10.4.). The areas of all the linear and branched isomer peaks observed in the CAL standards for each of these analytes must be summed and the concentrations reported as a total for each of these analytes.

11.5. Calculations must utilize all available digits of precision, but final reported concentrations should be rounded to an appropriate number of significant figures (one digit of uncertainty), typically two, and not more than three significant figures.

11.6. Peak responses must be at least 3 times the background noise level (S/N 3:1). If the S/N ratio is not due to high background noise, the sample may be diluted (either by extraction or analytically) to reduce the noise.

11.7. Target analytes, Extracted Internal Standards, and Response Internal Standard RTs must fall within +/-0.4minutes of the predicted retention times of the midpoint of the initial calibration curve or daily calibration verification standard. Additionally, a target analyte with a corresponding isotopically labelled analog must elute within +/- 0.1minutes of the associated EIS.

11.8. The ion ratio between the quantification ion and confirmation ion response must fall within +/-50% of the ratio observed in the mid-point initial calibration standard. When this ratio is not met, the data must be flagged.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1. Section 9.0 outlines sample batch QC acceptance criteria. If non-compliant organic compound results are to be reported, the Organic Section Head and/or the Laboratory Director, and the Operations Manager must approve the reporting of these results. The laboratory Project Manager shall be notified and may choose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst, Data Reviewer, or Department Supervisor performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

12.2. All results for the organic compounds of interest are reportable without qualification if extraction and analytical holding times are met, preservation requirements (including cooler temperatures) are met, all QC criteria are met, and matrix interference is not suspected during extraction or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

13. Method Performance

13.1. Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

13.1.1. The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2. Demonstration of Capability Studies

13.2.1. Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.2. The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

14.1. Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

14.2. This method utilizes SPE to extract analytes from water. It requires the use of very small volumes of organic solvent and very small quantities of pure analytes, thereby minimizing the potential hazards to both the analyst and the environment as compared to the use of large volumes of organic solvents in conventional liquid-liquid extractions.

14.3. The analytical procedures described in this method generate relatively small amounts of waste since only small amounts of reagents and solvents are used. The matrices of concern are finished drinking water or source water. However, laboratory waste management practices must be conducted consistent with all applicable rules and regulations, and that laboratories protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Also, compliance is required with any sewage discharge permits and regulations, particularly the hazardous waste identification rules and land disposal restrictions.

15. Referenced Documents

Chemical Hygiene Plan – ID 2124
Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP – ID 1732
Demonstration of Capability (DOC) Generation SOP – ID 1739
Hazardous Waste Management and Disposal SOP – ID 1728

16. REVISION HISTORY

Revisions Made from Prior Version

Section	Description of Change
6.5.1.1	Updated Hold Time for Aqueous, Soils and Tissues.
6.5.1.2	Added specific hold time criteria for Maine
8.2.9	Revised the calibration standard levels
9.1.1.1	Added Corrective Action for Method Blanks
9.1.2	Changed FRB to project-specific basis
9.2.1	Added LCS criteria specific to NJ
9.2.4	Added Corrective Action for LCS
9.4.2	Revisions to EIS criteria
10.10.7	Revisions to Calibration Acceptance Criteria

16. Attachments

Table 7a: LC Method Conditions

Time (min)	2 mM Ammonium Acetate (5:95 MeOH/H_2O)	100% Methanol
Initial	100.0	0.0
1.0	100.0	0.0
2.2	85.0	15.0
11	20.0	80.0
11.4	0.0	100.0
12.4	100.0	00.0
15.5	100.0	0.0
Waters Aquity UPLC ® $BEHC_{18}$ 2.1 x 50 mm packed with 1.7 µm BEH C_{18} stationary phase Flow rate of 0.4 mL/min 3 µL injection		

Table 7b: LC Method Conditions (MeOH Fraction)

Time (min)	2 mM Ammonium Acetate (5:95 MeOH/H_2O)	100% Methanol
Initial	100.0	0.0
1.0	50.0	50.0
4.5	1.0	99.0
4.95	1.0	99.0
5.0	100.0	0.0
5.5	100.0	0.0
Waters Aquity UPLC ® $BEHC_{18}$ 2.1 x 50 mm packed with 1.7 µm BEH C_{18} stationary phase Flow rate of 0.6 mL/min 20 µL injection		

Table 8: ESI-MS Method Conditions

ESI Conditions	
Polarity	Negative ion
Capillary needle voltage	.5 kV
Cone Gas Flow	25 L/hr
Nitrogen Desolvation gas	1000 L/hr
Desolvation gas temp.	500 ºC

Table 9a: Method Analyte Source, Retention Times (RTs), and EIS References

#	Analyte	Transition	Confirmation Mass	RT	IS	Type
1	M3PFBA	216>171		2.65		REC
2	PFBA	213 > 169	N/A	2.65	3: M4PFBA	
3	M4PFBA	217 > 172		2.65	1: M3PBA	EIS
4	PFPeA	263 > 219	N/A	5.67	5: M5PFPEA	
5	M5PFPEA	268 > 223		5.66	1: M3PFBA	EIS
6	PFBS	299 > 80	99	6.35	7: M3PFBS	
7	M3PFBS	302 > 80		6.35	29:M4PFOS	EIS
8	FTS 4:2	327 > 307	81	7.47	9: M2-4:2FTS	
9	M2-4:2FTS	329 > 81		7.47	29:M4PFOS	EIS
10	PFHxA	313 > 269	119	7.57	11: M5PFHxA	
11	M5PFHxA	318 > 273		7.57	19:M2PFOA	EIS
12	PFPeS	349 > 80	99	7.88	18: M3PFHxS	
13	PFHpA	363 > 319	169	8.80	14: M4PFHpA	
14	M4PFHpA	367 > 322		8.80	19:M2PFOA	EIS
15	L-PFHxS	399 > 80	99	8.94	18: M3PFHxS	
16	br-PFHxS	399 > 80	99	8.72	18: M3PFHxS	
17	PFHxS Total	399 > 80		8.94	18: M3PFHxS	
18	M3PFHxS	402 > 80		8.94	29:M4PFOS	EIS
19	M2PFOA	415 > 370		9.7		REC
20	L-PFOA	413 > 369	169	9.7	23: M8PFOA	
21	br-PFOA	413 > 369	169	9.48	23: M8PFOA	
22	PFOA Total	413 > 369		9.7	23: M8PFOA	
23	M8PFOA	421 > 376		9.7	19: M2PFOA	EIS
24	FTS 6:2	427 > 407	81	9.66	25: M2-6:2FTS	
25	M2-6:2FTS	429 > 409		9.66	29:M4PFOS	EIS
26	PFHpS	449 > 80	99	9.78	33: M8PFOS	
27	PFNA	463 > 419	219	10.41	33: M9PFNA	
28	M9PFNA	472 > 427		10.41	19: M2PFOA	EIS
29	M4PFOS	503 > 80		10.45		REC
30	L-PFOS	499 > 80	99	10.45	33: M8PFOS	
31	br-PFOS	499 > 80	99	10.27	33: M8PFOS	
32	PFOS Total	499 > 80		10.45	33: M8PFOS	
33	M8PFOS	507 > 80		10.45	29: M4PFOS	EIS
34	FTS 8:2	527 > 507	81	10.99	35: M2-8:2FTS	
35	M2-8:2FTS	529 > 509		10.99	29:M4PFOS	EIS
36	M2PFDA	515 > 470		11.00		REC
37	PFDA	513 > 469	219	11.00	38: M6PFDA	
38	M6PFDA	519 > 474		11.00	36: M2PFDA	EIS
39	PFNS	549 > 80	99	11.02	33:M8PFOS	
40	br-NMeFOSAA	570 > 419	483	11.41	41: D3-NMeFOSAA	

#	Analyte	Transition	Confirmation Mass	RT	IS	Type
41	L-NMeFOSAA	570 > 419	483	11.41	41: D3-NMeFOSAA	
42	NMeFOSAA total	570 > 419		11.41	41: D3-NMeFOSAA	
43	d3-NMeFOSAA	573 > 419		11.41	36: M2PFOA	EIS
44	PFOSA	498 > 78	478	11.48	29: M8FOSA	
45	M8FOSA	506 > 78		11.48	19: M2PFOA	EIS

#	Analyte	Transition	Confirmation Mass	RT	IS	Type
46	PFUnDA	563 > 519	269	11.51	41: M7-PFUDA	
47	M7-PFUDA	570 > 525		11.51	36: M2PFDA	EIS
48	PFDS	599 > 80	99	11.51	33:M8PFOS	
49	br-NEtFOSAA	584 > 419	526	11.68	48: d5-NEtFOSAA	
50	L-NEtFOSAA	584 > 419	526	11.68	48: d5-NEtFOSAA	
51	NEtFOSAA total	584 > 419		11.68	48: d5-NEtFOSAA	
52	d5-NEtFOSAA	589 > 419		11.68	36: M2PFOA	EIS
53	PFDoA	613 > 569	319	11.96	50: MPFDOA	
54	MPFDOA	615 > 570		11.96	36: M2PFDA	EIS
55	PFTrdA	663 > 619	169	12.34	53: M2PFTEDA and 50:MPFDOA avg	
56	PFTeA	713 > 669	169	12.6	53: M2PFTEDA	
57	M2PFTEDA	715 > 670		12.6	36: M2PFDA	EIS
58	M3HFPO-DA	287>169		7.97	19: M2PFOA	EIS
59	HFPO-DA	285>169	185	7.97	54: M3HFPO-DA	
60	ADONA	377>251	85	8.00	23: M8PFOA	
61	PFHxDA	813>769		13.20	59: M2PFHxDA	
62	PFODA	913>869		13.50	59: M2PFHxDA	
63	M2PFHxDA	815>770		13.20	36:M2PFDA	EIS
64	NEtFOSA	526>169	119	11.00	61: d5-NEtFOSA	
65	NMeFOSA	512>169	219	10.50	63: d3-NMeFOSA	
66	d3-NMeFOSA	515>169		10.50	19: M2PFOA	EIS
67	d5-NEtFOSA	531>169		11.00	19: M2PFOA	EIS
68	NMeFOSE	616>59	N/A	11.25	66: d7-NMeFOSE	
69	NEtFOSE	630>59	N/A	10.75	67: d9-NEtFOSE	
70	d7-NMeFOSE	623>59		11.25	19: M2PFOA	EIS
71	d9-NEtFOSE	639>59		10.75	19: M2PFOA	EIS
72	FTS 10:2	627>607		11.50	25: M2-8:2FTS	
73	PFDoS	699>80	99	12.50	33: M8PFOS	
74	9ClPF3ONS	531>351	353	10.23	33: M8PFOS	
75	11ClPF3OUdS	631>451	453	11.27	33: M8PFOS	
76	PFPrS	249>80		3.40	7: M3PFBS	
77	PFMPA	229>85	N/A	3.39	1: M3PFBA	
78	PFMBA	279 .85	N/A	5.75	1: M5PFPEA	
79	PFEESA	315>135	83	6.45	18: M3PFHxS	
80	NFDHA	295>201	85	6.79	11: M5PFHxA	

Table 9b: Methanol Analyte Source, Retention Times (RTs), and EIS References

#	Analyte	Transition	RT	IS	Type
1	M2PFNA	472 > 427	2.55		IS
2	M2PFUdA	213 > 169	2.87		IS
3	M8FOSA	217 > 172	2.86	1: M2PFNA	EIS
4 5	FOSA	263 > 219	2.86	3: M8FOSA	
6	D3-NMeFOSA	515>169	3.22	1: M2PFNA	EIS
7	NMeFOSA	512>169	3.22	6: d3-NMeFOSA	
8	D5-NEtFOSAA	531>169	3.41	1: M2PFNA	EIS
9	NEtFOSAA	526>169	3.41	8: d5-NEtFOSA	
10	D7-NMeFOSE	563>126	3.23	1: M2PFNA	EIS
11	NMeFOSE	556>122	3.23	10: d7-NMeFOSE	
12	D9-NEtFOSE	579>142	3.40	11: M2PFNA	EIS
13	NEtFOSE	570>136	3.40	67: d9-NEtFOSE	

Determination of Polychlorinated Biphenyls (PCBs) as Aroclors by Gas Chromatography/Electron Capture Detection (GC/ECD)

References: Method 8082A, Rev. 1, February 2007 and Method 8000C Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Final update III, December 1996 (USEPA, Office of Solid Waste and Emergency Response, Washington, DC).

Quality Assurance and Quality Control for SW-846 Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography (GC) for the Massachusetts Contingency Plan (MCP)

State of Connecticut, Department of Environmental Protection, RRCP, Version 2.0, July 2006.

Compendium Method TO10A – Determination of Pesticides and PCBs in Ambient Air Using Low Volume Polyurethane Foam (PUF) Sampling Followed by GC/ECD Detection

Compendium Method TO-4A – Determination of Pesticides and PCB's in Ambient Air Using High Volume Polyurethane Foam (PUF) Sampling Followed by Gar Chromatographic/Multi-Detector Detection (GC/MD)

1. Scope and Application

Matrices: Water, soil, sediment, sludge, product, PUF, and tissue (either animal or vegetable)

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the analysis and quantification of sample extracts for Polychlorinated Biphenyls (PCB) Aroclors as Total PCB by gas chromatography/electron capture detector. See Table I for the target compound list of PCB as Aroclors with associated CAS Registry Numbers.

The sensitivity of the method usually depends on the concentration of interferences rather than on instrumental limitations. If interferences prevent detection of the analytes, the method may also be performed on samples that have undergone cleanup.

The following extraction and cleanup methods may apply, prior to sample analysis:

- *Extraction of Water Samples by Separatory Funnel* (2156),
- *Microscale Solvent Extraction - 3570* (2172)
- *Soxhlet Extraction (2173)*
- *Soxhlet Extraction of PUFs (*2167*)*
- *Tissue Preparation and Homogenization* (2166),
- *Shaker Table Extraction* (2261),
- *Sulfur Cleanup* (2168),
- *Sulfuric Acid Cleanup* (2169),
- *Silica Gel cleanup* (2170)
- *GPC Cleanup by HPLC* (2167)

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC-ECD and in the interpretation of GC-ECD data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Sample extracts are analyzed on a gas chromatograph which is fitted with two capillary columns of differing polarities each employing separate detectors. The extracts are spiked with internal standard prior to analysis. The target analytes are resolved on each column and detected using an electron capture detector (ECD). Analytes are introduced into the GC/ECD by injecting a known volume of the calibration standards, quality control samples, and sample extracts into the GC which is temperature and flow programmed to separate the analytes. Identification of target analytes is accomplished by confirming a target hit on two dissimilar columns using Retention Time (RT) and Pattern Recognition (PR). Concentrations are calculated from the ECD response using internal standard techniques. Concentrations are determined using mean Relative Response Factor derived from multi-level calibration curve.

2.1 Method Modifications from Reference

Internal standard calibration is used. The internal standard used is 1-bromo-2- nitrobenzene.

3. Reporting Limits

The Reporting Limit for aqueous samples is 0.02 ug/L for individual Aroclors. This value is based on a 1mL final volume and 500 or 1000 mL of environmental sample extracted. The Reporting Limit for solid/tissue samples is 2.5ug/kg and 15 ug/Kg for individual Aroclors. This value is based on 2 mL final volume and 10-30 grams of environmental sample extracted (wet weight) using extraction method 3570 (MSE). The reporting limit for Aroclors in PUF air samples is 20-100ng/PUF cartridge, based on the Soxhlet extraction of one low or high volume PUF and a final volume of 1mL. Detection limits will vary with the individual sample matrix, sample preparation procedures, instrument calibration range, and volume of sample analyzed.

4. Interferences

4.1 Instrumental

4.1.1 Only high purity gases are used in the GC system to eliminate this source of possible contamination. Both Hydrogen (carrier gas) and Nitrogen (detector make-up gas) are certified by the gas supplier.

4.1.2 Preventive instrument maintenance is performed routinely, and whenever highly contaminated extracts are analyzed that could result in chromatographic interferences or result in degradation of system performance.

4.1.3 Glassware must be scrupulously cleaned. This procedure is detailed in the Laboratory Glassware cleaning SOP/1753. Store dry glassware in a clean environment.

4.2 Parameters

4.2.1 Contaminants co-extracted from the sample may cause matrix interferences. The extent of matrix interferences will vary considerably from sample to sample, depending upon the nature of the environment and matrix being investigated. In some cases depending on the severity of the observed interferences, a target hit on one column may be biased high when interfered with. Skilled analyst judgment is imperative in determining if a chromatographic peak appears interfered. In these cases the non-interfered, non-biased hit can be forced for reporting purposes providing a more suitable value. In extreme cases of interference

additional cleanups can be employed, or it may be necessary to analytically re-run the sample at a dilution.

4.2.2 The ECD is theoretically a halogen specific detector. However, phthalate esters can be a major source of contamination in that they do respond to the ECD and can interfere with accurate determination of the analytes of interest. No forms of plastic or any material containing plasticizers (phthalates) should be used in conjunction with any part of this analysis.

4.2.3 Elemental sulfur is readily extracted from soil samples and may cause chromatographic interferences in the determination of pesticides. Sulfur can be removed through the use of Method 3660B. In addition, all samples for PCB analysis are acid cleaned following Method 3665A. Associated SOPs are noted in Section 1.0, above.

4.2.4 Care must be taken to ensure a complete hexane exchange is performed on the extract, because residual methylene chloride would be an interferent in the analysis. Residual methylene chloride can also damage the electron capture detectors.

4.2.5 The injector system of the gas chromatograph can become contaminated with non-volatile components from samples. This can act to decrease the resolution and responsiveness of the system. Maintenance of the analytical system should be performed when system performance degrades (liner changes, injection port cleaning, etc.) to avoid prolonged down time.

4.2.6 Aldol condensation products (4-methyl-4-hydroxy-2-pentanone and 4-methyl-3-pentene-2-one) can arise during soil extraction when using methylene chloride:acetone. These products may interfere with detection of analytes and should be monitored.

4.2.7 Raw GC data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

4.2.8 Per method 8082A each analytical column analyzing PCB's must be evaluated for the presence of coelutions amongst PCB congeners with pesticides. An aliquot of PCB pesticide LCS solution is prepared and analyzed routinely on each analytical column in the laboratory. The subsequent coelutions of these compounds are documented and stored internally in the "ECD Standard Verification Logbook" within the facility.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. PCBs have been tentatively classified as known or suspected human or mammalian carcinogens. Additional references to laboratory safety are available in the Chemical Hygiene Plan. All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in 500mL, 1L amber glass bottles and stored without preservative at 4°C

Soil/sediment/tissue samples are collected in 4-8oz. glass soil jars.

Air PUF samples are collected following the guidance in reference method TO-10A and TO-4A Compendium.

6.2 Sample Preservation

Aqueous and Air PUF samples are stored without preservative at 4°C

Tissue Samples are stored at 4°C, or if desired, frozen.

Soil/sediment samples are stored at 4°C, or if desired, frozen.

6.3 Sample Handling

The hold time for this method is 7 days for the extraction of Air PUF samples, and 14 days for the extraction of tissue samples. Aqueous and solid samples are a 1 year holding time. If a sediment or tissue samples are frozen, this suspends the holding time until removal from the freezer. All extracts must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph, Agilent 6890, 7890: An analytical system complete with gas chromatograph configured for split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system.

7.2 GC Columns: Alpha utilizes dual-column analyses. The dual-column approach involves either a single injection that is split between two columns that are mounted in a single gas chromatograph. Typical column pair used is listed below. Other columns may be used as long as method performance criteria can be met.

7.2.1 Column pair:

RTX-CLP: Cat. #11141 from Restek or equivalent; 30m, 0.32mm, 0.32µm

RTX-CLPII Cat. #11324 from Restek or equivalent; 30m, 0.32mm, 0.25µm

7.3 Guard Column:

Cat. #10027 from Restek or equivalent; 5m, 0.32mm

7.4 Class "A" Volumetric Flasks: 10mL and 25mL, for standards preparation

7.5 Syringes: 10 µL – 1000 µL

7.6 Gooseneck splitless injecton liner, Cat #20799-214.5 from Restek or equivalent
7.7 Universal "Y" Press-tight tee split: Cat. #20406 from Restek or equivalent

7.7 Autosampler: Adapted onto the Gas Chromatograph. Model is HP 7890 or 6890 series autosampler with a GC autosampler controller.

7.8 Computer: With Windows NT operating software utilizing HP Enviroquant G1701BA Version B.01.00 software.

7.9 Gases: Hydrogen, ultra high purity grade and Nitrogen of equivalent purity. Hydrogen is generally supplied by a Proton Hogen GC 600 Hydrogen generator.

7.10 Glassware: Assorted Class-A volumetric flasks consisting of 5mL through 200mL flasks with ground-glass stoppers for preparing reagents, standards, and measuring sample volumes for sample requiring dilution beyond 1:100.

7.11 Pipettes: Disposable Pasteur brand, or equivalent.

7.12 Cahn balance: Capable of accurate measurement to the nearest 0.001 mg.

7.13 Analytical balance: Capable of accurate measurement to the nearest 0.0001 g.

7.14 Non-preserved, certified, pre-cleaned 40mL VOA vials, with Teflon-lined screw caps.

7.15 2 mL amber and clear GC vials, with Teflon-lined crew caps.

7.16 Stainless steel Spatula.

8. Reagents and Standards

8.1 Solvents: All solvent expirations determined as indicated by manufacturer guidelines.

8.1.1 Methylene Chloride, ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (1816) for additional details regarding solvent purity.

8.1.2 Acetone, ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (1816) for additional details regarding solvent purity.

8.1.3 Hexane, ACS approved, Pesticide grade, see SOP *Reagent, Solvent, and Standard Control* (1816) for additional details regarding solvent purity.

8.1.4 Methanol, Purge and Trap grade, see SOP *Reagent, Solvent, and Standard Control* (1816) for additional details regarding solvent purity.

8.1.5 Diethyl ether, High purity, glass distilled

8.2 Primary Standard Solutions: All Primary standard solutions are purchased from commercial vendors as flame sealed ampule certified solutions. When a flame sealed ampule primary solution is opened, it is transferred to a labeled amber 2mL vial.

Primary Standards are stored as indicated by the vendor, away from light when not in use. Primary standards are discarded as indicated by the vendor expiration. If breakdown of a solution is observed, the solution will be discarded.

8.2.1 Aroclor 1016 Solution: Obtained from Accustandard at a concentration of 100ug/mL (catalog # C-216S-H-10X-PAK) – for the calibration curve and LCS and obtained from Restek at a concentration of 1000 ug/mL (catalog # 32006) – for ICV.

8.2.1 Aroclor 1260 Solution: Obtained from Accustandard at a concentration of 100ug/mL (catalog # C-260S-H-10X-PAK) – for the calibration curve and LCS and

obtained from Restek at a concentration of 1000ug/mL (catalog # C32012) – for LCS and ICV.

8.2.2 Primary source calibration solutions

Compound	Part Number
Aroclor 1016	C-261S-H-10X-PAK
Aroclor 1221	APP-9-159
Aroclor 1232	APP-9-160
Aroclor 1242	C-242S-H-10X
Aroclor 1248	C-248S-H-10X
Aroclor 1254	C-254S-H-10X
Arocor 1260	C-260S-H-10X-PAK
Aroclor 1262	APP-9-165
Aroclor 1268	APP-9-166

8.2.3 ICV Aroclor Solutions: Obtained from Ultra at a concentration of 100ug/mL

Compound	Part Number
Aroclor 1016	32006
Aroclor 1221	PP-291
Aroclor 1232	PP-301
Aroclor 1242	PP-311
Aroclor 1248	PP-341
Aroclor 1254	PP-351
Aroclor 1260	C32012
Aroclor 1262	PP-371
Aroclor 1268	PP-381

8.2.4 Pesticides Surrogate Standard Spiking Solution: Obtained from Ultra at a concentration of 200ug/mL (catalog # ISM-320) – for PCB Aroclors analysis.

8.2.5 Internal Standard Solution: 1-Bromo-2-nitrobenzene (Ultra, Cat. #PPS-351) is used as the internal standard and is added to all single-component calibration standards and sample extracts to achieve a concentration of 0.25µg/mL.

8.3 Stock Standard Solutions: All Stock Standard Solutions are prepared volumetrically by diluting and mixing the appropriate primary standard solutions in hexane. Stock standards expire 1 year from the date of preparation or sooner if routine QC tests indicate a problem. All stock solutions are stored in the freezer (-20°C -10C) in 10-40mL vials depending on the amount prepared. If breakdown of a solution is observed, the solution will be discarded.

8.3.1 Aroclor 1660 Calibration Stock Solution: Consists of the Aroclor 1016 and 1260 from Accustandard or equivalent, mixed with the extraction surrogates (TMX and DCB), at a stock concentration of 20,000ug/L for AR1660 and 1000ug/L for surrogates, in hexane. To prepare calibration stock solution, add 1mL of *Aroclor 1016/1260 Primary Solution* at 1000ug/mL and 0.5mL of *Pesticides Surrogate Standard Spiking Solution* at 200ug/mL to a 50mL volumetric flask. Dilute to the volume with hexane.

8.3.2 Aroclor 1660 ICV Stock Solution: Consists of the Aroclor 1016 and 1260 from Restek or equivalent, at a stock concentration of 20,000ug/L in hexane. To prepare calibration stock solution, add 1mL of an individual *Aroclor Primary Solution* at 1000ug/mL to a 50mL volumetric flask. Dilute to the volume with hexane.

8.3.3 Other Aroclors Calibration Stock Solution: Consists of the individual Aroclor from Accustandard or equivalent at a stock concentration of 20,000ug/L, in hexane. To prepare calibration stock solution, add 1mL of an *Aroclor Primary Solution* at 1000ug/mL to a 50mL volumetric flask. Dilute to the volume with hexane.

8.3.4 Other Aroclors ICV Stock Solution: Consists of the individual Aroclor from Ultra or equivalent at a stock concentration of 10,000ug/L, in hexane. To prepare calibration stock solution, add 1mL of an *Aroclor Primary Solution* at 100ug/mL to a 10mL volumetric flask. Dilute to the volume with hexane.

8.3.5 Pesticides Surrogate Stock Solution: Consists of Pesticides Surrogates (TMX and DCB) from Ultra or equivalent, at a stock concentration of 5000ug/L. To prepare pest surrogate stock solution add 0.625 mL of *Pesticides Surrogate Standard Spiking Solution* at 200ug/L to a 25mL volumetric flask. Dilute to the volume with hexane.

8.4 Working/Spiking Standard Solutions: All working/spiking standard solutions are prepared volumetrically by diluting and mixing appropriate primary or stock standard solutions in acetone, hexane or methylene chloride. Working/Spiking standards expire 6 months from the date of preparation or sooner if routine QC tests indicate the problem. All working/spiking solutions are stored in the freezer (-20℃ -10 ℃) in 10-40mL vials depending on the amount prepared. All spiking solutions must be assayed for use by analysis before release to the preparation lab***. All compounds must be within 20% of their true value.*** If breakdown of a solution is observed, the solution will be discarded.

8.4.1 Aroclor 1660 calibration standard solution: Consists of Aroclor 1016 and 1260 from our "Aroclor 1660 **calibration stock solution"** combined into one standard called Aroclor 1660 and the extraction surrogates (TMX and DCB) through dilution of Aroclor 1660 Calibration Stock solution. The extraction surrogates will only be combined with the 1660 calibration. An ICAL is run for each of the nine Aroclors before analysis. A 7 point calibration is analyzed for Aroclor 1660 while 1pt calibration is allowed per method for the other 7 aroclors. Aroclor 1660 must use average response for calibration type to have the ability to 1pt the other aroclors.

7-Level Curve Preparation for Aroclors

Calibration Level	Aroclor Conc.	Surrogate Conc.	Volume of Std Added	Final Volume in Hexane
Stock	20 00μg/L	2000μg/L		
Level1	20μg/L	2.0μg/mL	0.1mL of Level7.	10mL
Level 2	50μg/L	5.0μg/L	0.25mL of Level7	10mL
Level 3	100μg/L	10μg/mL	0.5mL of Level7	10mL
Level 4	250μg/L	25μg/mL	1.25mL of Level7	10mL
Level 5**	500μg/L	50μg/mL	1.25mL of Stock	50mL
Level 6	1000μg/L	100μg/mL	0.5mL of Stock	10mL
Level 7	2000μg/L	200μg/mL	1mL of stock	10mL

** CCV Level

8.4.2 PEST/PCB Surrogate Spike Solution: TMX and DCB obtained from All samples, blank and matrix spike are spiked with 1mL of the surrogate solution prior to extraction to monitor efficiency of sample extraction, chromatographic and calibration system. This amount may be adjusted to meet project specific concentrations, as needed.

8.4.3 **PCB Aroclors LCS/LCSD/MS/MSD:** Aroclor 1660 commercially obtained from Accustandard at the concentration of 10,000ug/L in Acetone. To prepare:

Spiking solution add 1mL of each primary solution at 1000μg/mL to a 100mL volumetric flask and bring it to volume with Acetone.

1mL is spiked into each QC and field QC sample. This amount may be adjusted to meet project specific concentrations, as needed.

8.4.4 **Independent Verification Standard (ICV) for Aroclors:** This is a source separate from the calibration curve containing one of the nine Aroclors, obtained from Ultra or Restek. Prepare from Stock Solution by diluting in hexane to the concentration of 500ug/L

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Quality Control (QC) samples are necessary to monitor both the sample extraction and instrument analysis procedures. The Quality Control samples described below are considered the method defaults, and are the minimum requirements, except where noted. Client and Project specific Data Quality Objectives (DQOs) supersede the requirements in this section where applicable. Client or Project specified DQOs shall be included, or referenced, in the final report to the client.

9.1 Blank(s)

9.1.1 A method blank must be extracted (spiked with surrogates and/or internal standards) and analyzed once per every 20 samples or per extraction batch, whichever is more frequent. The analysis of the method blank extract will demonstrate the background contamination.

9.1.2 An *acceptable* method blank should not contain any individual compound at the concentration of reporting limit, or above. All efforts must be made to identify and eliminate the source of contamination. The presence of analytes at concentrations at or above the reporting limit will warrant application of a “B” qualifier to that target compound(s) on all associated report forms, and perhaps re-extraction of all associated samples. The results are qualified with a “B” for any associated sample concentrations that are less then 10x the blank concentration for the analyte. Surrogate and internal standard recoveries must meet the QC limits for the method blank, see Section 12. Re-extraction must be initiated immediately so that the minimum of time is wasted before re-extraction can occur - if at all possible, this re-extraction should take place within holding time. Re-extraction *corrective action* that would exceed the sample holding time criteria should be discussed with the client, Laboratory Director, QA Manager, and/or Department Manager prior to implementation. Exceptions may be made with approval of the Department Manager if the samples associated with an out of control method blank are non-detect for the affected compound(s) or if the concentrations of the affected compound(s) are greater than 5x the blank level in the samples. In such cases, the sample results are accepted without corrective action for the high method blank result. The client must be notified, via the project narrative, of any method blank non-compliance associated with the sample results. Since air PUF samples cannot be re-extracted, re-analysis for confirmation of any issues must be performed. If similar results are observed, the data is reported with a narrative note discussing the non-compliance.

9.2 Laboratory Control Sample / Laboratory Control Sample (LCS / LCSD)

9.2.1 The LCS/ LCSD are extracted along with the samples as a measure of precision and accuracy. At a minimum, one LCS/LCSD sample set must be analyzed at a frequency of once per 20 samples, or per analytical batch, whichever is more frequent. The LCS contains Aroclor 1660.

9.2.2 The acceptable recovery QC limits are found in Section 12 for an aqueous, solid, and tissue LCS.

9.2.3 Acceptable relative percent difference (RPD) for LCS/LCSD analysis is ± 50 % for both aqueous and solid matrices. See section 12 for QA acceptance criteria. Calculate RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

9.2.4 Corrective Action: Repeat analysis or check to see if an analytical error has occurred. If the LCS recovery is still out of control, re-extract and re-analyze the LCS and all samples associated with that LCS/LCSD. If the LCS/LCSD do not meet quality control criteria for recovery of some of the spike and surrogate compounds, the results for the other samples and quality control samples within the batch must be evaluated to determine if this is an isolated problem for the LCS/LCSD and whether the data should be reported with the affected LCS/LCSD. If the recoveries for all components in the LCS/LCSD are outside of criteria, the entire analytical batch should be re-extracted and re-analyzed. Exceptions may be made with approval of the Department Manager if the samples associated with the out of control LCS/LCSD are also associated with a matrix spike and matrix spike duplicate that is in control which demonstrates an isolated problem pertaining to the LCS/LCSD only. An explanation of this out of control LCS/LCSD recovery must be included in the project narrative to the client and the sample data reported with the acceptable MS/MSD results as batch QC. Since air PUF samples cannot be re-extracted, re-analysis for confirmation of any issues must be performed. If similar results are observed, the data is reported with a narrative note discussing the non-compliance.

9.3 Initial Calibration Verification (ICV)

Refer to section 10.2

9.4 Continuing Calibration Verification (CCV)

Refer to section 10.4

9.5 Matrix Spike / Matrix Spike Duplicate (MS / MSD)

9.5.1 MS/MSD analyses are performed once per 20 samples (5% frequency) only per client request. It is preferable to extract samples that have been selected specifically by the client. If none have been assigned then the laboratory analyst must choose a representative sample for each type of matrix prepared. The MS/MSD must be matrix specific. Aqueous and air PUF MS/MSD cannot be done unless client supplies sufficient sample. These samples are also spiked with extraction surrogate. MS/MSD are spiked with the LCS spiking solution at the same level as LCS/LCSD.

9.5.2 The acceptable recovery and RPD QC limits are found in Section 12 for an aqueous, solid, tissue and product MS/MSDs.

9.5.3 Acceptable relative percent difference (RPD) for matrix spike analysis is ± 50 % for both aqueous and solid matrices. Calculate RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

9.5.4 Corrective Action: The recovery of the matrix spikes are evaluated relative to the what was in the unspiked sample to indicate how well the methods worked on extraction of the analytes of interest from the sample matrix. The results for the unspiked and MS/MSD samples need to be compared carefully to determine how well the spiking study worked and if there was any indication of matrix-related problems that might affect the analytical batch. Re-extraction of the sample based on the MS results is rare. Repeat analysis or check to see if an analytical error has occurred. If the % recovery or %RPD still exceeds the control limits and the associated LCS is within control, include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the affected results as evidenced by the matrix spike and matrix spike duplicate exceedance. Since air PUF samples cannot be re-extracted, re-analysis for confirmation of any issues must be performed. If similar results are observed, the data is reported with a narrative note discussing the non-compliance.

9.6 Laboratory Duplicate

9.6.1 Laboratory matrix or sample duplicates are analyzed if requested by the client. The client must supply sufficient additional sample volume for this duplicate. Duplicates are used to evaluate the precision of the method. The QC limit is 50% RPD for target compounds found above 5 times the reporting limit.

9.6.2 Corrective Action: If the %RPD exceeds the 50% control limit and the associated MS/MSD %RPD is within 50%, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results isolated to this sample, as evidenced by the matrix duplicate exceedance and the MS/MSD acceptance. If both the sample/duplicate and the MS/MSD exceed the control limits, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results as evidenced by the sample/duplicate and the MS/MSD exceedences. Since air PUF samples cannot be re-extracted, re-analysis for confirmation of any issues must be performed. If similar results are observed, the data is reported with a narrative note discussing the non-compliance.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

9.7.1.1 Surrogates are monitored for recovery for all matrices. Two surrogates are used, one that elutes at the beginning and one at the end of GC run: TMX/DCB. Every sample, blank, and quality control sample (LCS, MS, MSD, Dup) must be spiked with the surrogates prior to extraction. The recovery limits are found in Section 12.

9.7.1.2 Corrective Action: Check to see if an analytical, spiking, or dilution error occurred and re-calculate. If only one surrogate falls below the recovery limit and all QC samples have acceptable recovery indicating a matrix effect isolated to the sample, the exceedance is noted, with approval of the Section Supervisor, and the results are reported to the client with a notation in the case narrative. If all surrogates are recovered below the limit or there are outliers in any QC, re-extract the sample. If re-extraction occurred beyond the holding time and the re-extract surrogates are within the QC limits, report the re-extract results along with the original results. If the surrogates are recovered below the limit in the re-extract, this confirms suspected matrix interference on the surrogates, and only the original analysis needs to be reported unless a specific QAPP requires otherwise. If the chromatogram shows obvious matrix interference, no re-analysis or re-extraction is necessary. *This decision must be made with approval of the Section Supervisor and/or Project Manager.* Surrogate outliers and sample re-extracts must be noted in the case narrative to the client.

9.7.2 Internal Standards

9.7.2.1 Internal Standards will be evaluated for relative retention time drift and relative area counts compared to the associated opening CCV. Internal standards are added to every field sample, QC sample, standard, and method blank. The internal standard retention times for associated samples should be ± 0.05 minutes relative to the retention times from the associated opening CCV. The area counts for the internal standards in the associated samples should be +/-50% of the area of the internal standard calculated during initial calibration. The daily opening CCV IS areas are also compared to the ICAL.

9.7.2.2 Corrective Action: Check to see if an analytical, dilution or spiking error occurred. If the chromatogram shows obvious matrix interference, no re-analysis is necessary. *This decision must be made with approval of the Department Manager.* Note the exceedance in the case narrative to the client. If no obvious interference is present, re-analyze the extract. If internal standards are now within the acceptance limits, report only the re-analysis, as long as the re-analysis occurred within the 40-day analytical hold time. If the re-analysis occurred outside of the 40-day analytical hold time, both the original and re-analysis must be reported. If the internal standards again are outside the acceptance limits, after re-analysis, either within or outside of the 40-day hold time, report only the original analysis, and include a narrative to the client that the suspected matrix interference on the internal standards was confirmed by sample re-analysis. If specified by the client or project data quality objectives (DQOs) or other specifications, both results of both analysis might have to be reported.

9.7.3 Standard Reference Materials (SRMs)

9.7.3.1 Standard reference materials (SRMs) are available from the National Institute of Standards and Technology (NIST) and are extracted and analyzed with samples only on a project specific basis. For sediment and tissue sample batches, a certified SRM will be analyzed at the rate of one per batch of up to 20 samples per matrix. These are not used as controls, but to evaluate potential matrix effects in associated samples for the target compounds being evaluated.

9.7.3.2 Measured results will be compared against certified values for those target analytes that are certified in these SRMs as follows. Acceptance criteria for SRM analysis will vary from project to project depending upon client data quality objectives (DQOs). Generally, ± 50% difference (%D) based on the true certified values of the target compounds of interest, or 40% - 140% recovery, serve as advisory acceptance criteria.

9.7.3.3 Corrective Action: Repeat analysis and/or check to see if an analytical error has occurred. If the % recovery or %D still exceeds the control limits and the associated LCS/LCSD and/or MS/MSD are within control, include a project narrative with the results to client noting that the observed recovered of the SRM are isolated to this sample as evidenced by the LCS/LCSD and/or MS/MSD acceptance. Individual analyte exceedances will be narrated and may be used to assess potential interferences or system bias for individual analytes.

9.8 Method Sequence

9.8.1 An example calibration and sample sequence for 8082 Aroclor analysis listed below. Calibration points, ICV, CCV identifiers and/or nomenclature can be associated according to date/instrument or a specifically generated WG (workgroup) reference number assigned by the company LIMS system. Laboratory standard identification should be entered under the misc information section of each datafile for traceability of all standards utilized.

9.8.2 Listed below is an example of a calibration whereas seven calibration points were analyzed on 3/6/18. “I6” = Instrument ECD6. “CQ6” = ICV ECD6

9.8.3 Example Calibration Sequence

1. Prime and/or Hexane
2. I603161801 (calibration standard level 1 1660 + surrogates)
3. I603161802 (calibration standard level 2 1660 + surrogates)
4. I603161803 (calibration standard level 3 1660 + surrogates)
5. I603161804 (calibration standard level 4 1660 + surrogates)
6. I603161805 (calibration standard level 5 1660 + surrogates)
7. I603161806 (calibration standard level 6 1660 + surrogates)
8. I603161807 (calibration standard level 7 1660 + surrogates)
9. Hexane
10. CQ603161801 (Initial Calibration Verification Standard 1660)
11. Hexane
12. I603161808 – 2162
13. CQ603161802 – 2162
14. I603161809 – 3268
15. CQ603161803 – 3268
16. I6031618010 – 1242
17. CQ603161804 – 1242
18. I603161811 – 1248
19. CQ603161805 – 1248
20. I603161812 – 1254
21. CQ603161806 – 1254

NOTE: If multiple calibration mixtures are analyzed, it is acceptable to analyze appropriate ICVs after all calibration standards have been injected.

9.8.4 Example environmental sample sequence

An example sequence for 8082 Aroclor analysis listed below. CCV identifiers and/or nomenclature can be associated according to date/instrument or a specifically generated WG (workgroup) reference number assigned by the company LIMS system.

1.	Prime and/or Hexane	
2.	C4120401STD2162	AR1221/1262 Level 5 @ 500ug/L *
3.	C4120401STD3268	AR12321268 Level 5 @ 500ug/L *
4.	C4120401STD1242	AR1242 Level 5 @ 500ug/L *
5.	C4120401STD1248	AR1248 Level 5 @ 500ug/L *
6.	C4120401STD1254	AR1254 Level 5 @ 500ug/L *
7.	C4120401STD1660	AR1660 Level 5 @ 500ug/L
8.	Method Blank	
9.	Method LCS	
10.	Method LCSD	
11.	Up to 7 client sample injections	
12.	Repeat injections 1-11 as needed	

10. Procedure

10.1 Equipment Set-up

10.1.1 Prior to performing GC/ECD analysis, the operator must read and become familiar with the operating procedure guidelines specified in the instrument operating manuals. The analyst must be trained and familiarized with the instrument software provided by the manufacturer. The instrument must be set up with the proper operating parameters (data acquisition and processing) and conditions described in the operating manual. The criteria for the analytical calibration ranges and method and instrument detection limits must be established and documented prior to initial calibration. All analytical equipment is traceable to NIST via internal and external calibration checks and *Certificates of Analysis* that are received with the calibration standards.

10.1.2 Establish daily retention time windows as the retention time of the component in the opening verification standard ± 0.05. Retention time windows are calculated each time a new GC column is installed. It has been found that this window works well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results. The window listed above is used as guidance; however the experience of the analyst weighs heavily in the interpretation of the chromatograms.

10.1.3 GC Instrument Conditions:

The dual-column / dual-detector approach involves the use of the columns listed in section 7.2.1 the columns are connected to an injection tee or dual injection GC, and separate electron capture detectors. Alpha typical GC conditions are listed below, but may be altered as long as method performance criteria are met. Inject an aliquot of 1ul into the capillary column of the gas chromatograph at the following conditions.

Temperature1: 120 ^{0}C	Injector temperature: 250^{0}C
Time1: 0 minutes	Injector mode: Pulsed Split

Ramp1: 45^{0}C/minute	1.4:1 split, 0.20 min pulse
Temperature2: 200^{0}C	Injector Flow: 5.7 ml/min split flow
Time2: 0 minutes	Detector temperature: 350^{0}C
Ramp2: 15^{0}C/minute	Carrier gas: Helium
Temperature3: 230^{0}C	Carrier flow: 20ml/min
Time3: 0 minutes	Carrier mode: Constant flow
Ramp3: 30^{0}C/minute	Makeup gas: Nitrogen
Final temperature 330^{0}C	Total detector flow: 55ml/min
Final time: 2 minutes	Injection Volume: 1 µL

10.2 Initial Calibration

10.2.1 Prepare calibration standards using the standards listed in Section 8.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in section 10.1.3.

10.2.3 A 1µL injection volume of each calibration standard is typically used. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume must be used for all standards and samples.

10.2.4 Column adsorption may be a problem when the GC has not been used for a day or more or after system maintenance. The GC column may be primed (or deactivated) by injecting a PCB standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification. Alternately, the system may be primed by baking at the final analytical temperature for approximately 30 minutes. Several analytes may be observed in the injection just following system priming. Always run an instrument blank after system priming.

10.2.5 Calibration Factor: Internal standard calibration techniques are employed in this method. In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formula listed in Alpha's Quality Manual.

10.2.6 Initial Calibration Criteria If the RSD for an analyte is < 20%, then the response of the instrument for this compound is considered linear over the range and the mean calibration factor can be used to quantitate sample results. If the RSD for any analyte is > 20%, then linearity through the origin cannot be assumed. The mean response factor cannot be used for quantitation. An alternative calculation may be done by the use of linearity as long as the correlation coefficient is >0.995. If both of these quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated and a new calibration curve must be analyzed.

10.2.7 Initial Calibration Verification An initial calibration verification standard must be run immediately after each initial calibration, near the midpoint of the curve. The standard must be prepared using a second source that is different than the source used for the initial calibration. (Standards listed in Section 8). The %D has to be within 20%. The retention time window used for the identification of target analytes is ± 0.07 minutes.

These criteria have been adopted from the EPA CLP Statement of Work (OLM04.2). It has been found that these limits work well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results.

10.2.8 The windows listed above are used as guidance; however the experience of the analyst weighs heavily in the interpretation of the chromatograms. For example, it has been observed that certain oil matrices can cause the retention times to shift more dramatically.

10.2.9 The Relative Error (%RE) is evaluated for the midpoint of the calibration and should be 50-150%.

10.3 Equipment Operation and Sample Processing

10.3.1 The determination of PCB Aroclors is accomplished by comparing the sample chromatogram to that of the most similar Aroclor standard. The aroclor pattern must be present on both columns to confirm the Aroclor identification. The use of PCB overlays is extremely helpful, either by using hardcopies of chromatograms or by utilizing the Enviroquant software. A choice must be made as to which Aroclor is most similar and whether that standard is truly representative of the PCB in the sample. Both retention time and pattern are important when determining PCBs in a sample. Samples that contained weathered PCB present special analytical challenges. Weathering could alter the Aroclor pattern to the extent that different peaks have to be selected for quantitation. Samples that contained more than one Aroclor present similar problems. For these samples, the Analyst may have to consider selecting the earlier eluting peaks for the lower boiling Aroclor and selecting the later eluting peaks for the higher boiling Aroclors to minimize overlapping peaks. Minimum of 3 peaks must be chosen for each Aroclor. If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be needed. If instrument problems are suspected, rerun the extract on another instrument to determine if the problem results from analytical hardware or the sample matrix. The laboratory must report the higher of the two columns' results unless obvious interference is present on one of the columns.

10.3.2 Verify calibration each 12-hours shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than once every twenty injections. A bracketing CCV is not required with the use of internal standard calibration (Method 8082A 11.6.8) with the exception of samples ran under CT RCP method. For Aroclor analysis, the calibration verification standard should be a mixture of Aroclor 1016 and 1260.

10.3.3 The response factor (for internal standard compounds) for each analyte to be quantitated must not exceed a ± 20% difference when compared to the initial calibration curve (+ 15% for CT RCP). The Target data processing software automatically calculates the %D for all analytes according to the formulae in Alpha's Quality Manual. A retention time shift >30 seconds for the internal standard necessitates reanalysis of all affected samples.

10.3.4 Routine preventive maintenance is performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and replacement of injector seals. Other Maintenance: ECD detectors may become contaminated, requiring bake out at elevated temperatures, (no greater than 375°C) or repair by the manufacturer.

10.3.5 If the on-column concentration of any compound exceeds the calibration range of 2000ug/L, the sample must be diluted, re-spiked with the appropriate amount of internal standard and re-analyzed.

10.4 Continuing Calibration

10.4.1 Verify calibration each 12-hours shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than once every twenty injections. A bracketing CCV is not required with the use of internal standard calibration (Method 8082A 11.6.8) with the exception of samples ran under CT RCP method. For Aroclor analysis, the calibration verification standard should be a mixture of Aroclor 1016 and 1260. The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition (Method 8082A 11.6.2)

10.4.2 The response factor (for internal standard compounds) for each analyte to be quantitated must not exceed a ± 20% difference when compared to the initial calibration curve (+ 15% for CT RCP). A retention time shift >30 seconds for the internal standard necessitates reanalysis of all affected samples.

10.5 Preventive Maintenance

10.5.1 Preventive maintenance may or may not include the following; replacing system uniliner, o-ring, merlin septum, MXTY connector or clipping a length of guard column.

10.5.2 Additionally, preventive or routine maintenance for GC/ECD systems may involve baking out the ECD detectors, replacing guard, RTX-5 or CLPII columns.

11. Data Evaluation, Calculations and Reporting

11.1 Quantitation of Aroclors

Quantitation is based on the use of a minimum of 3 of the major peaks present in the analyte, although the use of 5 of the major peaks is recommended. Each of these peaks is individually calibrated with a minimum of five calibration points based on average response factors. The %RSD must meet the criteria of <20% for the ICAL. The five major peaks are calculated as described below. After individual calculation meets criteria, the average of the peaks selected for quantitation is used to determine the final concentration.

11.2 Aqueous samples

$$\text{Concentration } (\mu g/L) = \frac{C \times DF \times Vf \times 1000}{Vo}$$

C = Extract concentration (µg/mL), NOTE: ng on column = ng/injection volume = ng/uL = ug/mL
DF = Dilution factor
Vf = Final extract volume (mL)
Vo = Sample volume (mL)

11.3 Soil/sediment samples

$$\text{Concentration } (\mu g/Kg, \text{ dry weight}) = \frac{C \times DF \times Vf \times 1000 \div \%S}{W\ (g)}$$

C = Extract concentration (µg/mL), NOTE: ng on column = ng/injection volume =

ng/uL = ug/mL
DF = Dilution factor
Vf = Final extract volume (mL)
W = Weight of the sample extracted (10g for high, 30g for low)
%S = Percent solids, as a decimal value

11.4 PUF samples

$$\text{Concentration (ng/cart)} = \frac{C \times DF \times Vf}{W}$$

C = Extract concentration (µg/L)
DF = Dilution factor
Vf = Final extract volume (mL)
W = Weight of the sample. for Air PUF, sample amount is 1

11.5 Reporting Results

After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and/or improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written into the LIMS by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

All PCB Aroclor results are reportable without qualification if analytical holding times are met, preservation requirements (including cooler temperatures) are met, and all QC criteria defined in the table below are met. If any of the below QC parameters are not met, all associated samples must be evaluated for re-analysis.

QC Parameter	Acceptance Criteria
Initial Calibration Curve	20% RSD for all target analytes Correlation coefficient must be $\geq$ 0.99 – for linear model The COD or r^2 must be $\geq$ 0.99 – for non-linear model
Independent Check Verification	+/- 20% recovery of the true values, For Aroclors the Average of the 5 representative peaks are calculated to get the %D.

Continuing Calibration Verification	Analyzed every 12hr or at the minimum of every 20 samples, 20% D for all target analytes. For Aroclors the Average of the 5 representative peaks are calculated to get the %D.
Method Blank	No analyte at or above the reporting limit, "B" qualify analyte if detected < 10x the blank contamination
Laboratory Control Sample	Soil/Tissue/Aqueous/PUF: 40-140%; 50% RPD
Matrix Spike / Matrix Spike Duplicate	Same as for LCS; 50% RPD between the duplicates.
Sample / Sample Duplicate	50% RPD between the duplicates.
Surrogates	Soil: 30-150%; Aqueous: 30-150%; PUF: 60-120%
Internal Standards	50% - 200% of the daily CCV area for the Internal Standards
SRM	Same as for LCS, 40% - 140% recovery

Any deviations and observations made about the analyses must be documented in the instrument logbook and or project narrative. If a problem arises during analysis, document the problem and initiate corrective action. If there is a problem with a sample analysis which indicates that re-extraction should be performed, and if there is no additional sample available for re-extraction, the Project Manager needs to be informed immediately so that the client can be involved in the corrective action process. Re-extraction of a sample should be done within holding time if at all possible.

Evaluation of a sample result often requires knowledge about the results of all samples within a job. Therefore, use all of the data, if possible, from a job to judge whether or not corrective actions are needed. For example, if a sample is run and the surrogates are low, one might request a re-extraction immediately only to find that a MS/MSD were also done on this sample, with low surrogate and MS recoveries, confirming a matrix effect. In this case, a re-extraction is not required and the results would be reported with a narration explaining the evidence of a matrix effect on the low surrogate recoveries. The characteristics of the overall job may be invaluable in deciding how to report the data to the client.

If non-compliant PCB Aroclor results are to be reported, the Department Manager and/or the Laboratory Director, and the QA Manager must approve the reporting of these results. The laboratory Project Manager shall be notified, and may choose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan SOP/2124

DL/LOD/LOQ Generation SOP/1732

IDC/DOC Generation SOP/1739

Waste Management and Disposal SOP/1797

Laboratory Glassware cleaning SOP/1753

Reagent, Solvent, and Standard Control SOP/1816

Soxhlet Extraction of PUF cartridges SOP/2174

16. Attachments

Table 1: Reporting Limits (RLs) and CAS Registry Numbers

Table 2: LCS/LCSD, MS/MSD and Precision and Accuracy Evaluation Criteria

Table 3: Surrogate Recovery Evaluation Criteria

Table I

Reporting Limits (RLs) and CAS Registry Numbers for PCB Aroclors by GC/ECD

Compound	CAS Registry	Aqueous in µg/L	Solid in µg/Kg	PUF in ng/PUF
		RLs	RLs	RLs
Aroclor 1016	12674-11-2	0.02	2.5, 15	30

Compound	CAS Registry	Aqueous in µg/L	Solid in µg/Kg	PUF in ng/PUF
Aroclor 1221	11104-28-2	0.02	2.5, 15	100
Aroclor 1232	11141-16-5	0.02	2.5, 15	30
Aroclor 1242	53469-21-9	0.02	2.5, 15	20
Aroclor 1248	12672-29-6	0.02	2.5, 15	20
Aroclor 1254	11097-69-1	0.02	2.5, 15	20
Aroclor 1260	11096-82-5	0.02	2.5, 15	20
Aroclor 1262	11100-14-4	0.02	2.5, 15	20
Aroclor 1268	37324-23-5	0.02	2.5, 15	20

Table II

LCS/LCSD, MS/MSD and Precision and Accuracy Evaluation Criteria

Spiked Component	Aqueous/Solid/Tissue/PUF (% recovery)	RPD
AR1016	40-140	50
AR1260	40-140	50

Table III

Surrogate Recovery Evaluation Criteria

Surrogate	Aqueous/Solid/Tissue (% recovery)	PUF (% recovery)
2,4,5,6-tetrachloro-m-ylene(TMX)	30-150	60-120
Decachlorobiphenyl (DCB)	30-150	60-120

NOTE: Depending on project specific requirements, MS/LCS/Surrogate recoveries may be validated against a specified project QAPP, 8082 SW-846, TO-10A, CLP OLM04.2, DoD QSM 5.3, or in-house determined limits.

Total Petroleum and Saturated Hydrocarbons by Gas Chromatography/Flame Ionization Detector

References: USEPA, Method **8015D** – "Nonhalogenated Organics Using GC/FID", in Test Methods for Evaluating Solid Waste, SW-846, Revision 4, June 2003.

1. Scope and Application

Matrices: This standard operating procedure (SOP) describes a method for quantification and qualitative fingerprinting of petroleum products in extracts of water, soil, sediment and petroleum products. Extractable petroleum hydrocarbon in petroleum products such as kerosene, aviation fuels, diesel and heating oils may be analyzed by this method, but products with primarily volatile constituents such as gasoline range organic compounds are not amenable to this method. To determine volatile organic material, see Alpha Analytical Lab SOP 2255, *PIANO Volatiles by GC/MS*.

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the quantification and qualitative fingerprinting of petroleum products. If quantifying petroleum ranges using the individual n-alkanes, the reporting limit for the petroleum ranges is calculated by taking the sample concentration factor and multiplying it by the summation of the number of calibrated analyte(s) that elute in the range. The quantifying total petroleum ranges are generally C_9-C_{44}. Client specific ranges, as requested, are accommodated (*i.e.*, C_{10}-C_{28}, C_9-C_{36}, and C_9-C_{40}). The qualitative identification is dependent on the product pattern, how weathered it may be, and how much is present. This is the subjective aspect of the analysis that requires an experienced analyst. Care should be taken to ensure consistency in petroleum identification. Pattern books generated from the analysis of "typical" petroleum products (Section 8.12), analyzed under the same conditions as the samples may be used for reference.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC-FID and in the interpretation of GC-FID data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Please refer to the appropriate Alpha Analytical Lab SOPs for extraction methods and sample preparation information:

- *Method 3510C – Extraction of Water Samples by Separatory Funnel* (2165).
- *Shaker Table Extraction* (2261).
- *Tissue Preparation and Homogenization* (2166) and *Tissue Extraction* (2264)
- *Organic Waste Dilution* (2265)
- *Gravimetric Determination* (2263)

Analytes are introduced into the GC/FID by injecting a known volume of the calibration standards, quality control samples, and sample extracts into the GC equipped with a narrow-bore capillary column. The GC column is temperature programmed to separate the analytes, which are then detected with a flame ionization detector. Identification of target analytes is accomplished by comparing their retention time with the retention time of the calibration standards. Concentrations are determined using mean relative response factors from a multi-level calibration curve. Response factors for target analytes and surrogate compounds are determined relative to the internal standards. Typically, Petroleum Fuel/Hydrocarbon Ranges (TPH ranges listed by example in Section 1.0) are assigned the response factors (Rf) of the average of the calibrated n-alkanes and isoprenoids. The isoprenoids and the alkane n-Nonatriacontane (C39) are assigned the response factors (Rf) of the calibrated alkane eluting immediately after (for example the isoprenoid 1380 is assigned the response factor (Rf) of n-Tetradecane (C14). If defined by client or project data quality objectives (DQOs) or other specifications, sample concentrations may be measured by calibrating with a Diesel fuel or another site specific NAPL, or product. Sample quantification in these cases would be from the average Rf obtained from a multi-level calibration curve for the fuel product.

2.1 Method Modifications from Reference

The continuing calibration verification %D for each calibrated compound must be below 25%, with no more than 10% of all compounds greater than 25% but less than 35%. Each CCV must be analyzed within 24 hours of the previous CCV.

Matrix spike and duplicate samples are analyzed only if requested by the client.

3. Reporting Limits

Reporting limits for the individual n-alkane and isoprenoid are 200 mg/Kg (ppm) for petroleum products, 0.0667 mg/Kg (ppm) for soil/sediment samples, 0.267 mg/Kg (ppm) for tissue samples, and 0.001 mg/L (ppm) for water samples. The associated reporting limits for TPH (C9-C44) are as follows: 6600 mg/kg for petroleum products, 2.2 mg/kg for soil/sediment samples, 6.6 mg/kg for tissue samples, and 0.033 mg/L for water samples.

4. Interferences

4.1 Contaminants in solvents, reagents, glassware, and other sample processing hardware may cause interferences that lead to discrete artifacts and/or elevated baselines in the chromatogram. Demonstrate that all of these materials are free from interferences under the conditions of the preparation and analysis by extracting and analyzing a laboratory method blank with each batch of up to 20 samples.

4.2 Emulsions can form during the extraction especially in aqueous samples with high concentrations of petroleum contamination. This may require mechanical means to disperse such as glass stirring rods or centrifugation. Alternatively, filtration of emulsions through pre-dried sodium sulfate can be employed. Additional filtration through sodium sulfate after emulsion reduction is required to ensure adequate sample drying has been achieved.

4.3 The injector system of the GC can become contaminated with non-volatile components from prepared samples, decreasing the resolution and responsiveness of the system. High molecular weight polyaromatic hydrocarbons (PAH) in the calibration mix can act as indicators of chromatographic problems. C_{40} response decreases with increased injector contamination and is a good indicator compound. Maintenance of the analytical system should be performed when system performance degrades.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples: Collected in pre-cleaned 1-L amber glass bottles.

Soil/sediment samples: Typically collected in a pre-cleaned 250ml soil jar with a teflon lined screw cap.

6.2 Sample Preservation

Aqueous samples: Stored without preservative and at 4°C.

Soil/sediment samples: Preserved by storing at 4°C.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Aqueous samples: Extraction is performed within 7 days. Sample extracts are analyzed within 40 days of extraction.

Soil/sediment samples: Extraction is performed within 14 days of collection; extract is analyzed within 40 days of extraction.

There is no extraction holding time applied to petroleum product samples. If sediment samples are frozen, this suspends holding time until removal from the freezer. All extracts must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph - The instrumentation includes a temperature-programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port is designed for splitless injection onto a capillary column. The injection port includes a silanized glass liner containing a plug of silanized glass wool to reduce high-molecular-weight mass discrimination. The model is HP6890 or equivalent. The injector port will require maintenance on an as needed basis if degradation or contamination is apparent. Please refer to the front of the *Instrument Maintenance Logbook*, which outlines the routine maintenance procedures.

7.2 Column – Restek 60-m x 0.25 mm ID, 0.25um film thickness, fused-silica capillary column with RTX-5 bonded phase, or equivalent.

7.3 Flame Ionization Detector (FID) – The FID must operate at 20Hz data collection rate and have the attenuation optimized for signal to noise response. The GC capillary column is fed directly into the flame ionization detector. The FID will require cleaning and/or jet replacement on an as needed basis. Please refer to the instrument hardware manual for detailed procedures, located in the laboratory next to the instrument.

7.4 Auto sampler - Adapted onto the Gas Chromatograph. The model is HP 6890 series autosampler with a GC autosampler controller, or equivalent.

7.5 Computer - with Windows XP operating software utilizing HP Enviroquant G1701DA Version D.01.02 software or a more current release; Audit Trail: audit.txt function is used for audit trail purposes.

7.6 Hydrogen – Ultra high purity grade (99.9999% pure).

7.7 Air – Ultra Zero high purity grade (99.9999% pure).

7.8 Helium - Ultra Zero high purity grade (99.9999% pure).

7.9 Glassware - Assorted Class-A volumetric flasks consisting of 5mL through 200mL flasks with ground-glass stoppers for preparing reagents, standards, and measuring sample volumes for sample requiring dilution beyond 1:100.

7.10 Pipettes: Disposable Pasteur brand, or equivalent.

7.11 Cahn balance: Capable of accurate measurement to the nearest 0.001 mg.

7.12 Analytical balance: Capable of accurate measurement to the nearest 0.0001 g.

7.13 Top-loading analytical balance: Capable of accurate measurement to the nearest 0.1g.

7.14 Micro syringes - 10, 25, 50, 100, 250, 500, and 1000 μL.

7.15 VOA vials: Non-preserved, certified, pre-cleaned, 40 mL, with Teflon-lined screw caps.

7.16 Vials, 2 mL: with Teflon-lined crimp caps for standard solutions.

7.17 Spatula - Stainless steel.

8. Reagents and Standards

Use reagent grade chemicals for all reagents. Deionized (DI) water is ASTM Type II laboratory reagent grade water.

All stock standard solutions are purchased from commercial vendors as ampulated certified solutions or prepared in house from neat materials. Standards are stored according to the vendor recommendations. When an ampulated stock solution is opened, or prepared fresh from neat materials, it must be transferred into a PTFE-lined screw capped vial. Any extraction working standards are stored at -10 to -20°C, away from light, when not in use. All analytical working standard solutions are stored at room temperature. They are discarded after 1 year unless the vendor expiration date states otherwise or if breakdown is observed.

8.1 Methylene Chloride: ACS approved, Pesticide grade, see Alpha Analytical Lab SOP *Reagent, Solvent and Standard Control* (1816) for additional details regarding solvent purity.

8.2 Acetone: ACS approved, Pesticide grade, see Alpha Analytical Lab SOP *Reagent, Solvent and Standard Control* (1816) for additional details regarding solvent purity.

8.3 Hexane: ACS approved, Pesticide grade, see Alpha Analytical Lab SOP *Reagent, Solvent and Standard Control* (1816) for additional details regarding solvent purity.

8.4 Multi State Hydrocarbon Standard: Obtained from Accustandard at 500ug/mL

8.5 Internal Standard Calibration Stock: 5-alpha-androstane custom standard from Accustandard at 10,000ug/mL. Prepare a 1:20 dilution (2500ul into 50mL Methylene Chloride) for a 500ug/mL solution.

8.6 Surrogate Stock: Ortho-terphenyl custom from Accustandard and n-Tetracosane-d50 custom from Chemservice both at 20,000ug/mL. Prepare a 1:20 combined stock solution (5mL of each surrogate standard at 20,000ug/mL into 100mL Methylene Chloride) for a 1,000ug/mL solution.

8.7 Surrogate Spiking Solution (SIS): Prepare a solution by taking 2.5mL of each surrogate into 100mL of Methylene Chloride for a 500ug/mL spiking solution. The solution must be assayed for use by analysis before release to the preparation lab. All compounds must be within 10% of their true value. A manager's approval is needed for all surrogate spiking solutions before use. 100ul is spiked into each QC and field sample. This amount may be adjusted to meet project specific concentrations, as needed.

8.8 Internal Standard (RIS): Prepare a solution by taking 10mL of 5-alpha-androstane into 200mL of Methylene Chloride for a 500ug/mL spiking solution. 100ul is spiked into each QC sample and field sample.

8.9 Laboratory Control Sample, Matrix Spike, and Matrix Spike Duplicate (LCS/MS/MSD): A solution of 14 alkanes from Ultra Scientific, or equivalent, at 1000ug/mL. This is a source separate from the calibration curve. Prepare a spike mix by adding 10mL of solution to 50mL of Methylene Chloride for a 200ug/mL LCS/MS/MSD spiking solution. The solution must be assayed for use by analysis before release to the preparation lab. All compounds must be within 20% of their true value. 100ul is spiked into each QC and field QC sample. This amount or component type may be adjusted to meet project specific concentrations, as needed.

8.10 Alaska North Slope Crude Oil: Neat. Weigh approximately 1.0g into 90mL of Methylene Chloride. To the 90mL add 10mL of the internal standard (RIS) for a stock solution of 50ug/mL.

8.11 Independent Calibration Verification Standard (ICV): A solution of all calibrated alkanes that are a separate lot from the calibration mixture can be obtained from Accustandard. The solution is prepared to the exact specs of the Continuing Calibration Verification standard.

8.12 Petroleum Hydrocarbon Reference Standards: These standards are used for a chromatographic library and qualitative identification. If requested, any of these petroleum standards may be used for instrument calibration or LCS Solution. All standards are diluted to be at a concentration of 5mg/mL for analysis when used as part of the Reference Library. The library should include but not be limited to standards of PAH, gasoline, jet fuels, kerosene, fuel oils, paint thinner, asphalt, lubricating oils, creosote, coal tar, cutting oil and hydraulic oil. Because reference materials cannot always be obtained as certified materials, the source of the reference standards should be documented (*i.e*. Fuel Oil #2 obtained from a particular fuel oil company on a particular date). Several reference standards can be obtained from Accustandard's complete list of Total Petroleum Hydrocarbon Recognition Standards.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A method blank must be extracted (spiked with surrogates and internal standards) and analyzed once per every 20 samples or per extraction batch, whichever is more frequent.

Acceptance Criteria: Method Blanks should not contain any individual compound at or above the concentration of the reporting limit. If a blank does contain target compounds greater than the reporting limit, they should be less than 10% of any sample results for the same compound(s). Individual compounds may be detected in the blank below the reporting limit. Associated sample results are flagged with a “B” qualifier if the concentration of the analyte in the sample is less than 10x the concentration in the blank.

Corrective Action: If the blank does not meet the above criteria, all efforts must be made to identify and eliminate any source of contamination, and all samples associated with the contaminated blank should be re-extracted and reanalyzed. Exceptions may be made with the approval of the Department Manager. Any exceedances are noted in the case narrative.

9.2 Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)

The LCS spike is from a second source and can additionally be used to verify the accuracy of the standard curve. The LCS spiking solution is a mixture of 14 n-alkanes spread over the C_9 to C_{40} calibration range. The LCS and LCSD are extracted along with the samples as a measure of precision and accuracy. At a minimum, one LCS/LCSD must be analyzed at a frequency of once per 20 samples, or per analytical batch, whichever is more frequent.

Acceptance Criteria: All LCS compound recoveries must be between 50-130% of the true values. The acceptable recovery QC limits are found in Section 12 for aqueous, solid, tissue, and product LCSs.

Corrective Action: Repeat analysis once to see if an analytical error has occurred. If the LCS/ LCSD % RPD or % Recovery is still out of the control limit check the sample archive. Samples cannot be analyzed until an acceptable LCS is obtained. The samples associated to the LCS/ LCSD may need to be re-extracted. Exceptions may be made with approval of the Section Head if the samples associated with the out-of-control LCS/ LCSD are also associated with a MS/MSD pair that is in control. This is an acceptable measure of precision and accuracy of analytical procedures. An explanation of this out-of-control LCS/LCSD recovery must be included in the project narrative to the client and the sample data reported noting the acceptable MS/MSD results as the batch QC.

9.3 Initial Calibration Verification (ICV)

The analysis of an ICV standard must follow the initial calibration curve.

After final processing, calculate the percent recovery of each n-alkane and isoprenoid by using the following calculation:

% Recovery = Found Amount / True Value x 100

Acceptance Criteria: All recoveries must be +/- 20% of the true values.

Corrective Action: If the initial calibration fails, perform instrument maintenance and repeat.

9.4 Continuing Calibration Verification (CCV)

A continuing calibration verification (CCV) standard, at the concentration of a mid-level initial calibration standard, must be analyzed at the beginning and end of every analytical sequence, and every 24 hours within the sequence, to confirm instrument stability, via response factor, for each calibrated n-alkane.

Acceptance Criteria: Compare the CCV resulting response against the average response for the initial calibration for each calibrated n-alkane and isoprenoid. The %D for each calibrated compound must be below 25%, with no more than 10% of all compounds greater than 25% but less than 35%. If multiple CCVs are analyzed within an analytical sequence, each CCV must be analyzed within 24 hours of the previous CCV. Each CCV, including the ending CCV must meet the acceptance criteria. The acceptable QC limits are found in Section 12.

Corrective Action: If the CCV does not meet the acceptance criteria for each calibrated n-alkane and isoprenoid, perform instrument maintenance and repeat the continuing calibration. Re-analyze all affected samples. Reported results for the failing CCV may be "J" qualified. If the failure of the suspect response appears related to a loss in FID sensitivity, instrument maintenance and repeat analysis of the continuing calibration, and all affected samples, must be performed. Exceptions may be made with the approval of the Department Section Head. Any exceedances are noted in the case narrative.

9.5 Matrix Spike / Matrix Spike Duplicate (MS / MSD)

Matrix spike samples are analyzed only if requested by the client. Matrix duplicate analyses (as an MS/MSD pair) can be performed once per 20 samples (5% frequency) at the clients' request. It is preferable to extract samples that have been selected specifically by the client. If none have been assigned, then the laboratory analyst must choose a representative sample for each type of matrix prepared. Aqueous duplicates can only be done if the client supplies sufficient sample. These samples are also spiked with extraction surrogate.

Acceptance Criteria: All matrix spike compound recoveries must be between 50-150% of the true values. The acceptable recovery QC limits are found in Section 12 for an aqueous, solid, tissue, and product matrix spike analysis.

Acceptable relative percent difference (RPD) for matrix spike analysis is ± 30 % for both aqueous and solid matrices. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

Corrective Action: Repeat analysis once to see if an analytical error has occurred. If the % RPD or % Recovery still exceeds the control limits and the LCS/ LCSD is compliant; include a project narrative with the results to the client noting that there may be potential matrix effects on the precision of the affected results as evidenced by the MS/MSD recoveries or %RPD outside of QC limits.

9.6 Laboratory Duplicate

Acceptance Criteria: Laboratory matrix or sample duplicates are analyzed if requested by the client. The QC limit is 30% RPD for target compounds found above 5 times the reporting limit.

Corrective Action: If the %RPD exceeds the 30% control limit and the associated MS/MSD %RPD is within 30%, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results isolated to this sample, as evidenced by the matrix duplicate exceedence and the MS/MSD acceptance. If both the sample/duplicate and the MS/MSD exceed the control limits, include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the results as evidenced by the sample/duplicate and the MS/MSD exceedences.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

Acceptance Criteria: All surrogate recoveries must be between 50-130% of the true values. Surrogates are monitored for percent recovery for all matrices. The recovery limits are found in Section 12.

Corrective Action: If the surrogate does not fall within the QC limits, check to be sure there are no errors in peak integration, calculations, and surrogate solutions. If only one surrogate falls below the 50% recovery limit, the exceedence is noted, with approval of the Department Manager, and the results are reported to the client with a notation in the case narrative. If all surrogates are recovered below the 50% limit, re-extract the sample and report the re-extract results along with the original results, if re-extraction occurred beyond the holding time, and the re-extract surrogates are within the QC limits. If the surrogates are recovered below 50% in the re-extract, this confirms a suspected matrix interference on the surrogates, and only the original analysis needs to be reported. If the chromatogram shows obvious matrix interference, no re-analysis or re-extraction is necessary. *This decision must be made with approval of the* Department Manager. Surrogate outliers and sample re-extracts must be noted in the case narrative to the client.

9.7.2 Internal Standards

Acceptance Criteria: Internal standards are added to every field sample, QC sample, standard, and method blank. The acceptance limits are 50-200% of the internal standard response (or area) of the daily continuing calibration verification standard.

Corrective Action: Check to see if an analytical, dilution or spiking error occurred. If the chromatogram shows obvious matrix interference, no re-analysis is necessary. This decision must be made with approval of the Department Manager. Note the exceedence in the case narrative to the client. If no obvious interference is present, re-analyze the extract. If internal standards are now within the acceptance limits, report only the re-analysis, as long as the re-analysis occurred within the 40-day analytical hold time. If the re-analysis occurred outside of the 40-day analytical hold time, both the original and re-analysis must be reported. If the internal standards again are outside the acceptance limits, after re-analysis, either within or outside of the 40-day hold time, report only the original analysis, and include a narrative to the client that the suspected matrix interference on the internal standards was confirmed by sample re-analysis.

9.8 Method Sequence

Samples are prioritized for analysis by the Department Manager or Team Leader based on client due date and sample analytical hold time. Samples are retrieved from the sample storage refrigerator.

The sequence is prepared and run using the Enviroquant software. Printouts of all sequences are kept in a three-ring notebook for each instrument. The sequence printouts are used to document run sequences; notations are manually added of any reruns or dilutions that will need to be performed in the daily SHC continuing calibration verification packages.

- Instrument Primer
- Crude Oil Reference Standard
- Continuing Calibration Verification (CCV)
- Instrument Solvent Blanks
- Method Blank
- Laboratory control samples (LCS/LCSD)
- Samples (1-10)
- Continuing Calibration Verification (CCV)

10. Procedure

Prior to performing Total Petroleum and Saturated Hydrocarbon GC/FID analysis, the operator must read and become familiar with the operating procedure guidelines specified in the instrument operating manuals. The analyst must be trained and familiarized with the instrument software provided by the manufacturer. The instrument must be set up with the proper operating parameters (data acquisition and processing) and conditions described in the operating manual. The criteria for the analytical calibration ranges and method and instrument detection limits must be established and documented prior to initial calibration.

All samples and standard solutions must be allowed to warm to ambient temperature before analysis.

10.1 Equipment Set-up

10.1.1 GC Instrumental Conditions

Inject a 1uL aliquot of sample extract or standard solution into the capillary column of the gas chromatograph at the following conditions.

GC Parameter	Setting
Injector Temp:	280 °C
Initial Oven Temp:	40 °C
Initial Hold Time:	1 minute
Column Flow:	3.0 mL/ minute
Ramp Rate:	6 °C / minute
Final Temperature:	315 °C
Final Hold Time:	30 minutes
Total runtime:	76.83 minutes
Mode:	Pulsed Splitless
Pulse:	15psi for 0.5 minute
Purge:	20 mL / minute – on at 0.8 minutes
FID Temperature	325 °C

10.2 Initial Calibration

	Volume added			
Calibration Level	**Alkane Standard**	**Surrogate Stock**	**Internal Standard Stock**	**Final Volume in Methylene Chloride**
Level 1 -1ug/mL	20ul	10ul	1000ul	10mL
Level 2 -10ug/mL	200ul	100ul	1000ul	10mL
Level 3 -50ug/mL	5000ul	2500ul	5000ul	50mL
Level 4 -100ug/mL	2000ul	1000ul	1000ul	10mL
Level 5 -200ug/mL	4000ul	2000ul	1000ul	10mL
Level 6 -500ug/mL	NA	5000ul	1000ul	10mL
SHC - ICV	1000ul	500ul	1000ul	10mL

10.2.1 Before analysis of sample extracts, establish a multi-point response factor calibration curve showing the linear range of the analysis for all individual target analytes. Use standard concentrations of 1, 10, 50, 100, 200 and 500, ug/mL to construct the curve. See Section 8 and Table above for the preparation of the standard solutions for the initial calibration curve. Conversely, and if project specifications require, a minimum 5-point calibration curve may also be constructed using a specified fuel product of interest. The calibration range, and thus the reporting limit for this type of calibration, is several factors higher than for the individual component calibration. A Fuel Product calibration curve is typically 100ug/mL to 50,000ug/mL. Section 8.12 lists fuel products typically used.

10.2.2 Run a sequence with the initial calibration standards.

- Create a processing method from a previous method on that instrument. Set retention time windows using a mid level standard.
- Quantitate and QEDIT the initial calibration standards. Update the method with these standards.
- Additionally, the response for the petroleum ranges must be populated into the method using the Abacus CalTools macro. The average area of all individual components is used to calculate the Rf for the petroleum ranges. The ranges are typically specified by the client or project DQOs. **NOTE**: It should be noted that quantification of a Fuel Product Range using the Rf generated from individual n-alkanes can lead to a *20% - 30% low bias* of the reported TPH range.
- The response factors for reportable DRO, ORO and TPH ranges are defined by taking the average RSD of target peaks within the relative ranges retention time ranges. C10-C28 DRO, C28-C40 ORO and C9-C44 TPH.
- To avoid the low bias discussed above, calibration of the Fuel Product of interest is recommended. The product of interest should be prepared at five concentrations, as in 10.2.1 above, to construct a calibration curve, integrated for the range of interest, which is typically specified by the client. The resulting average Rf for the fuel of interest is used to quantify the associated field samples. Typical fuel products are listed in Section 8.12. Calculations are found in Section 11.0.

Acceptance Criteria: 25% RSD for all individual target compounds and 20% RSD for fuel products. All calibration standards must be analyzed within 24 hours.

10.2.3 Initial Calibration Verification (ICV)

- The analysis of an ICV standard must follow the initial calibration curve.

- After final processing, calculate the percent recovery of each compound by using the following calculation:

% Recovery = Found Amount / True Value x 100

- Acceptance Criteria: All recoveries must be +/- 20% of the true values.
- ICALs are documented on the *TPH SHC Initial Calibration Checklist (13151)*. The initial calibration must be secondary reviewed before analyzing samples

The following *corrective actions* are recommended for failing initial calibrations:

- Check the calibration of the standard preparation that was performed. If the problem appears to be isolated to a single standard, the standard may be reanalyzed within twelve hours. The %RSD may then be recalculated.
- If chromatographic problems are indicated by the standard chromatograms already analyzed, then the injection port should be maintenanced. About 6 to 12 inches should be cut off the column, and the liner should be replaced. If applicable the septa should also be replaced. If the system continues to fail after maintenance and has poor sensitivity it is possible the detector jet may need to be cleaned or replaced. The gold seal at the base of the injection port may also need to be replaced. After maintenance is performed repeat the initial calibration and check the re-calculated %RSDs.
- If calibration continues to fail, determine if the column has degraded and should be replaced. The choice of corrective active must be made in consultation with the Department Manager. In some cases, the decision may need consultation from the QA Manager and Project Manager.

10.3 Equipment Operation and Sample Processing

Instructions on equipment operation and data processing can be found in the Agilent Enviroquant manuals. Operation of equipment and sample processing must only be performed by experienced and trained analysts. See Sections 10 and 11 for additional details.

The internal standard (100uL of a 500ug/mL solution to 900uL extract) is manually added to each sample extract prior to analysis. A 1ul aliquot of sample extract is injected into the column. Dilutions may be necessary to provide results within the calibration of the analytical instrument. Samples will be re-analyzed after a highly concentrated sample(s) exceed calibration due to potential carryover contamination. The internal standard should be within the control limits listed in Section 12.0. If the Internal Standard is outside this criterion; reanalyze. If the reanalysis confirms the initial analysis, then the results are reported and narrated appropriately.

Surrogate recoveries should be within the control limits listed in Section 12.0. If any surrogate recoveries are outside the control limits reanalysis or reextraction may be necessary. Any anomalous conditions, such as emulsions during extraction or matrix interference, could be the cause of poor surrogate recoveries. If these conditions occur, then the results are reported and narrated appropriately.

10.4 Continuing Calibration

The systems performance and calibration for each compound of interest must be checked and verified once every 24 hours of analysis time. This is accomplished by analyzing a Continuing Calibration Verification (CCV) n-alkane standard that is at a concentration near the midpoint for the working range of the GC/FID, 50µg/mL.

- Analysis of samples must be bracketed by a valid continuing calibration check standard.

- If a CCV fails the criteria outlined in Section 12, all samples since the last acceptable CCV must be re-analyzed.
- The analyst judgment from continuous daily use of the instrument is important in determining the validity of a continuing calibration. Even if the %D criteria is met, the peak shape may have deteriorated to a point of maintenance is required and a new initial calibration is in order.
- A low response for C_{40} is an indication that the GC liner should be replaced.
- When analyzing for extended lists of hydrocarbons, the ratio of phytane to n-C_{18} must be 60% or better for the test to appropriately provide weathering information. If the ratio is acceptable, it is assumed that the n-C_{17} to pristane ratio is also acceptable.
- Some programs / projects may require the mass discrimination evaluation between n-C36 and n-C20.
- The area counts of the internal standards within the continuing calibration must be within 50 to 200% of the mid-level in previous ICAL.
-

10.5 Preventive Maintenance

All maintenance records including routine upkeep and outside service visits are maintained in the instrument maintenance logbooks for all FID systems. The maintenance logbook contains the minimum preventative maintenance and a history of all instrument maintenance. The instrument operating manuals provide more detailed information. These instruments are NOT under a maintenance service contract with the vendors. For repairs beyond the scope of routine upkeep and preventative maintenance, an independent service contractor must be called for on-site service and repairs.

Injection Port and Column Maintenance: Maintenance should be done when the daily CCV starts to demonstrate degradation. The type of samples analyzed will have an effect on how soon maintenance should be performed. The injection port can be cleaned using cotton swabs and DCM until no contamination is observed. Generally, maintenance is performed by trimming 6 cm off the front of the column. The column is then installed into the injection port liner and the inlet nut is tightened. After cleaning the instrument must be checked for any air leaks while cooling. The first injection should be a primer to remove any active sites and to check if any of the windows need updating.

Merlin Maintenance: The Merlin High Pressure Microseal (Part #410 Phenomenex) should be replaced when it cannot hold back pressure. A septum may be used as a backup or for troubleshooting.

11. Data Evaluation, Calculations and Reporting

11.1 Qualitative Identification of the n-alkane compounds is based on gas chromatographic relative retention times (RRTs) from the analysis of the mid-level initial calibration standard. For these compounds, manual quantitations are performed, if necessary, by integrating the area of the quantitation peak. For *petroleum ranges*, the starting and ending n-alkanes are used as integration markers.

Note: Manual integration is not to be used solely to meet QC criteria, nor is it to be used as a substitute for corrective action on the chromatographic system. Refer to Manual Integration and Compound Rejection SOP 1731.

11.2 An Instrument Blank run within the analytical sequence with the samples shall be used to mathematically correct the range analyte areas to account for the normal baseline rise of the instrument. The instrument blank selected for this purpose will be selected on 1) the similarity of the baseline performance between the instrument blank and the sample, and 2) the conservative preference for using an instrument blank with a low minimum response voltage. The instrument blank for baseline correction shall represent the lowest baseline response within the sequence. The blank is integrated for petroleum ranges only. This integration is then applied to the samples, so an identical amount of baseline drift is removed from the sample leaving only petroleum contributions in the range amount.

11.3 The chromatograms produced by the analysis are compared to reference chromatograms for qualitative identification. The reference chromatograms are stored as hard copy results as well as electronically in the data acquisition software. Electronic storage allows for evaluation using computer software.

11.4 The qualitative analysis can be time consuming. Retention time information for the alkanes and any Unresolved Complex Mixtures is essential. The graphical comparison of sample patterns to standard patterns is extremely useful in the interpretation of results. For example, a low range UCM may indicate the presence of a petroleum based lubricant. A mid range UCM may indicate the presence of fuel oil. High range UCM may indicate heavy lubricating oils or asphalt materials. No UCM in a chromatogram may indicate the presence of gasoline. Hard copy reports of comparisons should be retained for future reference.

11.5 To calculate the ***Relative Standard Deviation*** (RSD) of all target analytes and surrogate compounds for the initial calibration use the formula below. The RSD of each target compound and surrogate must be below 25%. Additionally, use the initial six-point calibration to determine ***Relative Response Factors*** (RRF_Is) at each concentration level. Average the RRF_Is, to generate mean RRF_Is, for quantification of all target analytes and surrogate compounds. The RRF_Is are based on the internal standard compounds and are calculated using the formula below. (The relative response factors for the continuing calibration verifications (RRF_Cs) are calculated using the same formula). See Section 16, Table I, for the listing of target compounds.

$$\mathbf{RSD = SD / mean\ RRF_I \times 100}$$

where:

SD = Standard deviation between the five points, for that target analyte.

$$\mathbf{RRF_I = (A_c \times C_{IS}) / (A_{IS} \times C_c)}$$

where:

A_c = Area of the characteristic ion for the standard compound to be measured.
A_{IS} =Area of the characteristic ion for the representative internal standard compound.
C_{IS} = Concentration of the representative internal standard compound (ng/mL).
C_c = Concentration of the standard compound to be measured (ng/mL).

11.6 Based on the mean RRF_Is, calculate the ***Sample Extract Amount*** for each target analyte and surrogate in the extracts using the following formula:

$$\mathbf{Q_e = (A_a \times Q_{IS}) / (A_{IS} \times RRF_I)}$$

where:
Q_e = Sample extract amount (ng) of target analyte, from quantitation report.
A_a = Area of the characteristic peak for the target analyte.
A_{IS} = Area of the characteristic peak for the representative internal standard compound.
Q_{IS} = Amount of internal standard compound added to each extract (ng).

11.7 Based on the area calculate the **Corrected Range Amount** for the petroleum ranges:

$$\mathbf{Q_e = ((A_{SR}-A_{IB})/A_{SR})*C_S}$$

where:

Q_e = Corrected sample extract amount (ng) of petroleum range.
A_{SR} = Area of the sample petroleum range.
A_{IB} = Area of the Instrument Blank petroleum range.
C_S = Sample extract amount for petroleum range, from quantitation report.

The corrected range amount is then brought through the sample calculation found in Section 11.6

11.8 Calculate the ***Sample Concentration*** (C) for each compound by the following formula:

$$\mathbf{C = (Q_e / V_s) \times DF}$$

where:

C = Concentration in sample (ng/L water, ug/Kg sediment/tissue, or mg/Kg product).
V_s = Original volume or weight of sample extracted.
DF = Dilution factor or fraction of the original extract to which internal standard added.

11.9 If the response of any target compound in a sample exceeds the linear response range, as defined by the initial calibration standards in Section 10.2, dilute the extract so that the concentrations of all target compounds fall within the range of the calibration curve. If the response of any target compound in a sample exceeds the MDL but is below the reporting limit (RL), qualify the reported concentration with a “J”. If any target compound is found in the method blank and in the associated sample(s) at or below 10x the blank concentration, qualify the reported result with a “B”.

11.10 Calculate the ***Surrogate Recoveries*** relative to the internal standards by the following formula:

$$\mathbf{\%R_{sc} = (A_{sc} / A_{is}) \times (Q_{is} / Q_{sc}) \times (100 / RRF_{sc}) \times DF}$$

where:

Q_{is} = Amount of the representative internal standard (ng).
DF = Dilution factor or fraction of the original extract.

11.11 Compare response factors for each compound in the ***Continuing Calibration Verification*** (CCV), to those of the initial calibration curve by determining the percent difference.

Percent Difference (%D) = ($[RRF_I - RRF_C] / RRF_I$) x 100

where:

RRF_I = Mean response factor from initial calibration.
RRF_C = Response factor from CCV.

11.12 External Standard Calibration:

$$\textbf{Calibration Factor (CF)} = \frac{\text{Total area w/in RT Range}}{\text{Mass injected (ng)}}$$

$$\textbf{\%RSD} = \frac{\text{SD}}{\text{Ave. CF}} \times 100$$

$$\textbf{Raw Sample Concentration, C, (ng)} = \frac{\text{Total area w/in RT Range}}{\text{Ave. CF}}$$

$$\textbf{Final Sample Concentration} = (C / V_s) \times DF$$

11.13 All solids including soils, sediments, and sludges must be reported on a dry-weight basis. Tissue results may be reported in wet weight depending upon client request. Petroleum results are reported “as received” or on a wet-weight basis.

11.14 The primary analyst does data entry, or uploading of the data, into the LIMS system. The LIMS is “linked” to the instrument, so the analyst must choose the sample(s) to be reported from that instrument’s analytical sequence. All associated preparation and instrumental QC samples and dilutions are also chosen. Once the data/samples have been selected and “associated” with the proper QC samples, the batched data set is sent to be reported.

11.15 The laboratory generates two types of data packages from the LIMS: “Commercial” or “Standard” for routine projects, and “Full Deliverable” or “CLP-like” for fully data validated projects. A Commercial or Standard package consists of sample results and the associated method blank and LCS results. A Full Deliverable or CLP-like package includes all sample results, all preparation and instrumental QC results and the associated supporting raw data. Check the “Report Type” on the project folder to ensure all required deliverables are included. Checklists are used for primary and secondary review of analytical results.

11.16 Procedures for data and record management must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP, 1729. All records shall be stored in such a manner as to be safe and accessible for at least 10 years.

11.17 Notebooks: Laboratory notebooks are designed to accommodate specific analysis. Instrument printouts are used to document run sequences, and each daily sequence printout is filed in a three-ring notebook. If a sample requires re-analysis or re-extraction for any reason, a notation is made next to the sample entry on the sequence log in the FID continuing calibration package. After one month of sample analysis, the sequence run log is permanently bound, assigned an internal ID number, and filed accordingly. Such files shall be archived to remain available for at least 10 years. All laboratory notebooks must follow

the specifications in the Laboratory Notebook Usage Work Instructions WI/1556, and all record keeping and document control practices.

11.18 Electronic records: All data files from computers, attached to instruments, shall be backed up daily onto the proper directory on the server. The backups shall be stored to be accessible for 10 years. Movement of the data files to the server is the responsibility of the primary analyst. Server backup and storage is the responsibility of the IT department.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All Total Petroleum Hydrocarbon results are reportable without qualification if analytical holding times are met, preservation (including pH and cooler temperatures) are met, and all QC criteria defined in the table below are met. If any of the below QC parameters are not met, all associated samples must be evaluated for re-analysis. See Section 9 for additional QC discussion including corrective actions for any QC outliers.

QC Parameter	Acceptance Criteria
Initial Calibration Curve	≤ 25 %RSD for all compounds and within 0.06 RRT units for all compounds Fuel Product: ≤20% RSD
24 Hour Continuing Calibration Verification	≤ 25%D for all individual compounds with exception for 10% of target analytes can be >25%, but ≤ 35% Fuel Product: ≤20%D
Method Blank	No analyte at or above the reporting limit, “B” qualify analyte if detected in the samples ≤ 10x the concentration in the blank
LCS/LCSD	50-130% recovery
MS/MSD	50-150% recovery, 30%RPD
Matrix Duplicate	30%RPD for results >5X reporting limit
Surrogates	50-130% recovery
Internal Standards	IS area must be within 50-200% of the last CCV and the IS retention time must be with 30 seconds of the last CCV.

Section 9 outlines sample batch QC acceptance criteria. If non-compliant Total Petroleum Hydrocarbon results are to be reported, the Department Manager, and/or the Laboratory Director, and/or the QA Officer must approve the reporting of these results. The laboratory Project Manager shall be notified, and may choose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

Once satisfactory Petroleum results have been generated, the extracts are held for 30 days, or longer if specified by a client contract, then discarded into a 55-gallon drum labeled "Vial Waste".

15. Referenced Documents

2124 Chemical Hygiene Plan

SOP/1729 Document Control

SOP/1731 Manual Integration and Compound Rejection

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1797 Waste Management and Disposal SOP

SOP/1816 Reagent, Solvent and Standard Control

SOP/2255 PIANO Volatiles

SOP/2165 Separatory Funnel

SOP/2166 Tissue Preparation and Homogenization

SOP/2261 Shaker Table Extraction

SOP/2263 Gravimetric Determination

SOP/2264 Tissue Extraction

SOP/2265 Organic Waste Dilution

16. Attachments

Table I: Target Compounds

17. Revision History

Revisions Made from Prior Version

Section	Description of Change
10.3	Samples re-analyzed after high-concentration samples.

17	Revision History added

Document Succession: This version replaces the following document(s):

Document Number	Version	Document Title	Effective Date:
2246	9	ENV-SOP-MANS-0096 v01 Total Petroleum and Saturated Hydrocarbons by GC_FID 8015	4/24/2020

Table I

Target Compounds

n-Nonane (C9)	n-Nonacosane (C29)
n-Decane (C10)	n-Triacontane (C30)
n-Undecane (C11)	n-Hentriacontane (C31)
n-Dodecane (C12)	n-Dotriacontane (C32)
n-Tridecane (C13)	n-Tritriacontane (C33)
1380	n-tetratriacontane (C34)
n-Tetradecane (C14)	n-Pentatriacontane (C35)
1470	n-Hexatriacontane (C36)
n-Pentadecane (C15)	n-Heptatriacontane (C37)
n-Hexadecane (C16)	n-Octatriacontane (C38)
1650	n-Nonatriacontane (C39)
n-Heptadecane (C17)	n-Tetracontane (C40)
Pristane	C9-C44 Total Petroleum Hydrocarbons (TPH)
n-Octadecane (C18)	Total Saturated Hydrocarbons
Phytane	DRO(C10-C28)*
n-Nonadecane (C19)	ORO(C28-C40)**
n-Eicosane (C20)	
n-Heneicosane (C21)	**Surrogate Compounds**
n-Docosane (C22)	ortho-terphenyl
n-Tricosane (C23)	d50-Tetracosane
n-Tetracosane (C24)	
n-Pentacosane (C25)	**Internal Standard**
n-Hexacosane (C26)	5-alpha-androstane
n-Heptacosane (C27)	
n-Octacosane (C28)	

*DRO: Diesel Range Organics

**ORO: Oil Range Organics

Biochemical Oxygen Demand (BOD), 5-Day

Soluble BOD (SBOD), 5-Day
Carbonaceous BOD (cBOD), 5-Day

Reference Methods: **SM 5210B,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

1. Scope and Application

Matrices: This method is applicable to most water and wastewater samples.

Definitions: See Alpha Laboratories Quality Manual Appendix A

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Dissolved Oxygen (DO) Meter and in the interpretation of Dissolved Oxygen data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

The biochemical oxygen demand (BOD) test is used to determine the relative oxygen requirements of wastewaters, effluents, and polluted waters. The test has its widest application in measuring waste loadings to treatment plants and in evaluating the BOD-removal efficiency of such treatment systems. The test measures the oxygen utilized during a specified incubation period for the biochemical degradation of organic material (carbonaceous demand) and the oxygen used to oxidize inorganic materials such as sulfides and ferrous iron. It also may measure the oxygen used to oxidize reduced forms of nitrogen (nitrogenous demand). The seeding and dilution procedures provide an estimate of the BOD at pH 6.5 to 7.5.

The BOD concentration in most wastewaters exceeds the concentration of dissolved oxygen (DO) available in an air-saturated sample. Therefore, it is necessary to dilute the sample before incubation to bring the oxygen demand and supply into appropriate balance. Because bacterial growth requires nutrients such as nitrogen, phosphorus, and trace metals, these are added to the dilution water, which is buffered to ensure that the pH of the incubated sample remains in a range suitable for bacterial growth. Complete stabilization of a sample may require a period of incubation too long for practical purposes; therefore, 5 days has been accepted as the standard incubation period.

2. Summary of Method

The method consists of filling with sample, an airtight bottle of the specified size and incubating it at the specified temperature for 5 days. Dissolved oxygen is measured initially and after incubation, and the BOD is computed from the difference between initial and final DO.

2.1 Method Modifications from Reference

Soluble BOD determinations: Soluble BOD determinations may be performed by filtration of the sample through 0.45µm filter. Following filtration, the sample is placed in a 1L plastic

bottle and shaken vigorously for 1 minute, before any sample pretreatment procedures are performed.

3. Detection Limits

The minimum calculated RL for BOD is 2 mg O_2 / L, based on a 50% dilution of the sample (i.e. 150mL sample in the 300mL BOD bottle.)

The minimum dilution of the sample that meets acceptance criteria is used to determine the final calculated RL for the sample as follows:

$$\text{Reporting Limit, } BOD_5\text{, mg } O_2 / L = \frac{1}{\frac{\text{(mL sample used for minimum dilution meeting criteria)}}{300}}$$

Refer to Table 1.

4. Interferences

4.1 Instrumental

The DO Meter must be calibrated and maintained according to manufacturer's instructions in order to avoid any instrumental interferences.

BOD Analyzer must be maintained according to manufacturer's suggestions for optimum performance.

4.2 Parameters

The samples must be free of biological poisons or materials that produce oxygen.

5. Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in 1L plastic containers.

6.2 Sample Preservation

No preservative is added to samples.

6.3 Sample Shipping

No special shipping requirements other than samples should be on ice.

6.4 Sample Handling

Samples for BOD analysis may degrade significantly during storage between collection and analysis, resulting in low BOD values. Minimize reduction of BOD by analyzing sample promptly or by cooling it to near-freezing temperature during storage.

6.4.1 Grab samples: If analysis is begun within 2 hours of collection, cold storage is unnecessary. If analysis is not started within 2 hours of sample collection, keep sample at or below 4ºC, but not freezing, from the time of collection. Analysis must begin within 48 hours of collection.

6.4.2 Composite samples: Use the same criteria as for storage of grab samples, starting the measurement of holding time from the end of the compositing period. Analysis must begin within 48 hours of collection.

7. Equipment and Supplies

7.1 Backup use only: BOD Incubation Bottles: 300mL capacity, glass bottles with glass stoppers and plastic snap-caps. Clean bottles, stoppers and snap-caps with a laboratory grade detergent; rinse thoroughly, and drain before use.

7.2 Primary use: BOD Incubation Bottles: 300mL capacity “PET resin” disposable plastic BOD bottles (Environmental Express, part # D1001), with glass stoppers and plastic snap-caps.

7.3 Air Incubator: Thermostatically controlled at 20 ± 1ºC. Exclude all light to prevent possibility of photosynthetic production of DO.

7.4 pH Paper: Range 0-14

7.5 15mL Centrifuge Tubes.

7.6 COD Reactor: 150° C, Hach, Hanna Instruments or equivalent

7.7 Spectrophotometer: Model Genesys 10S UV-VIS or equivalent

7.8 Pipets: Disposable plastic, , 1mL to 30 mL graduated.

7.9 YSI Model DO Meter with DO Electrode: YSI ProOBOD or equivalent.

7.10 BOD Analyst Software: Seal Program or equivalent

7.11 Volumetric Flasks: 100mL volume, 1 L volume; Class A

7.12 Autopipettor: With disposable tips, 0.5 - 5mL volume.

7.13 Peroxide Test Strips: Range 0 – 30 mg/L.

7.14 BOD Analyzer System: By MANTECH, with AutoMax 122, and all associated equipment. Or BOD (Minilab) Analyzer System by Seal Analytical with all associated equipment.

7.15 Stir plates: 1 with heat; 1 without heat

7.16 Wide-mouth 500ml flask

7.17 pH Meter: Laboratory benchtop pH meter with automatic temperature compensator and combination electrode.

7.18 Disposable, plastic transfer pipettes: VWR # 16001-180

7.19 Stir bars, 1" length

7.20 50mL Centrifuge Tubes

8. Reagents and Standards

8.1 Glucose-Glutamic Acid:

8.1.1 Purchased, premade Glucose-Glutamic Acid standard solution ampules: Use 1 ampule per LCS bottle for analysis. Carefully empty all contents into a clean/new bod bottle. Rinse with nutrient solution from working tank for batch, using clean/new, plastic, disposable transfer pipette.

InterLab, product # P910	pk of 48 vials	or equivalent.
InterLab product # P510	bottle 125mL	or equivalent.

8.1.2 Lab prepared, Glucose-Glutamic Acid (for LCS and Spike): Dry reagent-grade glucose and reagent-grade glutamic acid at 105ºC for a minimum of one hour, and cool before use.

8.1.2.1 For LCS Stock Solution (198ppm): In a 1L volumetric flask, add 150mg (0.15g) glucose and 150mg (0.15g) glutamic acid to distilled water and dilute to volume. Stir this solution until dissolved; stir for at least 45 minutes. Prepare fresh immediately before use.

8.1.2.1.1 198ppm LCS: While stirring, withdraw 6mL of the glucose-glutamic acid stock solution (Section 8.1.2.1) and add to non-empty BOD bottles for analysis.

8.1.2.2 For Spike Stock Solution: In a 100mL volumetric flask, add 1.5g glucose and 1.5g glutamic acid to distilled water and dilute to volume. Slightly heat this solution until dissolved; stir for at least 45 minutes. Prepare fresh immediately before use.

8.1.2.2.1 100ppm Spike: While stirring, withdraw 0.5mL of the glucose-glutamic acid stock solution (Section 8.1.2.2) and add to 100mL of sample.

8.1.2.2.2 198ppm Spike: While stirring, withdraw 1mL of the glucose-glutamic acid stock solution (Section 8.1.2.2) and add to 100mL of sample.

8.2 BOD Nutrient Solution/Dilution Water: Fill tank with DI at least a day prior to use. Aerate tank with DI for 4 hours. The Nutrient Solution/ Dilution Water can be used for 3 days from the time of aeration/pillow addition. Store in the dark at room temperature.

- Fill carboy to 38L with DI water.
- After filling, saturate with DO by aerating for 4 hours. Protect water quality by using clean tubing.
- Just before sample batch preparation, add 2 BOD Nutrient Buffer Pillows (HACH Brand, 1 pillow is for preparation of 19L). Agitate tank vigorously for 1 minute to mix after adding pillows.
- Tank is now ready for use; expiration is 3 days from aeration/pillow addition.

8.3 Sulfuric Acid Solution (1N): For neutralization of caustic samples. Slowly and while stirring, add 2.8mL concentrated H_2SO_4 to distilled water. Dilute to 100mL with DI. Store refrigerated at 1-4 °C. Expires one year from date of preparation.

8.4 Sodium Hydroxide Solution (4N): For neutralization of acidic samples. Dissolve 40g sodium hydroxide (NaOH) in distilled water. Dilute to 250mL with DI. Store refrigerated at 1-4 °C. Expires one year from date of preparation.

8.5 Sodium Sulfite Solution: Dissolve 0.1575g Na_2SO_3 in 100mL distilled water. This solution is not stable; prepare fresh immediately before use.

8.6 Polyseed Inoculum: Dissolve one Polyseed capsule (InterLab Supply, commercial BOD seed inoculum) in 500mL nutrient solution; stir for a minimum of two hours prior to use. Stir at a spin speed which permits approximately ½ inch vortex. Warning: If the seed is set to spin too quickly, this could lead to the death of organisms in the seed and therefore a low recovery value. The Polyseed solution is viable for 6 hours from time of preparation. The sample can also be aerated for a minimum of 1 hour in place of stirring.

Adjust the Polyseed volume added to each batch based on the Polyseed lot strength. The desired seed recovery range is 0.6 – 1.0 mg/L. Test each lot of Polyseed prior to use to determine a volume that will produce results within this acceptance range.

8.6.1 If the Polyseed is to be used with the BOD Analyzer procedure (note: only when using the automated seed pump), then stop spinning the Polyseed after the minimum of 2 hours, allow sediment to settle for 5 minutes, then carefully pour the top portion of the solution into a different, clean 500ml flask, trying not to disturb any of the settled material while doing so.

8.6.2 This poured off portion of the Polyseed becomes the solution used with BOD and cBOD setup with the BOD Analyzer (to avoid having sedimentary material caught in the equipment lines).

Note: A capsule from new lots of polyseed should be analyzed *in advance*, to determine the correct dilution range. Record the use of a new lot of polyseed in the Bod logbook.

8.7 DPD Free Chlorine Reagent Powder HACH brand, Swiftest: for 10mL sample. Use with HACH Swiftest free chlorine reagent dispenser. Store at room temperature. Expires upon manufacturer specified date.

8.7.1 Hach Swiftest DPD free chlorine reagent dispenser: cat # 28023-00

8.7.2 Hach Swiftest DPD free chlorine reagent powder vial refill for 10ml sample cat # 2105560

8.8 COD Tubes:

8.8.1 CPI (catalog # COD1500), range 0 – 1500 mg/L.

8.8.2 Environmental Express (catalog # B1015), range 0 – 1500 mg/L. Store at room temperature. Expires upon manufacturer specified date.

8.9 1:1 Acetic Acid Solution: 50mL of DI + 50mL Acetic Acid. Dilute to 100mL with DI. Store refrigerated at 1-4 °C. Expires one year from date of preparation

8.10 KI Solution: 10 grams KI per 100mL DI. Dilute to 100mL with DI. Store refrigerated at 1-4 °C. Expires one year from date of preparation

8.11 Starch Indicator Solution: HACH brand. Store at room temperature. Expires upon manufacturer specified date.

8.12 Nitrification Inhibitor: HACH brand, with 0.2g dispensing scoop. Store at room temperature. Expires upon manufacturer specified date.

8.13 Respirometric BOD Nutrient Buffer Solution Pillows: HACH brand, Product #2962266. Store at room temperature. Expires upon manufacturer specified date.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

9.1.1 Dilution Water Check: Determine initial DO for 3 BOD bottles of dilution water for each analytical batch (Section 8.2). Incubate these bottles along with the samples for 5 days at 20 ± 1ºC. Determine the final DO.

The DO depletion after 5 days at 20 ± 1ºC should not be more than 0.2mg/L and preferably not more than 0.1mg/L. If the blank depletion falls outside of these limits, the analytical batch may be considered invalid. All other QC parameters must be evaluated. Notation should be done in the raw data. Notify the Department Supervisor and/or the Laboratory Manager for instructions on how to proceed.

9.1.2 Method Blank: Determine initial DO for 3 BOD bottles of DI water with volumes of 30mL, 60mL and 150mL for each analytical batch (Section 8.2). Incubate these bottles along with the samples for 5 days at 20 ± 1ºC. Determine the final DO after incubation for 5 days at 20 ± 1ºC. Results must be less than the RL. If failure occurs, the Inorganics Supervisor is notified.

9.2 Laboratory Control Sample (LCS)

Determine initial DO for the LCS as stated in Section 8.1.1 purchased, or 8.1.2.1.1 for in-house prepared standard. Incubate these bottles along with the samples for 5 days at 20 ± 1ºC. Determine the final DO.

The true value of the LCS equals 198mg O_2/L. The % Recovery must be between 85 – 115%. If the % Recovery for the LCS falls outside these limits, the analytical batch is not valid. Notify the Department Supervisor and/or the Laboratory Manager for instructions on how to proceed.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Prepare and analyze a matrix spike for one sample (as determined in Section 8.1.2.2.1 or 8.1.2.2.2) per batch of 20 samples or less. % Recovery must be 85-115%, or according to in house control limits.

9.6 Laboratory Duplicate

Prepare and analyze a duplicate set of dilutions for one sample per batch of 20 samples or less. % RPD must be ≤ 20%, or according to in house control limits.

9.7 Method-specific Quality Control Samples

9.7.1 Seed

Determine initial DO for the volumes of Polyseed as stated in Section 8.6. Incubate these bottles along with the samples for 5 days at 20 ± 1ºC. Determine the final DO.

The final seed depletion after 5-day incubation at 20 ± 1ºC ideally should be between 0.6 and 1.0 mg/L.

9.8 Method Sequence

The analytical sequence is:

- Dilution Water Blank
- Seed
- Glucose-Glutamic Acid LCS
- Method Blank
- Samples 1-20 or less
- Duplicate
- Matrix Spike

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation of Dilution Water Blank: Fill three 300mL BOD bottles with Nutrient Solution/Dilution Water (Section 8.2). Determine the BOD of the Dilution Water as for any other sample. (If setup is done with BOD Analyzer, skip to step 10.1.3).

Note: If more than 150 ml of the sample has to be used for analysis (per customer instructions only) , then additional Nutrient solution has to be added. It has to be done by adding one pillow of Respirometric BOD Nutrient Buffer Solution (8.13) to each bottle.

10.1.2 Preparation of Polyseed Value: Make dilutions of the Polyseed solution (Section 8.6) using volumes of 8mL, 10mL, 12mL, and 14mL (or 15mL, 18mLs and 21mLs). Determine the BOD of the seeding material as for any other sample. (If setup is done with BOD Analyzer, skip to step 10.1.3). This varies as the seed value is adjusted per lot of Polyseed.

10.1.3 Preparation of LCS

10.1.3.1 Purchased LCS ampules: Use as instructed in section 8.1.1.

10.1.3.2 In House Preparation of LCS: Use 6mL, 6mL, and 6mL volumes of the 198ppm LCS solution (Section 8.1.2.1.1). Determine the BOD of the LCS as for any other sample.

10.1.4 Preparation of Method Blank: Fill 3 bottles with DI volumes of 30mL, 60mL and 150mL. (If setup is done with BOD Analyzer, skip to step 10.1.5).

10.1.5 cBOD Dilution Bottle Preparation: If samples are to be analyzed for cBOD, add 0.2g (1 scoop) of Nitrification Inhibitor (Section 8.12) to each dilution bottle and QC bottle, prior to filling with sample, and prior to filling/reading on BOD Analyzer.

10.1.6 Sample Pretreatment:

10.1.6.1 Samples containing caustic alkalinity or acidity: Check the pH of all samples using pH paper. If the sample pH is between 6.0 and 8.0, then do not adjust the pH, but record the pH value in the BOD preparation logbook. However, any samples that are outside the range of 6.0 to 8.0 require pH adjustment. Neutralize these samples to pH 7.0 to 7.2 with sulfuric acid solution (Section 8.3) **or** sodium hydroxide solution (Section 8.4) using pH meter. *NOTE: Do not use both solutions on the same sample.*

The quantity of reagent added must not dilute the sample by more than 0.5%. If 0.5% is exceeded, notify the Department Supervisor and/or the Laboratory Manager for instructions on how to proceed.

10.1.6.2 Samples containing residual chlorine compounds: Test samples for chlorine by adding 1 DPD free chlorine reagent powder pillow (Section 8.7) to a 15mL centrifuge tube. Add 5mL sample. Mix. Chlorine is present if sample turns a pinkish color. If there is no color change, no chlorine is present. Proceed to Section 10.1.6.3.

(**NOTE:** If there is a change to a very rosy pink color, sample is checked for peroxide using a peroxide strip. If the sample is positive for peroxide, sample analysis is ended and results are narrated as invalid due to peroxide interference.)

If chlorine is present, it must be removed from the sample. To do this, it is necessary to determine the required volume of Sodium Sulfite (Na_2SO_3) solution (Section 8.5) to add to a 100mL portion of neutralized sample. Do this by adding 1mL of 1:1 acetic acid solution (Section 8.9), 1mL potassium iodide (KI) solution (Section 8.10) and 1mL starch indicator solution (Section 8.11) to 100mL of sample, and titrating with Na_2SO_3 solution to the starch-iodine end point. The end point is reached when blue color disappears.

Determine the volume titrated. This is the volume of Na_2SO_3 solution necessary to dechlorinate 100mL of sample. (**NOTE:** If the volume of Na_2SO_3 solution is greater than 10mL for 100mL sample, consult the Department Supervisor and/or the Laboratory Manager before proceeding.) (**NOTE**: Discard titrated sample.) Add this determined volume of Na_2SO_3 solution per 100mL of sample. Mix. After 10 to 20 minutes, check sample again for residual chlorine. (**NOTE**: Excess Na_2SO_3 exerts an oxygen demand and reacts slowly with certain organic chloramine compounds that may be present in chlorinated samples.)

10.1.6.3 COD Check for Determination of BOD Dilutions: A COD allowed to reflux for 30 minutes is enough time to be able to calculate an estimated BOD value. (Refer to the Standard Operating Procedure for COD ID#2208.) Once a COD value is calculated, use TABLE 1 to select dilutions for the BOD. TABLE 1 is based on the RDL at each dilution. Compare the COD result to the BOD RDL in TABLE 1. Since the BOD is always less than the COD, choose the 5 BOD sample volumes less than the COD value. In the case of a COD less than 20mg O_2/L, choose the 30mL, 60mL, and 150mL volumes (10, 20, 50% of a 300mL BOD bottle).

10.1.6.4 Sample temperature adjustment: Bring samples to 20 ± 1ºC before making dilutions.

10.2 Initial Calibration

10.2.1 If following the Manual procedure in Section 10.3.1, calibrate the YSI Dissolved Oxygen Meter per manufacturer's instructions.

10.2.2 Winkler Titration Calibration – Weekly Check

Note: This check is not needed for the YSI ProOBOD Optical Probes.

10.2.2.1 Fill 1 extra BOD bottle with only Nutrient Solution. Prepare this bottle for dissolved oxygen analysis as per the Dissolved Oxygen SOP 2249.

10.2.2.2 Immediately following the probe reading of the first blank BOD bottle in the batch (Section 10.3.2), titrate the extra Nutrient Solution bottle prepared in Section 10.2.2.1. according to the Dissolved Oxygen SOP 2249. Record the D.O. result from the Winkler titration in the BOD logbook

The D.O. results obtained from the probe and the Winkler titration should be very similar. If results are not within these criteria, refer to Section 10.5 for preventive maintenance procedures for the D.O. probe, and repeat Sections 10.2.2.1 – 10.2.2.2.

10.3 Equipment Operation and Sample Processing

10.3.1 MANUAL PROCEDURE: USE OF BOD ANALYST SOFTWARE

10.3.1.1 Using a wide-tip pipet, add the desired volumes of Polyseed, LCS and samples to the individual BOD bottles and dilute with BOD Nutrient Solution/Dilution Water (Section 8.2).

Note: Add approximately 25ml of Nutrient Solution to Polyseed bottles **before** adding the seed volumes.

For sample dilutions greater than 1:100, make a primary dilution using BOD Nutrient Solution/Dilution Water in a volumetric flask before making the final dilution in the BOD bottle

10.3.1.2 Preparing the Matrix Spike:

The spike concentration chosen (either Section 8.1.2.2 or Section 8.1.2.3), should be greater than or equal to the expected sample value as determined in Section 9.1.5.3. Therefore, choose a sample to spike that has both of the following:

(1) a COD result less than or equal to the spike amount chosen and

(2) enough volume in the sample container to allow setting both the sample and the spike.

Determine the BOD dilutions for the spike as in Section 8.1.2.1 or 8.1.2.3. Be sure to calculate the sample COD value and add in the spike amount chosen. Use this value to compare with TABLE 1. Four volumes are needed when setting the spike.

10.3.1.3 Using the autopipettor set at 2.0mL, add 2mL of seed material to the individual BOD bottles for the LCS and samples. ((Note: The seed volume added to the batch may vary, depending on the current seed value of the polyseed lot currently in use (Section 8.6)).

10.3.1.4 Fill BOD bottles with enough nutrient solution so as to come halfway up the neck of the bottle.

10.3.1.5 **Determination of Initial DO:** Following the manufacturer's instructions, use the DO electrode, DO meter, and BOD Analyst Software to determine initial DO for all sample dilutions, dilution water blanks, seed controls, and the glucose-glutamic acid LCS. Rinse electrode between determinations to prevent cross-contamination of samples.

10.3.1.6 Tightly insert a glass stopper to displace all air, leaving no bubbles and creating a water-seal. Place a plastic snap-cap over the flared mouth of the bottle to prevent evaporation.

10.3.1.7 Incubation:

10.3.1.7.1 Incubate BOD bottles for 5 days at 20 ºC ± 1 ºC.

10.3.1.7.2 Note the time IN and Incubator ID in the BOD prep logbook. (The initial incubation date/time is used as the "prep time" for the data).

10.3.1.8 Determination of Final DO:

10.3.1.8.1 After a 5-day incubation period, determine final DO for all sample dilutions, dilution water blanks, seed controls and the glucose-glutamic acid LCS, using the membrane electrode, DO meter and BOD Analyst Software.

10.3.1.8.2 Note the time OUT in the Bod prep logbook. (The final DO reading (end of incubation) date/time is used as the "analysis time" for the data).

10.3.1.8.3 The OUT time is derived from absolutely no more than +/- 6 hours of the IN date/time of the BOD batch of samples to the incubator. (example: If the IN time is 12:00pm, then the batch must be taken out of the incubator and the final DO readings conducted no earlier than 6:00am and no later than 6:00pm).

10.3.2 AUTOMATED PROCEDURE: USE OF BOD ANALYZER

10.3.2.1 Setup of Solutions Prior to Analysis of Samples

1. Make Nutrient Solution/Dilution Water (refer to Section 8.2). This should be made several hours in advance in order for the dissolved oxygen level of the tank to be relatively stable prior to use.
2. Make LCS/SPK stock standard (refer to Section 8.1) a minimum of two hours prior to use of analyzer.
3. Make Polyseed (refer to Section 8.6) a minimum of two hours prior to use of analyzer.

10.3.2.2 Setup of Equipment Prior to Sample Analysis

1. Turn on analyzer and associated computer. The analyzer should have 5 lights lit on the equipment, indicating that all pieces are powered on.
2. Make sure that the manifold shelf is pushed back behind the autosampler main section. (VERY IMPORTANT: if this is not in the correct location, the autosampler arm will crash during operation and disrupt analysis).
3. Fill the DI tank so there is ample water to supply the probe washing station.
4. Make sure that the autosampler tray base is completely on the laboratory countertop (refer to arrow markings on either side), to ensure the base does not become unstable during use.

10.3.2.3 Setup of Samples Prior to Analysis (Preparing Dilutions Directly in BOD Bottles)

10.3.2.3.1 Preparing the Polyseed Value and LCS bottles

Using a calibrated pipet or calibrated autopipettor, add the desired volumes of Polyseed and LCS to the individual BOD bottles. Use wide-mouth pipets for samples. (Do not **fill** the bottles with NS/Dilution water. It will be added automatically by the Analyzer).

Note: Only add polyseed to bottles as each rack for the autosampler becomes ready for Initial D.O. reading, to ensure samples are not seeded prematurely while waiting to be read.

Note: Add approximately 25ml of Nutrient Solution to Polyseed bottles **before** adding the seed volumes.

For sample dilutions greater than 1:100, make a primary dilution using BOD Nutrient Solution/Dilution Water in a volumetric flask before making the final dilution in the BOD bottle.

10.3.2.3.2 Preparing the Matrix Spike and Sample Bottles

The spike concentration chosen (either Section 8.1.2 or Section 8.1.3), should be greater than or equal to the expected sample value as determined in Section 10.1.6.3. Therefore, choose a sample to spike that has both of the following:

1. A COD result less than or equal to the spike amount chosen

   and

2. Enough volume in the sample container to allow setting both the sample and the spike.

Determine the BOD dilutions for the spike as in Section 10.1.6.3. Be sure to calculate the sample COD value and add in the spike amount chosen. Use this value to compare with TABLE 1. Four volumes are needed when setting the spike. Use a calibrated pipet or calibrated autopipettor to dispense spiked sample solution.

10.3.2.3.3 Preparing the Polyseed bottles

(Skip this section if the Polyseed is to be added automatically by the Analyzer).

Note: Add approximately 25ml of Nutrient Solution to Polyseed bottles **before** adding the seed volumes.

Using the autopipettor set at 2.0 mL, add 2mL of seed material to the individual BOD bottles for the LCS and samples. [Note: The seed volume added to a batch is determined in advance per lot of Polyseed to ensure the value should fall within the ideal range. (Refer to Section 8.6).]

10.3.2.4 Preparation of Analyzer Equipment for Use

10.3.2.4.1 On the computer desktop screen, double-click on the yellow “Z lightning bolt” icon to open the BOD Analyzer software.

10.3.2.4.2 The page which opens will be entitled “PC-BOD Main Menu”. (This will be referred to as the Main Menu screen going forward.)

NOTE: If a batch is being set up for initial readings, then proceed here. If a batch is to be read for final Bod/CBod, then skip to Section 10.3.A.9.

10.3.2.4.3 On the bottom of the Main Menu screen, there are several icons. They are defined as follows:

- #1 = BOD daily
- #2 = CBOD daily
- Blue/purple spots = change membrane
- Green cup = prime pumps
- Water drip = probe calibration
- Ringing bell = park the probe
- Raindrops = clean the system

10.3.2.5 Priming the System

10.3.2.5.1 Double-click on the prime pumps icon; this will initialize the software communication. A new page will open, called the Timetable/Sample Entry Screen. (This will be referred to as the Timetable screen going forward.)

10.3.2.5.2 Unhook the bod bottle from the autosample arm/probe (Note: HOLD autosampler arm/probe with one hand and gently twist bod bottle off to lower to tray. DO NOT twist and remove bottle without holding the arm/probe while doing this!)

10.3.2.5.3 Hit the start button (green rectangle on bottom right of Timetable page). A box will appear, asking “enter a valid rack #”. Type “0”. Hit ok. A box will appear, asking “is seed reservoir filled?”. (Refer to Section 8.6 for Polyseed preparation.) If yes, then hit ok. A box will appear, asking “is dilution water reservoir filled?”. (Refer to Section 8.2 for NS/dilution water preparation.) If yes, then hit ok.

10.3.2.5.4 At this time, the autosampler will move to wash the probe at the washing station. It is normal equipment behavior that some water will overflow during this process. When the priming of the system is completed, a box will appear, stating “pumps are now primed and ready for use”. Hit ok. Then hit the done button at the bottom right of the Timetable page. This will close the page back out to the Main Menu screen.

NOTE: Make sure the drain/waste line is secured to the equipment, and able to flow in a gravimetrically and unobstructed fashion, to prevent backwash into the equipment.

10.3.2.6 Calibrating the Probe

10.3.2.6.1 On the Main menu screen, press the water drip icon, for calibrating the probe. This will open the Timetable page.

10.3.2.6.2 On the Timetable page, press the start button (green rectangular box). A box will appear, asking “enter a valid rack #”. Enter “0”, and hit ok.

10.3.2.6.3 After the probe is finished at the washing station, a box will appear, asking to “wipe off excess water from probe”. Do this VERY GENTLY, just blotting the tip with a soft towel, so as not to tear or otherwise damage the membrane. Hit ok.

10.3.2.6.4 A box will appear, asking “is a bottle with 1 inch of water in position #1?”. If yes, then hit ok. Another box will appear, stating to “attach the probe to the bottle for a good seal”.

10.3.2.6.5 At this time, the system will automatically become immobile for 10 minutes, during the countdown to ensure probe stability prior to calibration.

10.3.2.6.6 Once the 10 minutes has passed, a box will appear, asking "is the reading stable?". At this time, the probe needs to be calibrated manually, as follows: [Refer to Section 10.2.A.1 for further information on calibrating the probe per the manufacturer's instructions.]

- Press the mode button on the probe meter twice to return the meter to the main menu, hit ok.
- Press the calibrate button, then adjust the meter reading to 100%, hit ok.
- Press the mode button twice, then the remote button twice to return the meter to the remote mode, hit ok.
- Make sure the meter is in remote mode, hit ok.
- **Very important!!** Unhook the BOD bottle from the probe.

10.3.2.6.6.1 The reading should be within 85 – 115 % on the probe meter. If yes, then hit ok.
The Shazam Report Wizard page will open; hit "X" to close (this page is not needed at this time). After closing this Shazam page, the green start button will be visible again.

10.3.2.6.6.2 A box will appear, stating that "now the system is primed and calibrated, and ready for use". Hit ok, and then done, to close out of the calibration page.

10.3.2.6.6.3 Park the probe!!! (Section 10.3.2.10)

10.3.2.7 Running a Batch of BOD/CBOD Samples

10.3.2.7.1 On the bottom left part of the Main Menu screen, there are 2 icons. The '1" is for BOD daily, and the "2" is for CBOD daily. Select the appropriate icon button for the samples to be analyzed. This will open the Timetable page.

10.3.2.7.2 Hit the edit button, located near the middle of the page, to enter sample/batch information. Enter name/date/time at the top of the page. Add rows as needed (using the add rows button). Hit the done edit button when the sample/batch process is completed.

10.3.2.7.3 Hit the green rectangular start button on the bottom right of the page, once the rack of sample bottles is placed on the autosampler base tray. A box will appear, asking "enter a rack #". Enter the rack #, and hit ok. The run will be initiated at this time, and the autosampler will begin to read and fill bottles.

Note: If multiple racks are used in the same batch, only the LAST rack can be less than completely full of bottles.

10.3.2.7.4 After the first rack (and each additional rack when applicable), a box will appear, instructing "remove rack and replace with next rack", at which time, the current rack is removed, the next rack is placed on the autosampler base tray, hit the start button, and a box will appear again, asking for the rack #..

Note: If there is no additional rack, then just hit ok. If all the racks have been read for the batch, then the Shazam Report Wizard page opens. Print a copy of the initial readings of the batch for future reference. Hang the copy on the appropriate BOD incubator where the batch will be housed. Close the Shazam page after printing. When the start button becomes green again, hit the done button to return to the Main Menu page.

(REMINDER: On the printout front page, write the date/time of when the BOD or cBOD batch is to come out of the incubator to be read.)

Tightly insert a glass stopper to displace all air, leaving no bubbles and creating a water-seal. Place a plastic snap-cap over the flared mouth of the bottle to prevent evaporation.

10.3.2.8 Incubation:

10.3.2.8.1 Incubate BOD bottles for 5 days at 20 ºC ± 1 ºC.

10.3.2.8.2 Note the time IN and Incubator ID in the BOD prep logbook. (The initial incubation date/time is used as the "prep time" for the data).

10.3.2.9 Cleaning the System After Use

10.3.2.9.1 On the Main Menu page, the icon with the multiple raindrops refers to the cleaning of the system. This procedure drain and rinses the dilution water and seed lines associated with the equipment.

Note: This procedure is done after all the initial and final readings are completed for the period of time that the equipment was continually in use.

10.3.2.9.2 Press the Clean System icon button. The page will appear stating that it is for the cleaning of the system. Hit the start button.

- A box will appear, asking "enter a valid rack #". Enter 0. Hit ok.
- A box will appear, instructing to "remove seed and dilution water lines from reservoirs". Hit ok. This process removes all liquid from the lines.
- Once completed, another box will appear, instructing to "put the lines in separate containers of DI". Hit ok. This process rinses DI thru the lines to clean them.
- Next, another box will appear, stating to "remove seed and dilution lines from the DI containers". Hit ok. This process will empty all remaining liquid from the lines.

- Finally, when this has been completed, a box will appear, stating that "the pumps are now clean". Hit ok.

10.3.2.10 Parking the Probe

It is important now to park the probe (the ringing bell icon).

- Push the park probe icon.
- A box will appear, stating to "place a bottle in position 1 of rack". Hit ok.
- A box will appear, stating to "snug the bottle on the probe for a good seal".

  Note: This keeps the probe membrane from drying out.
- Hit done to close the page back to the Main Menu page.
- If no further use of the equipment is necessary at this time, close out of the software by hitting the "X" at the top right of the Main Menu page. The software will automatically conduct a saving procedure, and then close entirely.

10.3.2.11 Determination of Final DO

10.3.2.11.1 After a 5-day incubation period, determine final DO for all sample dilutions, dilution water blanks, seed controls and the glucose-glutamic acid LCS, using the membrane electrode, DO meter and BOD Analyzer.

10.3.2.11.2 Note the time OUT in the Bod prep logbook. (The final DO reading (end of incubation) date/time is used as the "analysis time" for the data).

10.3.2.11.3 The OUT time is derived from absolutely no more than +/- 6 hours of the IN date/time of the BOD batch of samples to the incubator. (example: If the IN time is 12:00pm, then the batch must be taken out of the incubator and the final DO readings conducted no earlier than 6:00am and no later than 6:00pm).

- Open software (see Section 10.3.A.3) to Main Menu page.
- This page has a list of batches which need to be read, which are still in progress.
- Click on the icon "1" for Bod, or icon "2" for CBod.
- At the bottom left of the page, click on "load existing runs"
- Select the batch to read finals on (by date of setup). Hit "load selected".
- Then hit "start" (once probe calibration has been completed first! If it has not, refer to section 10.3.A.5). Probe should be unhooked from bottle at this time, and the rack of samples to be read first should be on the autosampler tray (with all plastic caps and glass corks removed).
- Read the racks, as in section 10.3.A.6, and when all readings have been finished, print data from the Shazam Wizard. Then hit "done".
- Park the probe and snug the bottle onto the probe.

- Close out of the software.
- Turn off computer, if done using for the day/night.

10.3.2.12 Printing a Previous BOD/cBOD batch

- If a copy of a previous batch is needed, simply open the software, as explained in Section 10.3.A.3, and go to the top of the Main Menu page.
- Click on the BOD heading, and then select “Post Run Bod Analysis”.
- On this new page which opens, at the bottom is a box which says “completed runs”.
- Select the start date of the batch to be opened. Click on the batch. Then hit “load selected”.
- Once the selected batch is visible, hit “run method” to provide results for the batch on the screen.
- Hit “print results”. Then select “printer”. Hit “print”. Then hit “done”.

IMPORTANT NOTE: If software is opened only to print a previous batch, then initiation of software with equipment is not necessary; therefore, the bottle does not need to be unhooked from the probe. But whenever the software AND equipment are to be used, for initial and final readings, the bottle MUST be unhooked prior to hitting ‘start”, or the probe could crash.

10.3.3 AUTOMATED PROCEDURE: USE OF SEAL BOD ANALYZER

10.3.3.1 Setup of solutions prior to analysis

Make Nutrient/Dilution water (Section 8.2)

Make Spike stock standard (Section 8.1.2.2) a minimum of two hours prior to use

Make Polyseed (Section 8.6) a minimum of two hours prior to use

10.3.3.2 Setup of equipment prior to use

- Turn on Seal Autosampler, computer, seed pump, nutrient solution pump and two DO meters. Lights will turn on and screens on DO meters will power on.
- Initilaize the system (Tools-> initialize system)
- Flush probes (Tool -> flush probes)
- Calibrate DO probes (Tools -> Cal DO probes -> Follow popup instructions to wipe down probe heads)
- On the DO meter display (Cal -> select ProODO -> then DO%)
- Place the larger plastic tubes into the Nutrient solution tank and the smaller tubes in the decanted seed.
- Prime the lines (Tool -> Flush dilution liquid and flush addition liquid)

10.3.3.3 Setup of Samples prior to analysis (preparing dilutions directly in BOD bottles)

10.3.3.3.1 Preparing the LCS Bottles

Add premeasured vial of GGA (Section 8.1.1) and rinse vial with DI

10.3.3.3.2 Preparing the Method Blank Bottles

Add 30mL, 60mLs and 150mLs of DI to bottles

10.3.3.3.3 Preparing the Matrix Spike

The spike concentration chosen (either Section 8.1.2.2.1 or Section 8.1.2.2.2), should be greater than or equal to the expected sample value as determined in Section 10.1.6.3. Therefore, choose a sample to spike that has both of the following:

- A COD result less than or equal to the spike amount

  chosen **and**

- Enough volume in the sample container to allow setting both the sample and the spike

Determine the BOD dilutions for the spike as in Section 10.1.6.3. Be sure to calculate the sample COD value and add in the spike amount chosen. Use this value to compare with TABLE 1. Four volumes are needed when setting the spike. Use a calibrated pipet or calibrated autopipettor to dispense spiked sample solution. For spike 100 use volumes 3, 6, 15 and 30mLs. For spike 200 use volumes 1.5, 5, 10, 30mLs. (i.e For spike 100 take 100mLs of sample and add 0.5 mLs of the spike stock standard. For spike 200 take 50mLs of samples and add 0.5 mLs of spike stock standard.)

10.3.3.3.4 Preparing the Sample bottles

Using the COD screening results in Table 2 add the specified volumes to each bottle

10.3.3.4 Sample Analysis

- In SEAL program go to “New table by template”
  - Name new sequence by the WG#
  - Add lines to sequence by hitting the down arrow
  - Scan samples 3 to 6 times, depending on the different dilutions / volumes needed
  - Fill in different volumes and dilution factors
- Under table -> Measure Start DO, Right click tray holders and select Load BOD tray. Once all bottles are loaded select start routine
- Number all the bottles (1, 2, 3, 4 etc)
- Remove from trays and stopper and cap each bottle
- Add samples to incubator for 5 days +/- 6 hours

10.3.3.5 Cleaning System and Determining of Final DO

- Flush the nutrient solution and seed lines with DI and then air
- Under start select “open active table”
- Remove caps and stoppers from bottles and load onto trays in numerical order
- Under table select “measure end DO”
- Start routine
- At end of run hit calculate

10.3.3.6 Data

- Export the file to BOD FINAL and name as WG#
- Select print data from table (Print to PDF)
- Always save as BOD final report (regardless of product)
- When printing select ALL and save as WG#_rawdata in the BOD rawdata folder
- Open the CSV file associated with WG and expand volumes to include all data
- Add WG# to each sample (WG column) and BOD11 to instrument ID for all samples
- Change start time DO and end time DO to the times in the preplog corresponding to Date/Time In and Date/Time Out
- Change LCS samples to WG#-2 and Method Blank to WG#-1,
- the operator is “sealbod” change that to the initials of the person who ran the batch
- For Soluble BOD add the letter S, cBOD the letter C and soluble cBOD SC
- Save, Drag and drop the CSV file in PC_BOD drive, Drag and drop the WG#_rawdata in to the WG_files drive. Scan and drop the preplog
- Approve data in LIMS

10.4 Continuing Calibration

Not applicable.

10.5 Preventative Maintenance

The membrane on the DO electrode should be changed and the electrode tip serviced at regular intervals per the manufacturer’s instructions (for membrane probes).

Temperatures of the BOD incubators are recorded continuously by an automated datalogger. The temperatures are checked by the analysts twice daily (with a minimum of 4 hours between readings) and adjusted as necessary.

The BOD incubators are calibrated on an annual basis by an instrument service company. These records are kept on file.

BOD Analyzer and optical probes must be maintained according to manufacturer’s suggestions for optimum performance.

11. Data Evaluation, Calculations and Reporting

11.1 Both the BOD Analyst Software and the BOD Analyzer Software will calculate the BOD results automatically upon completion of final dissolved oxygen readings. However, the following is an explanation of the manual calculation should it be necessary:

$$BOD_5, \text{ mg } O_2 / L = \frac{[\,(D_1 - D_2) - f\,]}{P}$$

where:

D_1 = DO of diluted sample immediately after preparation, mg/L,
D_2 = DO of diluted sample after 5-day incubation at 20ºC, mg/L,
P = Decimal volumetric fraction of sample used,
f = Average DO depletion for 2mL of seed.

11.2 If more than one sample dilution meets the criteria of a final DO of at least 1mg/L and a DO depletion of at least 2mg/L, and there is no evidence of toxicity at higher sample concentrations or the existence of an obvious anomaly, then calculate the average of the BOD results in the acceptable range.

11.3 In these calculations, do not make corrections for DO depletion by the dilution water blank during incubation. This correction is unnecessary if dilution water meets the blank criteria specified in Section 9.1.2.

11.3.1 If the dilution water does not meet these criteria, proper corrections are difficult and results become questionable. Notify the Department Supervisor and/or the Laboratory Manager for instructions on how to proceed.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 Dilution Blank (Section 9.1.1) If this falls outside the acceptance limit, a lab note is included with the batch if the seed is within the acceptable range. If the dilution blank and seed are both out of range, then the batch is invalid and the client is notified.

12.2 Seed (Section 9.7.1) If the seed is outside the ideal acceptance range, the reason for the failure is evaluated and eliminated by adjusting the seed volume used in future sample batches. If any batch falls outside of the ideal acceptance range, but the LCS is still within acceptable range, the data is released without further action to the client. However, if the LCS falls outside acceptable range for the method, the client is notified and the data is only released (with an appropriate narrative) upon the client's request.

12.3 LCS (Section 9.2) If the LCS is outside of the acceptance range, and the seed is within its acceptance range, then a narrative is included on the Client report and the data is reported (per the department manager evaluation of the data).

12.4 Method Blank (Section 9.1.2) If the method blank is too high, either a narrative is included on the Client report (per department manager) or the batch is considered invalid and the client is notified.

12.5 Matrix Spike (Section 9.5) If the spike is outside of the acceptance limits, the data is reported with a narrative that is included on the Client report.

12.6 Laboratory Duplicate (Section 9.6) If the duplicate is outside of the acceptance limits, the data is reported with a narrative that is included on the Client report.

12.7 Evaluation of data: Samples showing large differences between calculated BOD results for different dilutions, for example, greater than 30%, may indicate the presence of a toxic substance or analytical problems. If test replicates show more than 30% difference between high and low values, a narrative is added to the Alpha report. Canned narrative #1312 should be used.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

However, for BOD and CBOD analyses, the MDL is a calculated value. Therefore, MDL studies are not performed.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734 and 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1734, 1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

TABLE 1: Reported Detection Limits for Sample Volumes Indicated

TABLE 2: BOD Dilution Recommendation from the COD Result

TABLE 1

Reported Detection Limits for Sample Volumes Indicated

BOD RDL ($mg\ O_2$ / L)	Sample Volume (mLs)
100,000	0.003
50,000	0.006
20,000	0.015
10,000	0.03
5,000	0.06
2,000	0.15
1,000	0.3
500	0.6
200	1.5
100	3.0
50.0	6.0
20.0	15.0
10.0	30.0
5.0	60.0
2.0	150

TABLE 2

BOD Dilution Recommendation from the COD Result

COD Result (ppm)	BOD Dilution (mLs)
0-20	30, 60, 150
21-50	20, 60, 150
51-100	6, 20, 60, 150
101-200	3, 10, 25, 60
201-500	1.5, 5, 10, 30

Process Standard Operating Procedure (SOP): Pace® Analytical Services

Pace ANALYTICAL SERVICES	**Balance Verification and Maintenance**
	Effective Date: 04/18/2024

Management Approval:
Amy Rice Approved on 4/18/2024 7:14:09 AM

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1.0 PURPOSE

This Standard Operating Procedure (SOP) specifies the policies and procedures for verifying the calibration of laboratory balances using NIST traceable weights.

2.0 SCOPE & APPLICATION

The policies and procedures specified in this SOP apply to the following locations: PAS-Mansfield, PAS-Westborough

This Standard Operating Procedure describes how to check the calibration of balances and reflects the Quality Control (QC) limits and the procedures to follow if a balance falls outside of the QC limits.

The degree of accuracy of the balance is reflected in the accuracy of all data related to weight-prepared standards, spikes and sample dilutions and sample weight determinations. Because this accuracy is critical to our operation, each balance's calibration must be verified before it is put into service each day. The results are recorded in the logbook associated with the balance.

3.0 SUMMARY OF PROCESS

Balances are verified daily or on each day of use against Class S-1 weights that are certified to be traceable to the National Institute of Standards and Technology (NIST). These working weights are verified as accurate against reference weights which are calibrated and certified by an outside vendor. Certificates are kept on file, and records are kept for the daily balance calibration checks.

4.0 DEFINITIONS

Refer to the Laboratory Quality Manual for a glossary of common lab terms and definitions.

- **PAS:** Acronym for Pace® Analytical Services, LLC

5.0 HEALTH & SAFETY

Contact your supervisor or local safety coordinator with questions or concerns regarding safety protocol or safe handling procedures for this procedure.

The following sections provide general health and safety information about chemicals and materials that may be present in the laboratory.

- The toxicity or carcinogenicity of each chemical material used in the laboratory has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be as low as reasonably achievable.

The distribution of printed or electronic copies of this document outside of the PAS electronic document management system (eDMS) is not controlled or tracked. PAS employees must ensure any copy used for daily work, if not accessed directly from PAS eDMS, is the current version of the document.

- The laboratory maintains documentation of hazard assessments and OSHA regulations regarding the safe handling of the chemicals specified in each method. Safety data sheets for all hazardous chemicals are available to all personnel. Employees must abide by the health, safety and environmental (EHS) policies and procedures specified in this SOP and in the Pace® Chemical Hygiene / Safety Manual (COR-MAN-0001).

- Personal protective equipment (PPE) such as safety glasses, gloves, and a laboratory coat must be worn in designated areas and while handling samples and chemical materials to protect against physical contact with samples that contain potentially hazardous chemicals and exposure to chemical materials used in the procedure.

- Concentrated corrosives present additional hazards and are damaging to skin and mucus membranes. For procedures that require use of acids, use acids in a fume hood whenever possible with PPE designed for handing these materials. If eye or skin contact occurs, flush with large volumes of water. When working with acids, always add acid to water to prevent violent reactions. For procedures that that emit large volumes of solvents (evaporation/concentration processes), these activities must be performed in a fume hood or apparatus that reduces exposure.

6.0 PROCEDURE

6.1 Policy

Balances provide critical sample and reagent volume weights used to calculate sample results. Balances must be verified for accuracy using a set of certified weights traceable to NIST. Balances and their workstations must be kept clean and should be dusted before and after use. Balances must be set up on a level surface free from vibrations, and moving a balance should be kept to a bare minimum as they are sensitive pieces of equipment. Due to the sensitivity of balances, they must be allowed to acclimate to a new location for a minimum of 2 hours after being moved. Because of this, please discuss with your Manager and/or QA staff prior to moving a balance. Verification must be conducted after a balance has moved, even if verification has already been conducted for that day.

6.2 Procedure

6.2.1 Daily Verification of Laboratory balances

- Sign out weights using the Weight Sign-out Logbook. Confirm that no weights are missing, or record what weights are missing. If any weights are missing, contact your local Quality Assurance (QA) department.
- Make sure that the pan is clean (brush), and the balance is level. Please see a member of the QA Department if a brush is not available in the lab. Each balance used in the lab makes use of a bubble level and four adjustable legs. Make sure the bubble is within the circle by adjusting the balance legs.
- Tare the Balance

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- Select the appropriate weight based on the weight listed in the true Value column of the balance Verification logbook. Place the weight on the pan with the forceps provided with the set of weights. Weights should never be handled directly by hand, as the oils in your skin can change the weight. Please see a member of the Quality Team for any questions regarding Balance verification ranges.
- Record the measured weight to the N^{th} number of decimal places specific for the balance. The true values of the calibration weights are indicated on each weight. Also record the date and the Analyst's initials.
- If applicable, mark the day of verification on the laminated verification tracker affixed near the balance using a dry-erase marker. This notifies potential users that the balance has been verified for that day. Check weights back in using the Weight Sign-out Logbook.

7.0 RESPONSIBILITIES

- PAS employees that perform any part this procedure in their work activities must have a signed Read and Acknowledgement Statement (R&A) in their training file for the version(s) of the SOP that were in effect during the time the employee performed the activity.
- PAS supervisors and Managers however named, are responsible for training employees on the procedures in this SOP, implementing the SOP in the work area, and monitoring on-going adherence to the SOP in the work area(s) they oversee.
- PAS employees are responsible for following the procedures in this SOP. Unauthorized deviations or departures from this SOP are not allowed except with documented approval from the local QM and only when those deviations do not violate the Pace® Code of Ethics or Professional Conduct (COR-POL-0004) or associated policy and procedure(s). Hand-edits or manual changes to the SOP are not permitted. If a change is desired or necessary, Pace® employees must follow the procedures for document revision specified in corporate SOPs ENV-SOP-CORQ-0015, *Document Management* and ENV-SOP-CORQ-0016, *SOP for Creation of SOP and SWI*.

8.0 ATTACHMENTS

9.0 REFERENCES

- Balance Verification Logbook – Air
- Balance Verification Logbook – Metals Digestion
- Balance Verification Logbook – Organic Extractions (Mansfield)
- Balance Verification Logbook – Wet Chemistry (Mansfield)
- Cahn Balance Verification Logbook
- Balance Verification Logbook – PFAS Lab
- Balance Verification Logbook – Balance 15
- Balance Verification Logbook – Balance 3

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- Balance Verification Logbook – Volatiles and Organic Prep
- Balance Verification Logbook – Login
- Balance Verification Logbook – Wet Chemistry (Westborough)
- ENV-SOP-CORQ-0015, *Document Management*, current version.
- ENV-SOP-CORQ-0016, *SOP for SOP and SWI,* current version.
- ENV-MAN-(058), *Quality Manual*, current version.
- COR-POL-0004, *Code of Ethics and Professional Conduct*, current version.
- TNI Standard, "Volume 1 Management and Technical Requirements for Laboratories Performing Environmental Analysis", current version.
- U.S. Department of Defense (DoD) QSM V5.4, MA DEP 310 CMR 42.00, NH ELAP

10.0 REVISION HISTORY

Revisions Made from Prior Version

Section	Description of Change
X	X

Document Succession: This version replaces the following document(s):

Document Number	Version	Document Title	Effective Date:
X	##	X	X

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Appendix A: (LIST any appendices or attachments to the SOP)

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Free Cyanide

Method Reference: Method 9016, Revision 0, June 2010, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods.

1. Scope and Application

Matrices: This test method covers the determination of free cyanide in waste waters, ground waters, surface waters, drinking waters, soils and solid wastes.

Definitions: Refer to Alpha Analytical Quality Manual.

This test method reports the cyanide that dissociates from simple cyanides or weakly-bound metal cyanide complexes at room temperature, from a solution of pH 6-6.5.

This test method does not determine strongly-bound metal cyanide complexes that resist dissociation, such as the hexacyanoferrates and gold cyanide, nor does it determine thiocyanate and cyanohydrin.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of experienced analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Parameter	CAS
CN	57-12-5

2. Summary of Method

2.1 The sample preparation (Sec. 10.3.1) is carried out using a microdiffusion cell (Figure 1 and Figure 2) The water, wastewater or extract sample is introduced in the outer chamber of the microdiffusion cell and is buffered at pH 6 and placed in the dark for 6 hrs of diffusion. The free cyanide diffuses as HCN gas and is absorbed as CN- into the sodium hydroxide solution located in the center chamber of the microdiffusion cell.

2.2 An aliquot of the sodium hydroxide absorber solution is removed and treated with acidified phosphate buffer and chloramine-T to convert the CN- to cyanogen chloride. The cyanogen chloride is reacted with pyridine-barbituric acid to form a color complex that absorbs at 578 to 587 nm. The free cyanide is determined spectrophotometrically by measuring the absorbance of the sample and determining the concentration through comparison with a standard calibration curve (Sec. 10.2).

2.3 Solids are first extracted prior to analysis using pH 12.3-12.5 NaOH solution (Sec. 10.1.). The filtered extracts are then diffused (Sec. 10.3.1) and analyzed spectrophotometrically as

described in Sec. 10.3.2. The extraction conditions described in this method have been found to be effective at extracting free cyanide from a variety of solid wastes. Figure 3 illustrates the effects of various extraction conditions on the recovery of free cyanide in a complex, mixed-waste sample.

2.4 The extraction conditions in this method may be modified as needed to improve free cyanide extraction efficiency from other matrices. For example, certain relatively complicated wastes or waste mixtures may not yield acceptable matrix spike recoveries. In such cases, additional refinement of the extraction step and/or the use of chemical pretreatments may be necessary to improve free cyanide recovery. Such flexibility is allowed as long as the data meets the quality objectives of the specific project.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Reporting limit for water samples is 0.002 mg/l (2.0 ppb). Reporting limit for soil samples is 1.0 mg/kg

4. Interferences

4.1 Interferences, such as chlorine and sulfides, can degrade samples by reacting with the free cyanide present. All aqueous samples should be checked at the time of their collection to determine the presence of oxidizing agents and/or sulfides. If found to be present, the samples should be immediately treated, as noted in the following sections, prior to their storage for future analysis.

4.1.1 Oxidizing agents, such as chlorine, decompose free cyanide. Chlorine reacts with free cyanide to form cyanogen chloride (CNCl), which under alkaline conditions hydrolyzes to cyanate (CNO-). Chlorine interferences can be removed by adding excess amounts of sodium arsenite or sodium thiosulfate to the sample prior to storage. Both sodium arsenite and sodium thiosulfate reduce the chlorine to chloride, which does not react with free cyanide or otherwise interfere in its analysis

4.1.2 Sulfide oxidation products can rapidly convert free cyanide to thiocyanate (SCN-), especially at high pH .Sulfide interferences (namely hydrogen sulfide, metal sulfides, or other compounds that may produce sulfide) can be removed by adding an excess of either lead carbonate or lead acetate to the sample. The addition of either reagent forms insoluble lead sulfide (PbS), so that it may be removed via filtration, prior to storage or analysis

4.2 Volatility losses of free cyanide (as HCN) can occur in samples having pH values less than 12. If samples cannot be analyzed immediately after field collection and treatment for oxidizing agents and sulfides, they must be preserved by adjusting the pH to 12 or greater prior to storage.

4.3 In addition to oxidizing agents and sulfides, free cyanide can react with other chemicals such as aldehydes. Because of the reactivity of free cyanide, it is important that analysis is completed as soon as possible after sample collection. It is beyond the scope of this method to list all the possible cyanide reactions that may be encountered.

4.4 Hexacyanoferrate complexes in water samples can decompose when exposed to UV light and produce free cyanide. This decomposition is virtually eliminated during the test procedure by allowing samples to diffuse in the dark.

4.5 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be necessary. Refer to each method to be used for specific guidance on QC procedures and to Chapter Three for general guidance on the cleaning of glassware. Also, refer to the Method 9014 for additional discussion of interferences.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

WARNING: KCN and NaCN are highly toxic. Avoid skin and eye contact and inhalation.

Because of the toxicity of cyanide, exercise great care in its handling. Acidification of cyanide solutions produces toxic gaseous hydrogen cyanide (HCN). Perform all manipulations in the hood so that any HCN gas that is formed is safely vented.

Some of the reagents used in these methods, such as those containing cyanide and pyridine-barbituric acid solutions, are highly toxic in the environment. Dispose of such reagents and their solutions properly, according to applicable state or local regulations.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

All samples must be collected using a sampling plan that addresses the considerations discussed in Chapter Nine of the SW-846 manual.

Samples should be collected in plastic or glass (preferably plastic) containers that are either amber or covered with aluminum foil so as to filter light at 400 nm and below and prevent photodecomposition of metal cyanide complexes. All containers must be thoroughly cleaned and rinsed prior to use. All sample containers must be prewashed with acids, water, and metal-free detergents, if necessary, depending on the use history of the container.

All samples whose origin or composition is not well-known should be tested for the presence or absence of oxidizing agents, such as chlorine; sulfide ideally at the time of sample collection

6.2 Sample Preservation

Analyze samples immediately; otherwise preserve them at the time of collection, following any treatment for oxidizing agents or sulfides by adding 50% sodium hydroxide, until the pH is equal to or slightly greater than 12. This minimizes cyanide losses due to volatilization of HCN.

6.3 Sample Shipping

Ship and Store properly-preserved samples in the dark at 4 ± 2 °C.

6.4 Sample Handling

When properly preserved, cyanide samples can be stored for up to 14 days prior to analysis.

Extract solids within 14 days of sample collection.

Analyze extracts of solid samples within 24 hrs following extraction.

7. Equipment and Supplies

7.1 Supplies/equipment for sample collection, preservation, and handling

7.1.1 Potassium iodide-starch test paper.

7.1.2 Lead-acetate paper test paper

7.1.3 pH test paper

7.1.4 Plastic conway-type microdiffusion cell*, 83-mm OD — Equipped with a fitted lid or other type cover, for use in maintaining an airtight seal when in place manufactured by Bel-Art or other commercial vendor

7.2 Supplies/equipment for extraction of solid samples

7.2.1 Extraction bottles, HDPE, 1000-mL capacity or larger

7.2.2 Agitation apparatus — Rotary agitator (provides end-over-end motion), orbital shaker or rotary shaker or other equivalent apparatus, capable of providing thorough mixing of samples during extraction.

7.3 Supplies/equipment for microdiffusion processing and spectrophotometric analysis

7.3.1 Micropipets, 0.20-mL, 0.50-mL, 1.00-mL, 1.50-mL, 2.50-mL, 5.00-mL, 10.0-mL, 15.0-mL, and 30.0-mL

7.3.2 Adjustable pipette or calibrated syringe — Able to be adjusted to deliver exactly 3.00 mL of solution

7.3.3 Adjustable pipette or calibrated syringe — Able to be adjusted to deliver exactly 1.30 mL of solution

7.3.4 Spectrophotometer — Suitable for making absorbance measurements between 575 and 590 nm using a 1.0-cm cell

7.3.5 Spectrophotometer cell, 1-cm

8. Reagents and Standards

8.1 Reagent-grade chemicals must be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society

8.2 Reagent water: Reagent water must be interference-free. All references to water in this method refer to reagent water, as defined in Chapter One of SW-846 manual.

8.3 Reagents for sample collection, preservation, and handling

8.3.1 Sodium arsenite, NaAsO2

8.3.2 Sodium thiosulfate, Na2S2O3

8.3.3 Lead acetate trihydrate, Pb(CH3COO)2 • 3H2O

8.3.4 Lead carbonate, PbCO3.

8.3.5 Sodium hydroxide solution (50% w/w; ρ = 1.53 g/mL), NaOH: Commercially prepared.

8.4 Additional reagents needed for extraction of solid samples

8.4.1 Sodium hydroxide solution (50% w/w) (see Sec. 8.3.5)

8.4.2 Sodium hydroxide extraction solution (pH 12.3-12.5): Weigh either 2.53 g or measure 1.65 mL of 50% sodium hydroxide solution and transfer to a 1-L volumetric flask containing approximately 800 mL of reagent water. Stir until dissolved. Adjust the final volume to 1-L using reagent water. Using pH test paper, check to ensure that the extraction solution is in the proper pH range. Add additional 50% NaOH, if necessary, to adjust the pH to the proper range

8.5 Additional reagents needed for microdiffusion processing (Sec. 10.2) and spectrophotometric analysis (Sec. 10.3).

8.5.1 Sodium hydroxide solution (2.05 g/L), NaOH: Weigh either 4.10 g or measure 2.68 mL of 50% sodium hydroxide solution and transfer to a 1-L volumetric flask containing approximately 800 mL reagent water. Stir until dissolved. Cool the solution to room temperature and adjust the final volume to 1 L using reagent water. This solution is used only for the preparation of initial demonstration of proficiency samples, method blanks, matrix spikes and laboratory control standards.

8.5.2 Sodium hydroxide solution (4.1 g/L): Weigh either 8.20 g or measure 5.36 mL of 50% sodium hydroxide solution and transfer to a 1-L volumetric flask containing approximately 800 mL of reagent water. Stir until dissolved. Adjust the final volume to 1-L using reagent water.

CAUTION: Calibration standards are commercially available from several sources.

8.5.3 Potassium cyanide, KCN; or sodium cyanide, NaCN: Commercially available 1000 ppm; expiration date according to manufacturer specification.

8.5.4 Standard cyanide solution (1 mg/L): Pipet 1.00 mL of 1000-mg/L stock cyanide solution into a 1-L volumetric flask and dilute to volume using 4.1-g/L sodium hydroxide solution. This solution is used in the preparation of calibration standards (see Sec. 10.1).

WARNING: KCN or NaCN are highly toxic. Avoid skin and eye contact and inhalation

8.5.5 Stopcock grease, petroleum jelly or mineral oil: to help in providing an air-tight seal between the microdiffusion cell and lid

8.5.6 Potassium phosphate monobasic, KH2PO4

8.5.7 Sodium hydroxide solution (100 g/L), NaOH: Weigh either 200 g or measure 131 mL of 50% sodium hydroxide solution and transfer to a 1-L volumetric flask containing approximately 800 mL reagent water. Stir until dissolved. Cool the solution to room temperature and adjust the final volume to 1 L using reagent water.

8.5.8 Concentrated phosphoric acid (sp gr 1.69), H3PO4

8.5.9 Potassium phosphate solution (190 g/L): Add 400 mL of reagent water to a 2-L beaker. Next, either weigh 29 g or measure 19 mL of 50%-sodium hydroxide solution, transfer to beaker, and dissolve. Add 190 g of potassium phosphate monobasic and stir to dissolve. Bring the volume to approximately 950-mL to aid in dissolution. Adjust the pH of the solution so that it is within the range of 5.9-6.1, using 100-g/L sodium hydroxide or concentrated phosphoric acid. Transfer the solution to a 1-L volumetric flask, and dilute to volume with reagent water.

8.5.10 Potassium phosphate buffer solution (acidified): Add 8.0 mL of concentrated phosphoric acid to 100 mL of 190-g/L potassium phosphate solution.

8.5.11 Chloramine-T reagent (10 g/L): Dissolve 1.0 g of chloramine-T in 50 mL of reagent water contained in a 100-mL volumetric flask. Dilute to volume with additional reagent water. Prepare fresh daily.

8.5.12 Barbituric acid, NHCONHCOCH2CO

8.5.13 Pyridine, C5H5N

8.5.14 Concentrated hydrochloric acid HCl

8.5.15 Pyridine-barbituric acid reagent: Add 15.0 g of barbituric acid to a 250-mL volumetric flask. Wash down the sides of the flask with just enough reagent water to moisten the barbituric acid. Add 75 mL of pyridine and swirl to mix. Slowly, add 15 mL of concentrated hydrochloric acid and swirl to mix. Using additional reagent water, bring volume to approximately 230 mL. Stir to dissolve and cool the solution to room temperature. Dilute to final volume with additional reagent water and mix. It is recommended that the reagent be prepared fresh weekly and stored in a dark place. Discard and prepare a new batch if a precipitate develops.

WARNING: Pyridine is toxic; avoid contact or inhalation. Prepare this reagent in an exhaust hood.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A method blank (MB) is prepared from a 2.05 g/L sodium hydroxide solution and treated exactly as a field sample, including exposure to all glassware, equipment, solvents, filtration, and reagents that are used with field samples.

Any free cyanide measured in the indicates that contamination is present. The source of the contamination should be determined and corrected prior to performing any sample analysis. Any sample included in an analysis batch that has an unacceptable MB concentration should be reanalyzed in a subsequent batch after the contamination problem is resolved.

9.2 Laboratory Control Sample (LCS)

A laboratory control sample (LCS) should be included with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same analytes at the same concentrations as the matrix spike, when appropriate

Prepare 50 ug/l LCS solution: pipet 2.5 ml of 1 ppm cyanide standard to 50ml volumetric flask Adjust volume up to 50 ml with 0.1N NaOH solution.

Prepare 100 ug/l LCS solution: pipet 5 ml of 1 ppm cyanide standard to 50ml volumetric flask Adjust volume up to 50 ml with 0.1N NaOH solution

LCS acceptance criteria 75-125%. New criteria may be established based on in house control charts.

When the LCS recovery exceeds the control limits, the analysis should be terminated, the problem corrected

9.3 Initial Calibration Verification (ICV)

Immediately after the calibration standards have been analyzed, the accuracy of the calibration must be verified by the analysis of an ICV standard. The ICV is prepared in the same manner as a calibration standard (i.e., the sample is NOT processed through the microdiffusion procedure) at a concentration level within the calibration range of the method and using a second source standard (prepared using standards different from the calibration standards) spiked into 4.1 g/L sodium hydroxide .

The control limit for the ICV is ±15% of the true value. When the ICV exceeds the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified.

9.4 Continuing Calibration Verification (CCV)

Once the calibration curve has been established, the continuing accuracy must be verified by analysis of a CCV after every tenth field sample, and at the end of the analysis sequence. The CCV is equivalent to or prepared in the same manner as a calibration standard (i.e., the sample is NOT processed through the microdiffusion procedure) at a concentration level within the calibration range of the method and using the same source standard (prepared using the same source standards as those used to prepare the calibration standards) spiked into 4.1 g/L sodium hydroxide (see Sec. 10.2).

CCV concentrations alternating between the low- and mid-range calibration standard concentrations are recommended.

The control limit for the low-range CCV (5 ug/L) is ± 50% and for the mid-range CCV(100 ug/L) is ± 15% of the true value.

When the CCV exceeds the control limits, the analysis should be terminated, the problem corrected, the instrument recalibrated, and the calibration re-verified using an ICV analysis. Samples that are not bracketed by acceptable CCV runs must be reanalyzed.

9.5 Matrix Spike

Both aqueous samples and solid sample extracts should be represented by a minimum of one MS sample for each respective matrix.

Pipet 2.5 ml of 1 ppm cyanide standard to 50ml volumetric flask with sample. Adjust volume up to 50 ml with sample. Final spike concentration: 50 ug/l

Pipet 5 ml of 1 ppm cyanide standard to 50ml volumetric flask Adjust volume up to 50 ml with sample. Final spike concentration 100 ug/l

MS acceptance criteria 70-130% for waters and 65-135% for soils. New criteria may be established based on in house control charts.

If the Matrix Spike recovery does not meet acceptance criteria, and the LCS recovery is acceptable, matrix interference may be assumed. The associated data may be reported with a narrative included.

9.6 Laboratory Duplicate

One duplicate per batch has to be analyzed. . If samples are expected to contain the target analyte, laboratories may use a matrix spike (MS) and a duplicate analysis of an unspiked field sample. If samples are not expected to contain the target analyte, the laboratories should use a MS/MSD pair.

% RPD for the Duplicate must be within in-house control limits. If acceptance criteria are not met, the Duplicate is reanalyzed. If failure continues, a narrative is included with the data for inclusion on the Client report.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

1. Set equipment
2. Prep samples, using Microdiffusion procedure
3. Leave samples in dark place for 6 h
4. Make calibration standards and run calibration curve
5. Prep. and analyze ICV and ICB for curve verification
6. Use calorimetric procedure to analyze samples after Microdiffusion process is finished (after 6h)
7. Curve verification (ICB and ICV) after every twenty sample and at the end of the analytical run

10. Procedure

CAUTION: This method requires manual dexterity, practice, and meticulous attention to operational consistency. Best practices are noted throughout this section to assist analysts in achieving optimum results. Adherence to these practices is highly recommended for attaining precise and accurate data.

10.1 Equipment Set-up

10.1.1 Extraction of solid samples

Extract solid samples prior to microdiffusion and analysis, otherwise proceed to Section 10.3.

10.1.1.1 Weigh 250 mg $PbCO_3$ and transfer to an extraction bottle.

NOTE: Since, sulfides are often present in soils and solid wastes, lead carbonate is added to all extract samples in order to fix sulfides as PbS.

10.1.1.2 Weigh a 1 g sample of the solid to be analyzed, recording the sample mass to 0.001 g. Transfer the weighed sample to the extraction bottle containing the lead carbonate.

10.1.1.3 Add 500 mL of pH 12.3-12.5–NaOH solution to the extraction bottle and gently rotate the bottle so as to mix the contents.

10.1.1.4 10.1.4 Cap the extraction bottle, place on agitation apparatus and agitate for 5 min. After 5 min, use a strip of pH test paper check the sample mixture pH to verify that it is within the range of 12.3-12.5. If it is not, add 50% NaOH drop-wise, to bring the pH to within the proper range. Recap the bottle and restart agitation. Extract the free cyanide in this manner for a period of 60 min.

10.1.1.5 At the end of the extraction period, use a strip of pH test paper to verify that the sample mixture pH is 11 or greater. If the pH is in the correct range, the extraction may be deemed to be acceptable – proceed to Sec. 11.1.6. If it is not, extract a new sample by repeating Secs. 10.1.1-10.1.5, except using an initially higher-pH extraction solution (e.g., pH 13)

10.1.1.6 Following a successful extraction, decant the sample supernatant into a clean container. Filter the supernatant through a 0.45 µm membrane filter to remove particulate material and any PbS that may have formed.

10.1.1.7 If necessary, centrifuge the extraction mixture in order to remove fine particles and afford an adequate separation, as evidenced by a relatively clear final extract solution.

10.2 Initial Calibration

10.2.1 Preparation of calibration standards - Pipet 0, 0.20, 0.50, 1.00, 2.50, 5.00, 10.0, 15.0, and 30 mL of the 1.0-mg/L cyanide standard solution into nine separate 100-mL volumetric flasks. Dilute each of the flasks to volume with 4.1-g/L sodium hydroxide solution. These dilutions yield calibration standards that contain approximately 0, 2.0, 5.0, 10, 25, 50, 100, 150, and 300 µg/L cyanide, respectively.

NOTE: The 0 µg/L cyanide standard sample is used as the calibration blank.

10.2.2 To establish the calibration curve, analyze the calibration standards in accordance with

the procedure in Section 10.2. Prepare a duplicate set of spectrophotometric samples for each respective calibration standard (Sec. 10.3.2) to provide for duplicate spectrophotometric analyses of each standard. In order to avoid a method bias, the calibration standards must be analyzed directly and not processed through the microdiffusion procedure

10.2.3 Calculate the average absorbance reading for each duplicate set of calibration standards analyzed.

10.2.4 Establish the calibration curve by plotting average absorbance versus cyanide concentration. . A new calibration curve should be established daily with each analysis. Perform a first-order linear regression of the calibration curve (i.e., y = ax + b function). The acceptance criterion for the calibration curve should be a correlation coefficient of 0.995 or higher.

10.3 Equipment Operation and Sample Processing

10.3.1 Microdiffusion of free cyanide in aqueous samples and extracts

NOTE: At a minimum, perform microdiffusion of all samples in duplicate. More diffusion replicates can be performed, if desired, for greater statistical confidence in the final result.

10.3.1.1 Using a pipet or calibrated syringe, dispense 3.00 mL of sample into the outer chamber of a clean, dry microdiffusion cell (see Figure 2).

10.3.1.2 Using an adjustable pipet or calibrated syringe, dispense exactly 1.30 mL of 4.1-g/L sodium hydroxide solution into the center chamber of the microdiffusion cell (see Figure 2).

10.3.1.3 Smear the microdiffusion cell lid or other such cover with a sufficiently heavy layer of stopcock grease or petroleum jelly to prepare for airtight sealing of the microdiffusion cell, which occurs later in the test procedure. The most appropriate manner in which to apply the grease depends on the construction and geometry of the microdiffusion cell. In some cases a better air-tight seal may be achieved by applying the grease directly to the cell, instead of the lid. Either practice is considered to be acceptable.

NOTE: Use a liberal amount of stopcock grease or petroleum jelly in order to create an airtight seal between the microdiffusion cell and the lid.

10.3.1.4 Using a calibrated syringe (or pipet), immediately inject 1.0 mL of 190-g/L potassium phosphate solution into the sample located in the outer chamber of the microdiffusion cell. Inject at an angle in order to force the solution around the chamber.

NOTE: The force of ejection of aids in the proper mixing of the solutions.

10.3.1.5 Perform this step as quickly and carefully as possible in order to avoid loss of free cyanide: Carefully tilt and rotate the cell once to mix the constituents and immediately, use pH test paper to check the sample and ensure that it has attained a pH of 6. If the sample pH is greater than 6, add additional 190-g/L potassium phosphate solution, using 1.0-1.5 mL, increments, as necessary, to adjust the sample pH to 6.

NOTE: When analyzing an unknown sample, it is strongly recommended that the

sample be pre-tested prior to microdiffusion analysis in order to determine the exact amount of potassium phosphate solution needed to adjust the sample pH to 6. This may be accomplished by pipetting 3.0 mL of sample into a small container (e.g., volume capacity of 10 mL) followed by addition of 1.0 mL of 190-g/L potassium phosphate solution. After mixing the solutions well, determine the amount of potassium phosphate solution required to attain a pH of 6.

NOTE: The pH measurement step may be eliminated based upon historical knowledge of the sample.

10.3.1.6 Quickly, seal the cell by fitting it with a greased lid .Twist the lid and microdiffusion cell back and forth to ensure that the grease is evenly spread between the cell and the lid for an airtight seal.

10.3.1.7 Carefully tilt and rotate the cell for 5 seconds to ensure proper mixing.

NOTE: Exercise great care during mixing of solutions by tilting and rotating the microdiffusion cell carefully so as to avoid spilling or splashing the contents from one chamber of the cell to the other.

10.3.1.8 Place the covered cell in the dark at room temperature for a period of 6 hr.

NOTE: It is essential that the microdiffusion cell be protected from light at all times during the 6-hr diffusion period in order to avoid photolysis of hexacyanoferrate complexes.

10.3.1.9 Proceed to analyze the diffused samples and standards as described in Sec. 10.3.2.

10.3.2 Color development and spectrophotometric analysis of diffused samples and standards

NOTE: Ensure that the spectrophotometer cells used for analysis are optically-matched prior to use.

10.3.2.1 Prepare the spectrophotometer for analysis according to the manufacturer's instructions.

10.3.2.2 Set the spectrophotometer wavelength to 578 nm or other optimum wavelength, as determined by this method.

10.3.2.3 When the instrument has stabilized, zero the detector against the 4.1-g/L sodium hydroxide solution.

10.3.2.4 At the end of the diffusion period, pipet a 1.00-mL aliquot of the sodium hydroxide solution from the center chamber of the microdiffusion cell and dispense into a clean, dry, spectrophotometer cell.

CAUTION: Complete mixing as indicated in each of the following steps (Secs. 10.3.2.5- 10.3.2.7) is critical for accurate analysis.

10.3.2.5 Using a micropipet, dispense 0.1 mL of acidified potassium phosphate buffer into the spectrophotometer cell. Seal the cell with the stopper. The stopper should

form a watertight seal. While holding the stopper tightly against the cell, invert the cell 4-5 times to mix.

NOTE: Some spectrophotometer cell lids do not fit tightly and as such, mixing the cell contents through inversion can result in leaking and sample loss. A small piece of Parafilm M® may be used in place of the spectrophotometer stopper to provide a better seal and prevent leakage.

NOTE: In order to provide for more thorough mixing and at the same time prevent sample loss, Steps 10.3.4-10.3.7 may be carried out in a test tube instead of a spectrophotometer cell. After each addition of reagent, the sample test tube is vortexed (using a Vortex mixer) to stir the contents. Prior to spectrophotometric analysis (Sec. 10.3.8) the contents from the test tube are transferred to a clean, dry spectrophotometric cell.

10.3.2.6 Dispense 0.50 mL of chloramine-T reagent into the spectrophotometer cell using a micropipet. Seal the cell tightly again with the stopper and invert 4-5 times to mix.

10.3.2.7 Pipet 1.00 mL of pyridine-barbituric acid reagent into the spectrophotometer cell and replace the stopper. Note the time and invert the cell 8-10 times to mix.

10.3.2.8 Within 3-6 min from the start of mixing), the development of the color complex, as well as its stability, will be optimum for performing spectrophotometric readings. Read the absorbance of the spectrophotometric sample at 578 nm during this time interval. The time interval at which readings are taken must be consistent to the extent possible for all samples and standards.

NOTE: The wavelength maximum of the color-complex can vary slightly with each bottle or lot of barbituric acid reagent due solvatochromic effects. For optimum results, the absorbance maximum for the color complex can be determined by performing a spectral scan of a prepared 150-µg/L calibration standard prior to analysis each day. The sample and calibration standard absorbance readings may then be measured at the determined wavelength maximum, instead of at 578 nm.

10.3.2.9 Measure the absorbance for each sample replicate. Average the replicate results to yield a single absorbance value for each sample.

NOTE: The cells can be cleaned between usages by rinsing well with reagent water, followed by methanol. For a large number of determinations (10 or more), it may be necessary to periodically clean the cells with chromic-sulfuric acid cleaning solution or methanol-hydrochloric acid solution (prepare by mixing 3 volumes of water, 1 volume of concentrated HCI and 4 volumes of methanol). Avoid contact of these reagents with skin or eyes. Methanol may be used to dry the cell afterwards, if necessary.

WARNING: Keep any methanol used for cleaning purposes away from ignition sources.

NOTE: The total volume of liquid in the spectrophotometer cell is 2.6 mL. This is normally sufficient for measuring the absorbance in most spectrophotometers.

10.4 Continuing Calibration

Refer to Section 9.4.

10.5 Preventive Maintenance

An instrument service company calibrates the Spectrophotometers on a semi-annual basis. Certificates are kept on file.

11. Data Evaluation, Calculations and Reporting

11.1 For each spectrophotometric sample, obtain the µg/L free cyanide concentration from the calibration curve established in Sec. 10.2.

11.2 Determine the actual free cyanide concentration in an aqueous (or solid extract) sample by mathematically adjusting the free cyanide concentration determined in Sec. 11.1 to account for the concentration effect of the microdiffusion process as follows:

$$\frac{\mu g}{L}\ \text{free } CN^-\ \text{in aqueous sample} =$$

$$\frac{1.3\ \text{mL (center chamber solution volume)}}{3.0\ \text{mL (outer chamber solution volume)}} \times \frac{\mu g}{L}\ \text{free } CN^-\ \text{in spectrophotometric sample}$$

NOTE: Samples having a free cyanide concentration that exceeds that of the highest calibration standard, as determined in Sec. 11.1, must be diluted appropriately and reanalyzed spectrophotometrically Dilute the sample as needed using 4.1 g/L sodium hydroxide so that the concentration is within the calibration range. Analyze the diluted sample using a 3.00 mL aliquot as described in Section 10.3. Correct the final concentration result by multiplying by the appropriate dilution factor.

11.3 In the case of an extract, calculate the actual free cyanide concentration in the original solid that was extracted as follows:

$$\frac{\mu g}{g}\ \text{free } CN^-\ \text{in solid sample} = \frac{\mu g}{L}\ \text{free } CN^-\ \text{in extract sample} \times \frac{\text{Final extract solution volume (L)}}{\text{Sample mass (g)}}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Refer to Section 9.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734, 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)

SOP/1734, 1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

Figure 1: Microdiffusion Cell
Figure 2: Microdiffusion Process Overview

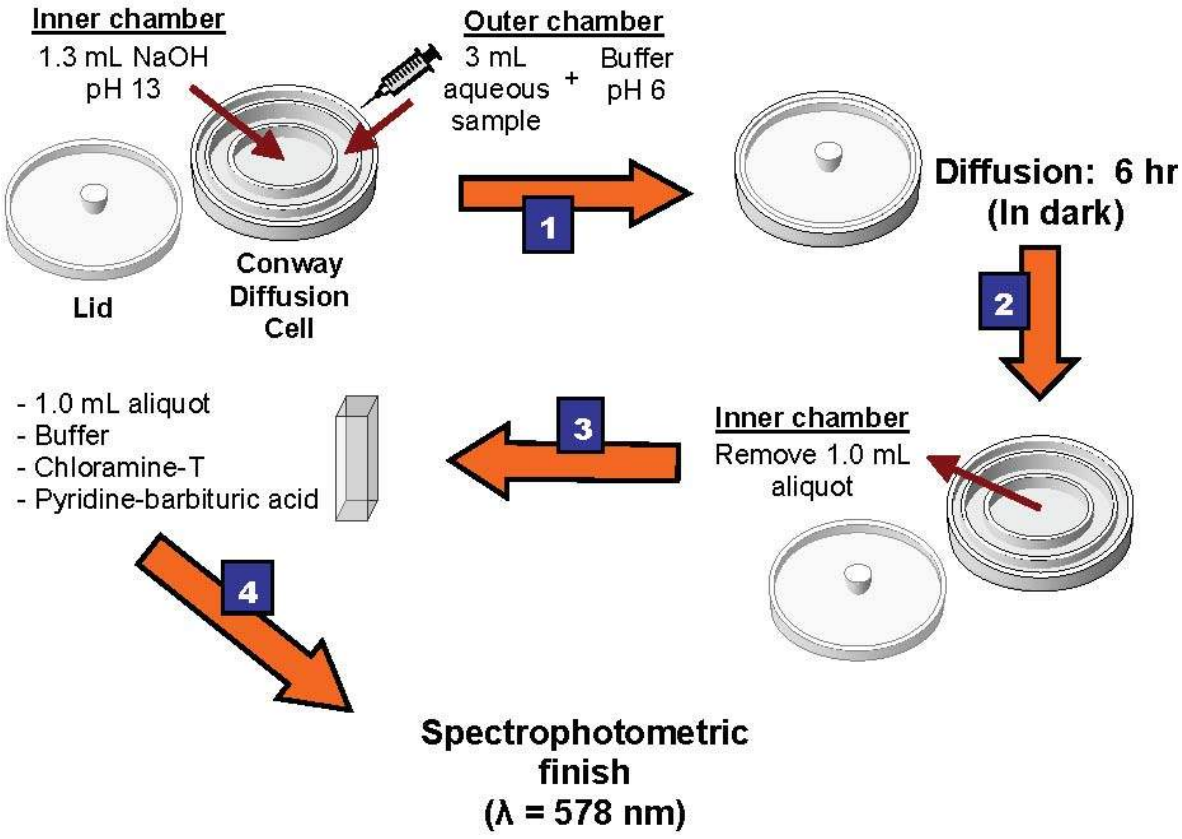
FIGURE 1

MICRODIFFUSION CELL

Center cell chamber for containment of sodium hydroxide absorber solution;
Outer cell chamber for introduction of sample;
Groove for holding and sealing cell cover; and
d Cell cover

FIGURE 2

MICRODIFFUSION PROCESS OVERVIEW



Standard Verification Procedure GTX- SVP- 13

Technical Information

Equipment Verified:	Atterberg limit device, grooving tool, gauge, glass plate
Applicable Test Methods:	ASTM D4318
Property:	Instructions for checking the critical dimensions and calibrating the equipment used to determine liquid and plastic limits of soils
Inspection Equipment Required:	Calipers, metal ruler, steel ball and resiliency tube, balance
Tolerances:	Calipers readable to 0.001 inches, metal ruler readable to 0.001 inches, 5/16 inch diameter steel ball and resiliency tube, balance with readability of 0.1 grams, wear on the base should not exceed 10 mm (3/8 in.), cup hanger should not be able to have side to side movement greater than 3 mm (1/8 in.)
Recalibrations:	Cup should be replaced when the grooving tool has worn a depression in the cup 0.1 mm (0.004 in.) deep, or when the rim has reached half of its original thickness. Recalibration was not specified.

Standard Operating Procedure:

Liquid Limit Device:

1. Check and record critical dimensions using calipers and ruler as shown in Figure 1 of ASTM D4318.

Base:

1. Check resilience of base with 5/16 inch steel ball by dropping ball from clear plastic tube conforming to requirements set forth in Appendix A of ASTM D4318.
2. Drop the ball to check at least three locations on the base, record the rebound height shown on the outside plastic case.
3. Check wear circle on base, the wear spot should not be worn greater than 10 mm (3/8 in.).

Cup:

1. Check and record weight of cup and hanger.
2. Verify that the cup is firmly attached to the cup hanger.

Cup Hanger:

1. Make sure that the cup hanger does not bind, or allow more than 3 mm (1/8 in.) side to side movement of the lowest point on the rim.

Grooving tool:

1. Inspect the grooving tools for wear on a regular basis.

2. Check and record critical dimensions using calipers as shown in Figure 2 of ASTM D4318.

Gauge:

1. Check and record critical dimensions using calipers as shown in Figure 3 of ASTM D4318.

Glass plate:

1. Check and record for ground glass plate dimensions (length, width, and thickness).

Remove old verification sticker and place a new updated sticker on the equipment with the current information.

Analysis of

Chlorinated Herbicides by GC Using Methylation Derivatization

Reference Methods: **EPA 8151A,** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1997.

MA BWS-CAM Section VC, Quality Assurance and Quality Control Requirements and Performance Standards for SW-846 Method 8151A, for the Massachusetts Contingency Plan (MCP). Revision 1. July 1, 2010.

SW-846 Chlorinated Herbicides by 8151, State of Connecticut Department of Environmental Protection Reasonable Confidence Protocol **(RCP)**, Version 2.0, July 2006

1. Scope and Application

Matrices: Aqueous, soil and waste.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Specifically, Method 8151A may be used to determine the following compounds:

Parameter		CAS No.[a]
2,4-D	[1, 2, 3]	94-75-7
2,4-DB		94-82-6
2,4,5-TP (silvex)	[1, 2, 3]	93-72-1
2,4,5-T	[1, 2]	93-76-5
Dalapon		75-99-0
Dicamba		1918-00-9
Dichloroprop		120-36-5
Dinoseb		88-85-7
MCPA		94-74-6
MCPP		93-65-2

[a] Chemical Abstract Service Registry Number

[1] RCRA List

[2] APA List

[3] TCLP List

Method 8151A is a capillary gas chromatographic (GC) method for determining certain chlorinated acid herbicides and related compounds in aqueous, soil and waste matrices.

Because these compounds are produced and used in various forms (i.e., acid, salt, ester, etc.), Method 8151A describes a hydrolysis step than can be used to convert herbicide esters into the acid form prior to analysis. Herbicide esters generally have a half-life of less than one week in soil.

When Method 8151A is used to analyze unfamiliar samples, compound identifications should be supported by at least one additional qualitative technique. Section 10.1 provides gas

chromatograph/mass spectrometer (GC/MS) criteria appropriate for the qualitative confirmation of compound identifications.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the use of gas chromatography and skilled in the interpretation of gas chromatograms. Only experienced analysts should be allowed to work with diazomethane due to the potential hazards associated with its use (explosive, carcinogenic). Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

This SOP for Method 8151A details gas chromatographic conditions for the analysis of chlorinated acid herbicides in water, soil, and waste samples. The hydrolysis of esters is also described in SOP number 1964.

Water, soil, and waste samples are extracted with diethyl ether and then derivatized with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

3. Reporting Limits

The sensitivity of Method 8151A depends on the level of interferences, in addition to instrumental limitations. Table 1 lists the estimated GC/ECD detection limits that can be obtained in aqueous and soil matrices in the absence of interferences. Detection limits for a typical waste sample should be greater.

4. Interferences

4.1 Refer to Method 8000 for instrumental interferences.

4.2 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts or elevated baselines in gas chromatograms. All these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis, by analyzing reagent blanks.

4.2.1 Glassware must be scrupulously cleaned. Clean each piece of glassware as soon as possible after use by rinsing it with the last solvent used in it. This should be followed by detergent washing with hot water and rinses with tap water, then with organic-free reagent water. Glassware should be solvent-rinsed with acetone and pesticide-quality hexane. After rinsing and drying, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store glassware inverted or capped with aluminum foil. Immediately prior to use, glassware should be rinsed with the next solvent to be used.

4.2.2 The use of high purity reagents and solvents helps to minimize interference problems.

4.3 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from waste to waste, depending upon the nature and diversity of the waste being sampled.

4.4 Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure.

4.5 Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with the electron capture analysis. However, hydrolysis may result in the loss of Dinoseb and the formation of aldol condensation products if any residual Acetone remains from the extraction of solids.

4.6 The herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. Therefore, glassware must be acid-rinsed and then rinsed to constant pH with organic-free reagent water. Sodium Sulfate must be acidified.

4.7 Sample extracts must be dry prior to methylation otherwise poor recoveries will be obtained.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling supplies, samples, extracts, standards or solvents, and when washing glassware.

5.2 All extract concentration steps must be performed in extraction hoods. All solvent and extract transfers must also be handled in a hood.

5.3 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the laboratory for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods. All ether bottles are to be checked for peroxide immediately upon opening.

5.5 All waste solvents must be transferred to the satellite waste storage containers located in the extraction laboratory. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.6 Inspect all glassware prior to use. Do not use any glassware that is chipped or cracked if it could present a safety hazard. Damaged glassware is put aside for repair; otherwise the piece is discarded. A number is etched onto each KD concentrator tube to keep track of calibration (Refer to SOP/08-04, KD Tube Calibration).

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two one-liter amber glass containers. Soil samples are collected in 8oz. glass jars.

6.2 Sample Preservation

Both aqueous and soil/solid samples are stored in refrigerators to maintain a temperature of 2 – 6 °C.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Both aqueous and soil/solid samples and extracts are stored under refrigeration (2 – 6 ºC) and protected from light. Aqueous samples must be extracted within 7 days from the time of collection; soil samples, within 14 days from collection. Sample extracts must be analyzed within 40 days following extraction.

After the extraction is completed, the sample extract is stored in a stoppered KD tube before being transferred to a crimp-top or screw cap vial for analysis and long-term storage.

7. Equipment and Supplies

7.1 Gas Chromatograph, Agilent 7890 (or equivalent): An analytical system complete with gas chromatograph configured for split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system.

7.2 GC Columns: Alpha utilizes dual-column analyses. The dual-column approach involves either a single injection that is split between two columns that are mounted in a single gas chromatograph, or dual injections of the split extract on a single GC equipped with two columns. Typical column pairs used are listed below. Other columns may be used as long as method performance criteria can be met.

Column Pair:

STX-CLP: Cat.: #11546 from Restek or equivalent, 30m, 0.32mm, 0.32 µm
STX-CLPII: Cat.: #11444 from Restek or equivalent, 30m, 0.32mm, 0.25µm

7.3 Guard Column: Cat. #10027 from Restek or equivalent; 5m, 0.32mm

7.4 Class “A” Volumetric Flasks: 10mL and 25mL (and other volumes), for standards preparation

7.5 Microsyringes: 10 µL – 1000 µL

7.6 Gooseneck splitless injecton liner, Cat #23303 from Restek or equivalent

7.7 Universal “Y” Press-tight tee split: Cat. #20406 from Restek or equivalent **/ Siltek MXT Connector:** Cat. #21388 from Restek or equivalent

8. Reagents and Standards

Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4±2 °C in Teflon(R)-sealed containers in the dark. When a large volume of standard is prepared, aliquots of that lot are stored in individual small vials.

All stock standard solutions must be replaced ***after one year or sooner*** if routine QC tests indicate a problem.

All other standard solutions must be replaced ***after six months or sooner*** if routine QC indicates a problem.

8.1 Reagent water: All references to water in this method refer to reagent water from Alpha's DI water treatment system.

8.2 n-Hexane: Pesticide grade or equivalent

8.3 Acetone: Pesticide grade or equivalent

8.4 Internal Standard Stock Solution: 4,4'-dibromooctafluorobiphenyl (DBOB) (AccuStandard, Cat # M-8151-IS) is used as the internal standard.

- **Working Internal Standard Solution**: To a 10mL volumetric flask, add 1mL of the 4,4'-dibromooctafluorobiphenyl (DBOB) (AccuStandard, Cat # M-8151-IS) and bring to volume with Acetone. Final concentration is 25µg/mL. 10µL of this solution is added to the 1mL sample and QC sample extracts as well as to all Calibration Standards to achieve a concentration of 0.25µg/mL for analysis.

8.5 Calibration Standards: Appropriate vendor derivatized standard(s) are volumetrically diluted with Hexane to obtain Initial Calibration Stock Standard which is used to prepare Initial Calibration Working Standards.

8.5.1 Initial Calibration Stock Standard: to a 10mL volumetric flask, add few mLs of Hexane:

- **1mL 2,4-Dichlorophenyl acetic acid (DCAA) methyl ester solution: (**100µg/mL in MTBE, Accustandard, Cat # M515-SS) and
- **1mL of Chlorinated Herbicides Methyl Derivatives** (100/10000µg/mL in MTBE, Accustandard, Cat # M-8151) Bring to volume using Hexane. Final concentration is 10µg/mL for all analytes except MCPP and MCPA at 1000µg/mL.

8.5.1.1 Initial Calibration Working Standards

8.5.1.1.1 Cal Level 1: To a 10mL volumetric flask, add 50µL of Initial Calibration Stock Standard. Bring to volume with Hexane. Final concentration is 0.05µg/mL for all analytes except MCPP and MCPA at 5.0µg/mL.

8.5.1.1.2 Cal Level 2: To a 10mL volumetric flask, add 100µL of Initial Calibration Stock Standard. Bring to volume with Hexane. Final concentration is 0.10µg/mL for all analytes except MCPP and MCPA at 10.0µg/mL.

8.5.1.1.3 Cal Level 3: To a 10mL volumetric flask, add 200µL of Initial Calibration Stock Standard. Bring to volume with Hexane. Final concentration is 0.20µg/mL for all analytes except MCPP and MCPA at 20.0µg/mL.

8.5.1.1.4 Cal Level 4: To a 10mL volumetric flask, add 500µL of Initial Calibration Stock Standard. Bring to volume with Hexane. Final concentration is 0.50µg/mL for all analytes except MCPP and MCPA at 50.0µg/mL.

8.5.1.1.5 Cal Level 5: To a 10mL volumetric flask, add 1.0mL of Initial Calibration Stock Standard. Bring to volume with Hexane. Final concentration is 1.0µg/mL for all analytes except MCPP and MCPA at 100µg/mL.

8.5.1.1.6 Cal Level 6: To a 10mL volumetric flask, add 2.0mL of Initial Calibration Stock Standard. Bring to volume with Hexane. Final concentration is 2.0µg/mL for all analytes except MCPP and MCPA at 200µg/mL.

8.6 Second Source Standard: (ICV) Appropriate vendor derivatized standard(s) are volumetrically diluted with Hexane to obtain Initial Calibration Verification Stock Standard which is used to prepare ICV Working Standard.

8.6.1 ICV Stock Standard: to a 10mL volumetric flask, add a few mLs of Acetone (to ensure mix of Methanol from the Herbicides Mixture):

- **1mL of Methylated Herbicides Mixture** (100/10000µg/mL in Methanol, Agilent, Cat # HBM-8151M) and
- **1mL of DCAA Methyl Ester Solution: 2,4-Dichlorophenyl acetic acid (DCAA) methyl ester solution –SURROGATE (**100µg/mL in Acetone, Agilent, Cat # PPS-166). Bring to volume using Hexane. Final concentration is 10µg/mL for all analytes except MCPP and MCPA at 1000µg/mL.

8.6.1.1 ICV Working Standard: To a 50mL volumetric flask, add 1mL ICV Stock Solution. Bring to volume with Hexane. Final concentration is 0.2µg/mL for all analytes except MCPP and MCPA at 20µg/mL.

8.6.2 ICV Standard (2,4-DB only): to a 50mL volumetric flask, add few mLs of Hexane and **2,4-DB methyl ester** (1000µg/mL in Hexane, Restek, Cat # 70931). Bring to final volume using Hexane. Final concentration is 0.2µg/mL.

8.7 Extraction Surrogate Standard: 2,4-Diclorophenylacetic Acid (DCAA) (1000µg/mL in Acetone, AccuStandard, M-8150B-SS-10X) The Surrogate standard is spiked into all samples and QC samples prior to extraction.

- To a 1000mL volumetric flask, add 10mL of ampulated surrogate solution. Bring to volume with Acetone. Final concentration is 10µg/mL.

8.8 LCS/MS Spiking Solutions: The LCS/MS spiking solutions are prepared volumetrically by diluting Underivatized Chlorinated Herbicides (100/10000µg/mL in Acetone, AccuStandard, Cat # M8151A.) in Acetone

- To a 200mL volumetric flask, add 20mL of ampulated LCS/MS solution. Bring to volume with Acetone. Final concentration is 10µg/mL for all analytes except MCPP and MCPA at 1000µg/mL.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A matrix-specific extraction blank is performed with each extraction batch of 20 or fewer samples, according to the extraction SOPs. The extraction blank must not contain any of the reportable analytes at or above the reporting limit. If any reportable analytes are detected in the blank, the entire extraction batch is suspect and re-extraction of all associated samples is required, unless the associated samples are non-detect or concentration of the analyte in the samples is 10 times greater than the concentration of this analyte in the blank. The surrogate recoveries must also be within the acceptance criteria listed in Table 3. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary. Actions associated with blank failures are listed in Table 4.

9.2 Laboratory Control Sample (LCS) / LCS Duplicate

A Laboratory Control Sample (LCS) and an LCS Duplicate (LCSD) is extracted with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with all target analytes. The concentrations of the spiking solutions are listed in Table 2. The recovery and RPD acceptance criteria are listed in Table 3. If any recovery criteria are not met, the extract should be reanalyzed. If the criteria are still not met, the entire batch should be re-extracted, unless the recoveries are high and the associated samples are non-detect. If this is not possible, due to insufficient sample or holding time exceedance, the analyst must write up the failure on a narrative sheet for inclusion in the client report. Actions associated with LCS/LCSD failures are listed in Table 4.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.7

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4

9.5 Matrix Spike (MS)/MS Duplicate

A matrix spike/matrix spike duplicate pair is extracted and analyzed for each batch of 20 or less samples only upon client request. MS and MSDs are field samples spiked with a known quantity of the target analyte(s). They are prepared by taking additional sample aliquots of the same weight or volume and adding the appropriate amounts of surrogate and spiking solutions. The spike compounds and levels are listed in Table 2. The recovery and RPD acceptance criteria are listed in Table 3. If the recovery criteria are not met, but are met in the LCS, the

failure may be attributed to sample matrix. Actions associated with MS/MSD failure are listed in Table 4.

9.6 Laboratory Duplicate (DUP)

A duplicate sample is extracted and analyzed upon Client request for each batch of 20 or less samples. Duplicates are laboratory selected replicate samples, prepared by taking an additional sample aliquot of the same weight or volume of a sample. The % RPD criteria are listed in Table 3. If the %RPD is not met, the failure may be attributed to sample matrix effects and must be noted on a narrative sheet for inclusion in the client report. Actions associated with duplicate failures are listed in Table 4.

For samples with a state of origin of New Jersey, a MS and MSD/DUP must be extracted for every twenty samples within a 24hr period.

9.7 Extraction Surrogates

All extracted samples and associated QC are spiked with surrogates at the levels listed in Table 2. The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate control limits for both columns listed in Table 3. If the surrogate limits are not met, the extract should be reanalyzed to determine if the failure was due to an instrument problem. If the criteria are still not met, the affected samples should be re-extracted to confirm that the failure was due to sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

9.8 Method Sequence

Typical Initial Calibration (each level has to be identified with the standard lot number)

1. Instrument Prime
2. Blank
3. Std Level 1
4. Std Level 2
5. Std Level 3
6. Std Level 4
7. Std Level 5
8. Std Level 6
9. Initial Calibration Verification Standard (ICV)
10. Initial Calibration Verification Standard (ICV)

Typical Daily Sequence:

1. Instrument Prime
2. Blank
3. Herbicide Continuing Calibration Standard **(identified with the standard lot number**)
4. Extraction Blank
5. Laboratory Control Sample
6. Laboratory Control Sample Duplicate
7. Matrix Spike (Upon Client Request)
8. Duplicate/Matrix Spike Duplicate (Upon Client Request)
9. Samples (10 field samples)
10. Repeat 3 – 9 (as needed)

10. Procedure

10.1 Equipment Set-up

The dual-column / dual-detector approach involves the use of the columns listed in section 7.2. The columns are connected to an Y-split or similar or dual injection GC, and separate electron capture detectors. Alpha typical GC conditions are listed below but may be altered as long as method performance criteria are met.

Temperature 1:	80 °C	**Detector temperature:**	350 °C
Ramp 1:	15°C/minute	**Carrier gas:**	Hydrogen
Temperature 2:	200°C	**Carrier flow:**	2.5 mL/min
Ramp 2:	20°C/minute	**Carrier mode:**	Constant flow
Temperature 3:	280°C	**Makeup gas:**	Nitrogen
Hold Time:	3 minutes	**Total detector flow:**	55 mL/min
Injector Temperature:	250°C		
Injector Mode:	Split		
Injector Flow:	36.5 mL/min		
Septum Purge Flow:	24 mL/min		
Split Flow:	10 mL/min		
Split Ratio:	4:1		
Injection Volume	1 µL		

10.2 Initial Calibration

10.2.1 Prepare calibration standards using the procedures in Section 8.5 and Table 2. The calibration standards are aliquoted into autosampler vials and capped prior to loading onto the autosampler tray. The calibration standards are analyzed and the curve is calculated prior to sample analysis.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in section 10.1. The same operating conditions are used for calibrations and sample analyses. Create the analytical sequence using the Agilent Chemstation data acquisition software. Record the calibration standard, unique lot number (PP#) and analyst's initials in the analytical sequence list.

10.2.3 A 1µL injection volume of each calibration standard is typically used. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume must be used for all standards and samples.

10.2.4 Column adsorption may be a problem when the GC has not been used for a day or more or after system maintenance. The GC column may be primed (or deactivated) by injecting a Herbicides standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

Alternately, the system may be primed by baking at the final analytical temperature for approximately 30 minutes.

Several analytes may be observed in the injection just following system priming. Always run an instrument blank after system priming.

10.2.5 Calibration Factors

Internal standard calibration techniques are employed in this method.

10.2.5.1 Internal Standard Procedure. In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

10.2.6 Initial Calibration Criteria

- If the **RSD for an analyte is < 20%,** then the response of the instrument for this compound is considered linear over the range and the mean calibration factor (Avg. RF) can be used to quantitate sample results.

- If the **RSD for any analyte is > 20%**, then linearity through the origin cannot be assumed. The mean response factor cannot be used for quantitation. An alternative calculation may be done by the use of **linear regression** or **quadratic regression** (minimum of six ICAL points are needed and regression must be weighted inversely proportional to concentration) as long as the **correlation coefficient is >0.990**. If both of these quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated and a new calibration curve must be analyzed. If quadratic regression is used for calibration, this must be noted in the laboratory narrative. The regression equation cannot be forced through the origin. The equation for linear regression may be found in Alpha's Quality Manual.

- **MCP requirement:** If linear or non-linear regression is used, RL must to be verified by recalculating concentrations in the lowest calibration standard using the final calibration curve. Recoveries must be **70-130%.**

- Minimum of five standards must be used for the linear regression and minimum of six for non-linear regression.

- Percent Relative Error (%RE): For linear and quadratic fit compounds, the %RE must be calculated for the standard at or near the midpoint and at the lowest level of the initial calibration. The %RE between the calculated and expected concentration of an analyte must be ≤ 30%, otherwise recalibration is necessary.

10.2.7 Initial Calibration Verification

An Initial Calibration Verification standards must be run immediately after each initial calibration, near the midpoint of the curve. The standards must be prepared using a second source that is different than the source used for the initial calibration. (Standards listed in Section 8.6). The **%D** has to be within **20%** (**15% for CT RCP**) when compared to the mean response factor from the initial calibration.

10.2.8 Retention Time Windows

- The retention time windows used for the identification of target analytes is ± 0.03 minutes. These criteria have been adopted from the EPA CLP Statement of Work (OLM04.2). It has been found that these limits work well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results.
- The windows listed above are used as guidance, however the experience of the analyst weighs heavily in the interpretation of the chromatograms. For example, it has been observed that certain oil matrices can cause the retention times to shift more dramatically. Additionally, if any positive results are questionable and at a sufficiently high concentration, GC/MS analysis is used for confirmation.

10.3 Sample Processing

10.3.1 The same GC operating conditions used for the initial calibration must be employed for sample analyses, including sample injection volume.

10.3.2 Tentative identification of an analyte occurs when a peak from a sample extract falls within the retention time window for the compound. Each tentative identification is confirmed using a second GC column of dissimilar stationary phase. In particularly difficult matrices, confirmation by GC/MS may be advisable.

10.3.3 The concentration reported for an identified target analyte in an extract is calculated using the Enviroquant data processing software. The Enviroquant methods have been configured to utilize the quantitation formulas found in Alpha's Quality Manual. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined. See the Manual Integration SOP for integration guidelines. Also refer to Section 11 for calculations.

10.3.3.1 If the responses exceed the calibration range of the system, dilute the extract and reanalyze.

10.3.4 The laboratory must report **HIGHER** of the two results unless obvious interference is present on one of the columns.

10.3.4.1 The **Relative Percent Difference (RPD)** should be 40 or lower. If the RPD exceeds 40, this will be denoted with a P flag. If the RPD exceedance is due to interference, the lower of the dual column values can be reported with I and P flags.

10.3.5 If compound identification or quantitation is precluded due to interference (e.g. broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be needed. If instrument problems are suspected, rerun the extract on another instrument to determine if the problem results from analytical hardware or

the sample matrix. Refer to the extraction SOPs for the procedures to be followed in sample cleanup.

10.3.6 GC/MS Confirmation

GC/MS confirmation may be used in conjunction with either single-column or dual-column analysis if the concentration is sufficient for detection by GC/MS.

10.4 Continuing Calibration

10.4.1 Verify calibration each **12-hours** shift by injecting Continuing Calibration standard prior to conducting any sample analyses. A calibration standard must also be injected at intervals of **not less than once every 10 filed samples.**

A bracketing CCV is not required with the use of internal standard calibration method with the exception of samples ran under CT RCP method.

10.4.2 The Response Factor (for internal standard compounds) for each analyte to be quantitated must not exceed a **± 20% difference (± 15% for MCP and CT RCP**) when compared to the initial calibration curve. The Enviroquant data processing software automatically calculates the %D for all analytes according to the formula in Alpha's Quality Manual.

If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.4.3 If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration must be performed. Due to the large number of analytes present, allowances may be made for a CF or RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples.

10.5 Internal Standard

The use of internal standard calibration does not require that all sample results be bracketed with CCV standard. However, when internal standard calibration is used, the retention times of internal standards and the area response of internal standards should be checked for each analysis.

10.5.1 **IS in CCAL** – The measured area of the internal standard must be no more than + 50% different from the average area calculated during initial calibration (-50 to 150%).

10.5.2 **IS in samples** - The measured area of the internal standard must be no more than -50% to +100% different from the area calculated from opening CCV (-50 to 200%)

Retention time shifts of more than 30 sec from the retention time of the most recent calibration standard are cause for concern and must be investigated.

10.6 Preventive Maintenance

- Routine preventive maintenance should be performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and replacement of injector seals. All maintenance is noted in individual Instrument Maintenance Logbooks.
- Other Maintenance: ECD detectors may become contaminated, requiring bake out at elevated temperatures, (no greater than 375℃) or repair by the manufacturer.

11. Data Evaluation, Calculations and Reporting

Herbicides are calculated as described in Section 11.1, 11.2, and reported in µg/L or µg/Kg units. After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. Two levels of review of the data in the LIMS system are preferred prior to release of data. These reviews include analyst review and review by the Department Supervisor or other trained analyst.

11.1 Aqueous Calculation

$$\mu g/L = \frac{(A_s)\,(C_{IS})\,(D)\,(V_f)}{(A_{IS})\,(\overline{RF})\,(V_s)\,(1000)}$$

Where:

A_s = Peak area of analyte
A_{IS} = Peak area of Internal Standard
C_{IS} = Concentration of IS (µg/L)
D = Dilution factor (D = 1 for no dilution)
V_f = Final extract volume (mL)
Vs = Initial volume extracted (mL)

$\overline{RF}$ = Mean response factor

11.2 Soil/Solid Calculation

$$\mu g/Kg = \frac{(A_s)\,(C_{IS})\,(D)\,(V_f)}{(A_{IS})\,(\overline{RF})\,(W_s)\,(1000)}$$

Where:

A_s = Peak area of analyte
A_{IS} = Peak area of Internal Standard
C_{IS} = Concentration of IS (µg/L)
D = Dilution factor (D = 1 for no dilution)
V_f = Final extract volume (mL)
Ws = Initial weight extracted (g)

$\overline{RF}$ = Mean response factor

11.2.1 Response Factor Calculation

$$RF = \frac{(A_s)\,(C_{IS})}{(A_{IS})\,(C_s)}$$

Where:

A_s = Peak area of analyte
A_{IS} = Peak area of Internal Standard
C_s = Concentration of analyte (µg/L)
C_{IS} = Concentration of IS (µg/L)

11.3 Molecular Weight Correction

In addition, conversion factors for the molecular weights have been listed for all analytes reported under EPA Method 8151.

The calibration is performed using the methyl ester compounds; therefore the calculation of the concentration includes a correction for the molecular weight of the methyl ester versus the acid herbicide.

Derivatized herbicides are used for the standard curve, and they are supplied as methyl esters as opposed to acid herbicide form which is used for the LCS. The difference between the acid form and the methyl ester is + 14 (i.e. In Derivatization, a $-CH_3$ group replaces the -H, (15-1 = 14)). The factors used for correction (listed in the Table below) vary for each herbicide and are applied to the standard concentration at each level of the calibration within the target processing software.

Example: 2,4-D

2,4-D MW = 221.04 2,4-D Methyl ester MW = 235.06

If you spike 10 ug of 2,4-D, and derivatized it, you end up with 10.634 ug of 2,4-D Methyl ester that you are measure, as you've stripped the -H and added a -CH3, increasing the molecular weight. The true value for the spike is 10 ug as the acid herbicide, and 10.634 ug as the methyl ester. So a spike of 10 ug of acid, would measure 10.634 ug of the methyl ester to get 100% recovery. The true value for the spike would be 10.634 as methyl ester, which you use to divide your measured value by.

If measuring recoveries, the value of the 10 ug of the methyl ester, would be 10 ug measured (as methyl ester)/10.634 ug spiked (as methyl ester) = 94.04%.

If, on the other hand you want to convert your final result using a correction factor, you would use the MW of the acid/MW of the ester (221.04/235.06 = 0.94).

Conversion Factors applied in EQ software:

Parameter	CAS No.[a]	MOL WT.	WT as methyl ester	Conversion Factor
2,4-D [1, 2, 3]	94-75-7	221.04	235.06	0.94
2,4-DB	94-82-6	326.18	340.18	0.96
2,4,5-TP (silvex) [1, 2, 3]	93-72-1	269.51	283.51	0.95
2,4,5-T [1, 2]	93-76-5	255.48	269.48	0.95
Dalapon	75-99-0	142.96	156.96	0.91
Dicamba	1918-00-9	221.04	235.04	0.94
Dichloroprop	120-36-5	235.06	249.06	0.94
Dinoseb	88-85-7	240.21	254.21	0.95
MCPA	94-74-6	200.62	214.62	0.93
MCPP	93-65-2	214.65	228.65	0.94

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Refer to Section 10 for specific corrective actions.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Qualtrax Document ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

- Chemical Hygiene Plan
- Qualtrax ID 1732 MDL/LOQ Generation
- Qualtrax ID 1739 IDC/DOC Generation
- Qualtrax ID 1728 Waste Management and Disposal SOP

16. Attachments

Table 1: REPORTING LIMITS

Table 2: STANDARD SOLUTIONS

Table 3: QC ACCEPTANCE CRITERIA

Table 4: QC FAILURE ACTION PLAN

TABLE 1

REPORTING LIMITS

Herbicides	RL (Aqueous)	RL (Soil)
2,4-D	1.0 ug/L	33 ug/kg
Dalapon	1.0 ug/L	33 ug/kg
2,4-DB	1.0 ug/L	33 ug/kg
Dicamba	1.0 ug/L	33 ug/kg
Dichloroprop	1.0 ug/L	33 ug/kg
Dinoseb	1.0 ug/L	33 ug/kg
MCPA	100.0 ug/L	3300 ug/kg
MCPP	100.0 ug/L	3300 ug/kg
Silvex (2,4,5-TP)	1.0 ug/L	33 ug/kg
2,4,5-T	1.0 ug/L	33 ug/kg

TABLE 2

STANDARD SOLUTIONS

Compound	Level 6	Level 5	Level 4	Level 3	Level 2	Level 1	Std. Spike Concentration *	Std. LCS Concentration *
	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)	(µg/mL)
2,4-D	2.0	1.0	0.5	0.2	0.1	0.05	10	10
Dalapon	2.0	1.0	0.5	0.2	0.1	0.05	10	10
2,4-DB	2.0	1.0	0.5	0.2	0.1	0.05	10	10
Dicamba	2.0	1.0	0.5	0.2	0.1	0.05	10	10
Dichloroprop	2.0	1.0	0.5	0.2	0.1	0.05	10	10
Dinoseb	2.0	1.0	0.5	0.2	0.1	0.05	10	10
MCPA	200	100	50	20	10	5	1000	1000
MCPP	200	100	50	20	10	5	1000	1000
Silvex (2,4,5-TP)	2.0	1.0	0.5	0.2	0.1	0.05	10	10
2,4,5-T	2.0	1.0	0.5	0.2	0.1	0.05	10	10
DCAA (Surrogate)	2.0	1.0	0.5	0.2	0.1	0.05	10	10

***** Calculations are based on an initial aqueous sample volume of 1L**,** initial soil sample weight of 30g, and final extract volume of 10mL.

TABLE 3

QC ACCEPTANCE CRITERIA

	Aqueous/Soil	
Surrogate % Recovery	**Lower Control Limit**	**Upper Control Limit**
2,4-Diclorophenylacetic acid (DCAA)	30%	150%

	% Recovery		% RPD		MCP %RPD	
LCS/LCSD	**Lower Control Limit**	**Upper Control Limit**	**waters**	**soils**	**waters**	**soils**
2,4-D	40%	140%	≤ 50%	≤ 50%	≤ 20%	≤ 30%
dalapon						
2,4-DB						
dicamba						
dichloroprop						
dinoseb						
MCPA						
MCPP						
Silvex (2,4,5-TP)						
2,4,5-T						

	% Recovery		% RPD		MCP % RPD	
MS / MSD	**Lower Control Limit**	**Upper Control Limit**	**waters**	**soils**	**waters**	**soils**
2,4-D	30%	150%	≤ 30%	≤ 30%	≤ 20%	≤ 30%
dalapon						
2,4-DB						
dicamba						
dichloroprop						
dinoseb						
MCPA						
MCPP						
Silvex (2,4,5-TP)						
2,4,5-T						

TABLE 4

QC FAILURE ACTION PLAN

Blank Failures	Failure Situation	Action(s)
Surrogate Failure	1 of 2 high or low	Report as is
	Both high	Report as is
	Both low, samples are non-detect with passing surrogates	Report as is
	Both low, samples have hits and/or failing surrogates	Revial/rerun Re-extract if still failing
IS Failure	IS not present (less than 10% recovery)	Add IS Rerun
	IS out high or low	Revial/rerun Re-extract if still failing
Contamination	Below RL; sample(s) non-detect, below RL, or concentration 10 times or more above the blank	Report as is
	Below RL, sample(s) concentration above RL	See team leader/manager
	Above RL, sample(s) concentration 10 times or more above the blank	Report as is
	Above RL with non-detect sample(s)	Report as is
	Above RL, sample(s) concentration less than 10 times above the blank and volume available	Revial/rerun Re-extract if contamination still present
	Above RL, sample(s) concentration less than 10 times above the blank and no volume available	Revial/rerun B-flag affected data

LCS/LCSD Failures	Failure Situation	Action(s)
Surrogate Failure	1 of 2 high or low	Report as is
	Both high or low	Report as is
IS Failure	IS not present (less than 10% recovery)	Add IS Rerun
	IS out high or low	Revial/rerun Re-extract if still failing
Spike Failure	Spike compounds out high and sample(s) are non-detect with passing surrogates	Report as is
	Spike compounds out high and sample(s) have hits	Revial/rerun Re-extract if still high
	Spike compounds out low	Revial/rerun Re-extract if still low
	RPD failure	Report as is

MS/MSD/DUP Failures	Failure Situation	Action(s)
Surrogate Failure	(MS/MSD) 1 of 2 high or low	Report as is
	(MS/MSD) Both high or low	Report as is
	(DUP) 1 or more high, parent sample non-detect	Report as is
	(DUP) 1 of 2 low, parent sample matches	Report as is
	(DUP) 1 or more low, parent sample doesn't match	Revial/rerun Re-extract if still failing
IS Failure	IS not present (less than 10% recovery)	Add IS Rerun
	IS out high or low	Revial/rerun Re-extract if still failing
Spike Failure (MS/MSD only)	Compounds out high or low due to sample matrix or hits	Report as is
	Compounds out high or low due to extraction error	Revial/rerun Re-extract if still failing
	RDP failure	Report as is

Chromium, Hexavalent

References: **SM3500 Cr-B**, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

7196A, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update I, 1992.

3060A, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1996.

1. Scope and Application

Matrices: This method is applicable for the determination of hexavalent chromium in natural or treated water or in a soil or solid matrix.

Definitions: See Alpha Analytical Quality Manual Appendix A

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Spectrophotometer and in the interpretation of Spectrophotometric data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

The hexavalent chromium concentration of U.S. drinking waters has been reported to vary between 3 and 40µg/L with a mean of 3.2µg/L. Chromium salts are used extensively in industrial processes and may enter a water supply through the discharge of wastes. Chromate compounds frequently are added to cooling water for corrosion control. Chromium may exist in water supplies in both the hexavalent and the trivalent state although the trivalent form rarely occurs in potable water.

2. Summary of Method

This procedure measures only hexavalent chromium (Cr^{6+}). The hexavalent chromium is determined colorimetrically by reaction with diphenylcarbazide in acid solution. A very sensitive red-violet color of unknown composition is produced, and its absorbance is measured photometrically at 540nm.

2.1 Method Modifications from Reference

The coloring reagent used for the standards in the calibration curve and for the samples is made by dissolving 1 gram of 1,5-diphenylcarbazine in 250mL of acetone, whereas the reference method 7196A dissolves 1 gram in 200mL of acetone.

Alpha uses a smaller sample volume for analysis; therefore reagent volumes are reduced accordingly.

Alpha doesn't perform Blank correction;

2.5 cm (not 1cm) absorption cell is used for water samples to increase sensitivity.

Soil samples with failed soluble or insoluble spikes will be analyzed for PH and ORP to check if sample has reducing condition. If reducing condition confirm then lab will not Reprep asnd reanalyze samples with failed insoluble and soluble spikes

3. Reporting Limits

The RDL reported is 0.01mg/L for 50mL of an aqueous sample. The RDL reported is 0.8mg/Kg for 2.5g of a soil/solid sample. The RDLs are not adjusted for % Moisture.

4. Interferences

4.1 Background Color

Turbidity and background sample color are removed by filtering the sample through a 0.45µm filter. In cases when filtration alone is not sufficient, background interference can be subtracted out by adding all reagents to the sample except diphenylcarbazide solution, measuring the background absorbance, and then subtracting this value from the colored sample absorbance value.

4.2 Other Metals

The reaction with diphenylcarbazide is nearly specific for chromium. Hexavalent molybdenum and mercury salts will react to form color with the reagent but the intensities are much lower than that for chromium at the specified pH. Concentrations as high as 200 mg/L of Mo or Hg can be tolerated. Vanadium interferes strongly but concentrations up to 10 times that of chromium will not cause trouble. Potential interference from permanganate is eliminated by prior reduction with azide. Iron in concentrations greater than 1mg/L may produce a yellow color but the ferric ion (Fe^{3+}) color is not strong and no difficulty is encountered normally if the absorbance is measured photometrically at the appropriate wavelength. Interfering amounts of molybdenum, vanadium, iron, and copper can be removed by extraction of the cupferrates of these metals into chloroform ($CHCl_3$). A procedure for this extraction is provided in the reference but do not use it unless necessary because residual cupferron and $CHCl_3$ in the aqueous solution complicate the later oxidation. Therefore, follow the extraction by additional treatment with acid fuming to decompose these compounds.

4.3 Reducing/oxidizing tendency of sample matrix

When analyzing a sample digest for total Cr(VI), it is appropriate to determine the reducing/oxidizing tendency of each sample matrix, if the matrix spike for that sample matrix has failed the quality control acceptance criteria for this method. Determination of the Reducing/oxidizing potential for failing matrix spike samples can be accomplished by characterization of each sample for additional parameters, such as pH (Method 9045) and Oxidation Reduction Potential (ORP) (ASTM 1498-93 –aqueous samples). Method 9045 (Section 7.2 of Method 9045) is referenced as the preparatory method for soil samples. The ORP and temperature probes are inserted directly into the soil slurry derived from the pH analysis. The displayed ORP value is allowed to equilibrate and the resulting measurement is recorded. Analysis of these parameters establishes the tendency of Cr(VI) to exist or not exist in the unspiked sample(s) and assists in the interpretation of QC data for matrix spike recoveries outside conventionally accepted criteria for total metals.

4.4 Soluble Cr(III) concentration

For waste materials or soils containing soluble Cr(III) concentrations greater than four times the laboratory Cr(VI) reporting limit, Cr(VI) results obtained using this method may be biased high due to method-induced oxidation. The addition of Mg^{2+} in a phosphate buffer to the alkaline extraction solution has been shown to suppress this oxidation. If an analytical method for Cr(VI) is used that can correct for possible method induced oxidation/reduction, then the Mg^{2+} addition is optional. The presence of soluble Cr(III) can be approximated by extracting the sample with deionized water.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available.Hexachrome is toxic and suspected carcinogen. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in plastic or glass containers with teflon-lined caps. Sample containers must not contain any stainless steel. Separate container has to be provided for MCP HexCr (see MCP addendum)

6.2 Sample Preservation

Samples are unpreserved and refrigerated at 4 ± 2 °C.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Aqueous samples must be analyzed within 24 hours of collection.

Soil and solid samples must be digested within 30 days of collection. The extracts must be analyzed within 168 hours (7 days) after extraction.

7. Equipment and Supplies

7.1 Spectrophotometer: Genesys 10 Vis or equivalent: for use at 540nm, with a light path of 1cm or longer.

7.2 100 ml digestion cups

7.3 pH Meter: Calibrated.

7.4 Volumetric flasks: 1000mL and 100mL.

7.5 Filtration apparatus.

7.6 0.45µm Membrane filters.

7.7 Hot block Model # SC151 with Stir Base Stirring device (part #SC160)

7.8 Balance.

7.9 Thermometers.

7.10 Volumetric pipets: various volumes.

7.11 50mL Centrifuge Tubes.

7.12 100mL Graduated cylinder

7.13 pH paper range 1-14.

7.14 Centrifuge

8. Reagents and Standards

8.1 1000ppm Stock Hexavalent Chromium Solution (for calibration and LCS): Commercial purchase. Expire upon manufacturer's expiration date. Use different lots for calibration and LCS.

8.2 1ppm Working Standard Hexavalent Chromium Solution (for calibration): Dilute 1.00mL stock chromium solution (Section 8.1) to 1000mL with DI. Store at room temperature. Expires three months from the date of preparation.

8.3 10ppm Working Standard Hexavalent Chromium Solution (for LCS/ICV): Dilute 1.00mL stock chromium solution (Section 8.1) to 100mL with DI. Store at room temperature. Expires six months from the date of preparation

8.4 Nitric acid, HNO_3, concentrated. Do not use if has a yellow tinge. Expires upon manufacturer's expiration date.

8.5 Nitric Acid, 50%: Into a 500mL volumetric flask, dilute 250mL of Concentrated Nitric Acid with 250mL of DI water. Expires six months from the date of preparation.

8.6 Sulfuric acid, H_2SO_4, concentrated.

8.7 Sulfuric acid, H_2SO_4, 10%: Dilute 50mL concentrated H2SO4 to 500mL with DI water. Expires six months from the date of preparation.

8.8 Diphenylcarbazide solution: Dissolve 1.0g 1,5-diphenylcarbazide (1,5-diphenylcarbo-hydrazide) in 250mL acetone. Store in a plastic bottle cover with aluminum foil or away from light. Discard when solution becomes discolored. Prepare fresh weekly.

8.9 Sodium carbonate: Na_2CO_3, anhydrous, analytical reagent grade. Store at 20 – 25°C in a tightly sealed container.

8.10 Sodium hydroxide: NaOH, analytical reagent grade. Store at 20 – 25°C in a tightly sealed container.

8.11 Magnesium Chloride: $MgCl_2$ (anhydrous), analytical reagent grade. 392.18mg $MgCl_2$ is equivalent to 100mg Mg^{2+}. Store at 20 – 25°C in a tightly sealed container. As an alternate, use 0.8366g $MgCl_2 \cdot 6\ H_2O$.

8.11.1 Magnesium Chloride solution: Dissolve 84g of $MgCl_2 \cdot 6\ H_2O$ in 100mL of DI. Store at 20 – 25°C in a tightly sealed container. Expires six months from the date of preparation.

8.12 Phosphate Buffer:

8.12.1 K_2HPO_4: Analytical reagent grade.

8.12.2 KH_2PO_4: Analytical reagent grade.

8.12.3 0.5M K_2HPO_4 / 0.5M KH_2PO_4 buffer: Dissolve 87.09g K_2HPO_4 and 68.04g KH_2PO_4 into 700mL of DI water. Transfer to a 1L volumetric flask and dilute to volume. (When prepared accordingly, the buffer solution has an approximate pH value of 7.)Record pH

in the reagent log book. Store at 20 – 25°C in a tightly sealed container. Expire one year from prep.

8.13 Lead Chromate: $PbCrO_4$, analytical reagent grade. Store under dry conditions at 20 – 25°C in a tightly sealed container. Expires upon manufacturer's expiration date.

8.13.1 Soil matrix insoluble spiking solution: Add 10 – 20mg $PbCrO_4$ (Section 8.12) to a separate aliquot of sample. This spike is used to evaluate the dissolution during the digestion process.

8.14 Digestion solution: Dissolve 20.0 ± 0.05g NaOH and 30.0 ± 0.05g Na_2CO_3 in deionized water in a one-liter volumetric flask and dilute to the mark. Store the solution in a tightly capped polyethylene bottle at 20 – 25 °C and prepare fresh monthly. The pH of the digestion solution must be checked before using. The pH must be 11.5 or greater; if not, discard.

8.15 Potassium Dichromate, $K_2Cr_2O_7$, spiking solution (1000mg/L Cr (VI)): Commercial purchase. Expire upon manufacturer's expiration date.

8.15.1 100mg/L Cr (VI): Add 10.0mL of the 1000mg/L $K_2Cr_2O_7$ spiking solution (Section 8.15) to a 100mL volumetric flask and dilute to volume with deionized water. Mix well.

8.15.2 5ppm: To a 100mL volumetric flask, add 5mL of 100ppm spiking solution (Section 8.15.1), bring to volume with DI.

8.15.3 Soil sample soluble spike: Add 1 ml of 1000mg/L Cr (VI) spiking solution (Section 8.15) to 2.5g of sample.

8.15.4 Post spike soil sample: Add 1 ml of 100mg/L Cr(VI) solution (section 8.15.1) to 25 ml extracted sample.

8.15.5 Aqueous sample spike: Add 1ml of 5 ppm (section 8.15.2) to 50 ml of sample**.**

8.16 ICV and CCV Standards: Commercially prepared solution of known hexavalent chromium concentration. A Certificate of Analysis is kept on file. This solution is diluted with DI to a concentration 0.2 ppm on each day of use

8.17 Acetone: Analytical reagent grade.

8.18 Ottawa Sand

8.19 SRM HexCr standard: ERA QC-Cat#921. Expires upon manufacturer's expiration date

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

One preparation blank is digested and analyzed per batch of 20 samples or less for method 3500. Method 7196: batch size is 10 samples or less. The concentration of the Blank must be less than the RDL, or one-tenth the regulatory limit or action level, whichever is greater, otherwise the entire batch must be redigested and reanalyzed.
Method Blanks consist of DI water for liquid samples or 2.5g of Ottawa sand for soil samples.

The ICB consists of DI water that has been colored and analyzed as per Section 10.3.4. Results must be less than the RDL, otherwise another ICB is colored and analyzed. It failure continues, a new calibration curve is generated (Section 10.1).

9.2 Laboratory Control Sample (LCS)

One LCS is prepared and analyzed per batch of 20 samples or less for method 3500. Method 7196: batch size is 10 samples or less.

The LCS for aqueous samples consists of the ICV (Section 10.2). LCS recovery for liquids must be within 85 – 115%, otherwise the entire sample batch must be reanalyzed.

For solids samples, a soluble LCS and insoluble LCS must be digested per batch of 20 samples or less (Refer to Section 10.3.3.1) LCS recovery for solids must be within 80 – 120%, otherwise the sample batch must be redigested and reanalyzed.

Note: SRM HexCr standard made by ERA is used for soil LCS: use 0.2-0.5 g, based on certified value. Expires upon manufacturer's expiration date.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4

9.5 Matrix Spike

9.5.1 Aqueous samples

One sample per delivery group per batch of 20 samples (method 3500) or 10 samples (method 7196) or less is required to be spiked.

Example: If a batch consists of 6 liquid samples (3 wastewater from Client A and 3 wastewater from Client B), then one wastewater from Client A and one wastewater from Client B each require spiking.

The spike is prepared by adding 1mL of 5ppm aqueous spiking solution (Section 8.15.2) to 50mL of sample. Spike recovery must be within 85 – 115. If spike recovery is outside of the range, adjust sample pH to 8.5 then add 1 ml of 5 ppm spiking solution and reanalyze. If spike recovery still outside of the range, then dilute sample and retest both diluted sample and spiked diluted sample. Report sample results at the dilution for which the spike recovered at 85 – 115%.

Sample dilutions can be made with DI, up to 200X or up to the client's specified detection limits.

If interferences remain after dilutions, and spike recovery is still less than 85% for aqueous samples, refer to Section 10.3.5.

9.5.2 Soil / solid matrix samples

Two spikes per batch of 10 samples or less are required; one with a soluble spike and one with an insoluble spike (Section 10.3.3.1). The acceptance range for spike recovery is 75 – 125%. If recoveries are not within the specified limits, then samples will be analyzed for PH and ORP to confirm matrix interference. If samples have reducing condition then a re-digestion of the sample and matrix spikes will not be needed..

For NJ-HexCr packages: If recoveries are not within the specified limits, then the matrix spike(s) and the associated samples must be re-homogenized/re-digested/re-analyzed. If upon reanalysis, it is found that the recoveries are acceptable, then the data is submitted with the new spike results. If it is found that the recoveries are still unacceptable upon redigestion, then it is assumed that the sample matrix is suspect and the pH (SOP 2203) and ORP (SOP 2237) are measured at once for the sample that was spiked. (for NJ HexCr method). For MCP method, pH and ORP are measured within 24h of sample collection.

If prior knowledge of an interfering sample matrix is known, then matrix spike duplicates (MSD) are set for the soluble and insoluble matrix spikes, along with the pH and ORP analyses for the samples to be spiked. Failure of the MS/MSD will verify that the sample matrix interferes with the digestion and will not necessitate a re-digestion of the sample and matrix spikes.

SOP addendum must be checked for NJ HexCr and MCP HexCr methods for additional actions.

9.6 Laboratory Duplicate

Analyze one sample in duplicate per batch of 20 samples (method 3500) or 10 samples (method 7196) or less. The %RPD must be $\leq$ 20% for aqueous samples and $\leq$ 20% for solid samples, if both the original sample and the duplicate results are greater than or equal to four times the reporting limit. A control limit of ± the laboratory reporting limit is used when either the original sample result or the duplicate sample result is less than four times the laboratory reporting limit.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Sample preparation (digestion and/or pH adjustment and filtration)
- Coloring procedure
- Analysis on Spectrophotometer
- Verification
- Calculation

10. Procedure

10.1 Equipment Set-up

10.1.1 A calibration curve is generated at a minimum of once per year, as follows:

10.1.1.1 **For Liquid**: Pipet the following volumes of the 1ppm working standard (Section 8.2) into separate 50mL volumetric flasks: 0.25 ml, 0.5 ml, 1 ml, 2.5 ml, 5 ml, 10 ml and 25 ml. Bring each to volume with DI to give the following concentrations respectively: 0.005ppm, 0.01ppm, 0.02 ppm, 0.05 ppm, 0.10ppm, 0.20ppm and 0.50ppm. Include a 50mL DI blank and proceed with subsequent treatment of the standards in the same manner as samples (Section 10.3.2).

10.1.1.2 **For Soil**: Prepare five 100 ml digestion cups, add 1 ml of Magnesium Chloride solution (8.11.1) into each cup, then pipet following volumes of the 10 ppm working standard (section 8.3) into separate digestion cups:0 ml, 0.1mL, 1.0mL, 5.0 ml and 10mL. To all digestion cups add 50 ml of digestion solution and 0.5 ml of phosphate buffer. Proceed to soil/solid digestion and extraction procedure(sections 10.3.3.2 to 10.3.3.7)

10.1.1.3 Develop color for standards and blank as in Section 10.3.4.

10.1.1.4 Construct a calibration curve by plotting the absorbance values against mg/L hexavalent chromium. An acceptable curve must have a calibration coefficient of $\geq$ 0.995. This calibration curve is valid for one year, unless failure of an ICV requires construction of a new calibration curve. For method 3500: analyze RL verification standard (at 0.01 mg/l) following calibration. % recovery must be within +/-30% from true value.

Note: see MCP-HexCr and NJ-HexCr addendum for frequency of calibrations for MCP and NJ sites.

10.2 Initial Calibration

A non-digested, colored ICV (Section 8.16) (from a different source than was used for the calibration standards) must be recovered between 85 – 115%. If these criteria are not met, then the ICV is re-analyzed. If failure repeats, then the ICV is re-made and re-analyzed. If failure continues, prepare a new calibration curve (Section 10.1). See Section 12 for corrective actions.

10.3 Equipment Operation and Sample Processing

10.3.1 Treatment of sample: If interfering amounts of molybdenum, vanadium, copper, or iron are present, then follow the removal procedures as stated in the reference method.

10.3.2 Aqueous Samples:

Samples containing sediment, floating or suspended matter, or turbidity are filtered through a 0.45µm membrane filter prior to pH adjustment. If background interferences persist, measure an absorbance reading before adding diphenylcarbazide reagent (Section 10.3.4) and correct the absorbance of the final colored solution by subtracting the absorbance measured previously.

Proceed to Section 10.3.4.

10.3.3 Solid / soil sample digestion and extraction procedure:

10.3.3.1 **Samples:** Thoroughly mix the sample and weigh 2.5g +/- 0.1g into a 100 ml digestion cup. Add 1 ml of $MgCl_2$ solution.

10.3.3.2 **Blank:** Weigh 2.5g Ottawa Sand into a 100ml digestion cup, add 1 ml of $MgCl_2$ solution.

10.3.3.3 **Insoluble LCS:** Add 0.2-0.5 g of SRM (ERA catalog # 921) and 1 ml of $MgCl_2$ solution.

10.3.3.4 **Soluble LCS:** Weigh 2.5g Ottawa Sand into a 100ml digestion cup, add 1 ml of $MgCl_2$ solution and 1.0mL 1000ppm $K_2Cr_2O_7$. (Section 8.15).

10.3.3.5 **Matrix Spike, insoluble:** Thoroughly mix the sample and weigh 2.5g into a 100 ml digestion cup. Add 1 ml of $MgCl_2$ solution and 0.01 – 0.02g $PbCrO_4$ (Section 8.13.1).

Matrix Spike, soluble: Thoroughly mix the sample and weigh 2.5g into a 100mL digestion cup. Add 1 ml of $MgCl_2$ solution and 1.0mL 1000ppm $K_2Cr_2O_7$. (Section 8.15).

To all samples and QC samples, add 50mL of digestion solution (8.14) and 0.5mL of phosphate buffer (Section 8.12.3).

10.3.3.6 Stir the sample on a stir plate (unheated) for at least 5 minutes.

10.3.3.7 Place samples on hot block with Stirring base; heat samples for 1 hour at 90 – 95 °C (after temperature reached 90°C)

10.3.3.8 While still stirring, allow to cool to room temperature. Transfer the contents of the digestion vessel to 50 ml centrifuge tube then centrifuge for 6 minutes .Pour centrifuged solution back to original cup. Save the solid centrifuged material from the matrix spikes for possible later analysis for assessing low Cr (VI) recoveries. Store the centrifuged solid at 4 ±2°C.

10.3.3.9 While constantly stirring, very slowly add 50% HNO_3 (Section 8.4) to the centrifuged liquid to carefully adjust the pH to 7.5 ± 0.5. If the pH drops below 7.0, discard the solution and re-digest.

10.3.3.10 Remove the stir-bar and rinse keeping rinsate in the cup, and transfer quantitatively to a 100mL volumetric flask, 100 ml graduated cylinder, or 100mL digestion cup. Bring to volume with DI.

10.3.3.11 Proceed with coloring and analysis as described in Section 10.3.4.

10.3.4 Color development and measurement:

Place 45mL of sample or standard solution into a 50mL centrifuge tube. Adjust to pH 2 ± 0.5 with 10% H_2SO_4 (Section 8.7). Record pH on excel format. Add 1mL Diphenylcarbazide solution (Section 8.8) and adjust to 50mL with DI water. Mix and let stand 5 – 10 minutes for full color development.

Set the spectrophotometer to zero at 540 nm, utilizing the colored reagent blank in a 1cm absorption cell for soil samples and 2.5 cm absorption call for waters. Next, transfer a suitable portion of each colored standard solution or sample to a 1cm absorption cell for soils and 2.5 cm absorption cell for waters. Measure and record each absorbance at 540nm. Determine mg/L chromium present by reference to the calibration curve (Section 10.1.1.3).

10.3.4.1 Dilute samples volumetrically with DI water if they are more concentrated than the highest standard in the calibration curve.

10.3.4.2 When necessary samples precipitate poured off for analysis.

10.3.5 Verification of Samples

10.3.5.1 For aqueous and soil samples, verification is required to ensure that neither a reducing condition nor a chemical interference is affecting color development. This is accomplished by analyzing a second 10mL aliquot of the pH adjusted filtrate of the soluble spike. 1mL of 100ppm $K_2Cr_2O_7$ solution (Section 8.15.1) is added to the filtrate and analyzed. To verify the absence of an interference, the spike recovery must be between 85% and 115%.

If the spike recovery is less than 85%, the presence of an interference or reducing condition is verified. Client Services must be notified and a narrative included on the final report that indicates “verification of this sample indicates the presence of a reducing condition or a chemical interference that could affect the results”.

10.3.5.2 If addition of the spike extends the concentration beyond the calibration curve, the analysis solution must be diluted with a blank solution and the calculated results adjusted accordingly.

10.3.5.3 If the result of verification indicates a suppressive interference, the sample must be diluted and reanalyzed.

10.3.5.4 If the interference persists after sample dilution then an alternative method must be considered. See the Inorganics Supervisor for further instruction.

Aqueous samples with acidic extracts that yield recoveries of less than 85% are re-tested to determine if the low spike recovery is due to the presence of residual reducing agent. This determination is performed by first making an aliquot of the extract alkaline (pH 8.0-8.5) using 1N NaOH. The pH is recorded in the laboratory notebook. This aliquot is respiked and analyzed. If a spike recovery of 85-115% is obtained on the alkaline aliquot of an acidic extract that initially was found to contain less than 5 mg/L Cr(VI), one can conclude that the analytical method has been verified.

10.3.5.5 If the spike recovery is still below 85%, then the presence of reducing agent is confirmed. Client Services must be notified and a narrative included on the final report that indicates “verification of this sample indicates the presence of a reducing condition or a chemical interference that could affect the results”.

10.4 Continuing Calibration

The calibration curve must be verified after every 10 samples or less, with a recovery of 85 – 115% of a non-digested, colored ICV/CCV standard (Section 8.16). If these criteria are not met, then the CCV is re-analyzed. If failure repeats, then the CCV is re-made and re-analyzed. If failure continues, prepare a new calibration curve (Section 10.1). See Section 12 for corrective actions.

10.5 Preventive Maintenance

The Spectrophotometer is calibrated on a semi-annual basis by an instrument service company. Records are kept on file.

11. Data Evaluation, Calculations and Reporting

For aqueous samples:

$$\text{mg } Cr^{+6}/L = \left[\frac{\text{absorbance} - \text{y-intercept}}{\text{slope}}\right] \times \text{dilution}$$

For solid samples:

$$\text{mg } Cr^{+6}/Kg = \left[\frac{\text{absorbance} - \text{y-intercept}}{\text{slope}}\right] \times \left[\frac{\text{dilution x 0.1L (final volume)}}{\text{weight (Kg) of sample}}\right]$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the ICV or LCS recovery falls outside the designated acceptance range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

Failure of the LCS for method 3060A requires that the entire sample batch be re-digested and re-analyzed.

If the method blank for method 3060A is greater than the reporting limit, then the following action must be taken. Samples that are non-detect may be reported, and the method blank result is narrated on the final report. If the sample concentration is greater than 10X the concentration of the method blank, then the sample may be reported, and the method blank is narrated on the final report. If the sample(s) concentration are above the reporting limit, but are not greater than 10X the concentration of the method blank, then those samples must be re-digested and re-analyzed. In this case, it is not necessary to submit a narrative unless there is a problem with the hold time, or there is not enough sample to re-digest. The Client Services Department must be made aware of the situation, and if the data is released then the method blank non-conformance is narrated.

The ICV for the analysis must have a recovery of 85-115%. If the ICV fails, it must be re-made and re-analyzed. If the failure persists, then a new calibration curve is constructed.

The ICB for the analysis must be below the detection limit for the analysis. If the ICB fails this requirement, then fresh blank must be colored and analyzed, if this does not solve the problem then a new calibration curve must be constructed.

If the CCV or the CCB fail, pour out a fresh aliquot of CCV and CCB and re-color and re-analyze. If the CCV or the CCB does not pass, then construct a new calibration curve and re-analyze the samples that followed the last acceptable CCV/CCB.

A duplicate result that is outside of the controls limits for this method must be narrated. The sample must be examined to determine the cause of the poor duplication. If it is determined that the poor duplication was due to analyst technique, then the sample and its duplicate are re-digested and re-analyzed. Otherwise, narrate the non-conformance on the final report.

If the matrix spike (aqueous samples) falls outside of the acceptable range of the method, then the analyst must go to the verification (section 10.3.5) part of this method. Poor spike recoveries must be narrated on the final report. Upon Client authorization, hexachromes with poor spike recoveries can be analyzed for total chromium by ICP-AES (Method 6010). The sample must be processed according to the Total Chromium procedure employed and the digestion procedure necessary. Clients will be invoiced for the Total Chromium analysis. Solids and soils digested under 3060A that have failing matrix spike recoveries must be re-digested and re-analyzed to determine if the failure is do to poor technique or the sample matrix. If upon reanalysis, it is found that the recoveries are acceptable, then the data is submitted with the new spike results. If it is found that the recoveries are still unacceptable upon redigestion, then it is assumed that the sample matrix is suspect The pH and ORP results are evaluated against the Eh/pH Phase Diagram (Figure 1) for MCP or NJ HexCr, and the result (reducing or oxidizing) is included in a sample narrative explaining the poor spike recovery.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734,1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

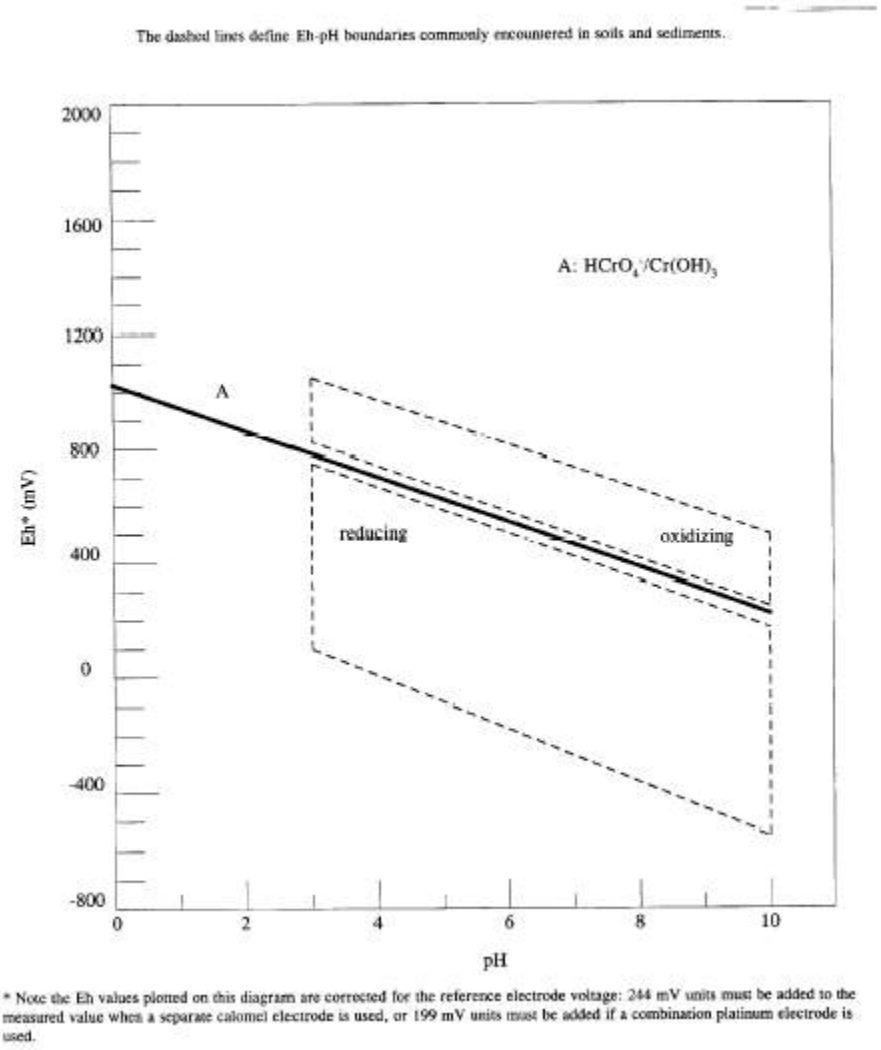
SOP/1734, 1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

Figure 1: Eh/pH Phase Diagram

FIGURE 1
Eh/pH PHASE DIAGRAM



Hydride Generation
Atomic Fluorescence Spectrometry

References: PSA 10.055 MILLENNIUM EXCALIBUR User Manual, Manual Part No. M055M050. Issue No. 1.3, January, 2003

PS Analytical CUSTOMER TECHNICAL INFORMATION FILE, Manual Part No. M023M059. Issue No. 7.0, July 2004

USEPA, "Method 7061A, 7062, 7741A and 7742" in Test Methods for Evaluating Solid Waste, Final Update 1992.

USEPA Office of Research and Development, "Standard Operating Procedure for the Operation of PS Analytical Hydride Generation Atomic Fluorescence Spectrometer" in NHX/SOP 300-001, Revision 0, June 1995

USEPA Office of Water Engineering and Analysis, "Method 1632 Chemical Speciation of Arsenic in Water and Tissue by Hydride Generation Quartz Furnace Atomoc Absorption Spectrometry", Revision A, August 1998.

1. Scope and Application

Matrices: Water and other aqueous samples (surface and saline waters, groundwaters, domestic and industrial wastes), soil/sediment samples,and tissues.

Definitions: Refer to Alpha Analytical Quality Manual.

Hydride generation Atomic Fluorescence Spectroscopy (HGAFS) determines trace metal concentrations in solution.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Mercury analyzer and in the interpretation of Mercury Analyzer data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

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Parameter	CAS
Arsenic, As	7440-38-2
Selenium, Se	7782-49-2

2. Summary of Method

Atomic fluorescence is an element specific, highly sensitive atomic spectrometric Technique, that when coupled to hydride generation, provides extremely high sensitivity for hydride forming elements.

The formation of hydrides is usually carried out by the addition of a reducing agent, in this case, sodium borohydride, to an acidified solution of the analyte. The newly formed hydride is then passed together with any excess hydrogen gas directly to a miniature flame where the hydride species dissociate to form free atoms. The free atoms that formed are excited by an intense excitation source. This source is a Boosted Discharge Hollow Cathode Lamp. The excited atoms are then focused onto the flame and the resultant fluorescence is measured using an interference filter and a photomultiplier tube detection system.

2.1 Method Modifications from Reference

Sediments and tissues may be digested and analyzed using HGAF..

3. Reporting Limits

See Table I in Section 16 for reporting limits.

4. Interferences

This technique is prone to interferences by transition metals, but this should not be significant on natural samples. Recovery tests should be preformed.

There are two oxidation states for Inorganic Arsenic; As (V) and As (III). All arsenic species have to be converted to the As (III) state before generating hydrides.

Physical interferences are effects associated with the sample which may change rate in which the gaseous hydride is purged from solution. If physical interferences are present, they may be reduced by diluting the sample.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

Care must be taken when handling all liquid samples, digestates, and standards since they are preserved to a pH <2.

Never look directly into the boosted discharge hollow cathode lamp, due to the intense UV radiation produced.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous total samples: The volume of sample required is dependent on the analyses requested. Typically, a 500 mL of sample is collected in plastic or glass containers.

Aqueous dissolved samples: Samples may be field filtered and preserved.

Solid samples: A minimum of 10.0 grams of sample must be collected in a glass jar.

6.2 Sample Preservation

Aqueous total samples: Samples are preserved with 2 mL of 1:1 nitric acid per liter of sample (pH $\leq$ 2) at time of collection. Samples that cannot be acid preserved at the time of collection because of sampling limitations or transport restrictions should be acidified with nitric acid to a pH $\leq$ 2 upon receipt at the laboratory. Following acidification, the sample must be held for a minimum 16 hours before pouring an aliquot for sample digestion.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Aqueous dissolved samples: If the laboratory is required to filter the samples, the sample must be filtered through a 0.45-µm membrane filter. Glass or plastic filtering apparatus must be acid rinsed to avoid possible contamination. A plastic apparatus must be used when determination of boron or silica is critical. Use a portion of the filtered sample to rinse the filter flask, discard this portion and collect the required volume of filtrate. Immediately following filtration acidify the filtrate with nitric acid to a pH $\leq$ 2.

All samples: The samples can be stored at room temperature or can be refrigerated and maintained at 4°$\pm$2°C until digestion and analysis. All samples must be analyzed within 6 months from date of collection.

The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. Eating, drinking, smoking, or the application of cosmetics is not permitted in the laboratory area. Horseplay of any kind is prohibited. Pipetting by mouth is not permitted. All Personal Protective Equipment (PPE) must be removed before leaving the laboratory area and befor entering the employee lounge or eating area. Always wash your hands before leaving the laboratory. All relevant Material Safety Data Sheets (MSDSs) are kept alphabetically in the centrally located file storage, in the common area outside of the Information Technology (IT) offices.

Approved PPE, which includes Safety Glasses, Gloves and Lab Coats, must be worn at ***all*** times when handling samples, reagents, chemicals, or when in the vicinity of others handling these items, so that dermal contact is avoided. All standards, reagents, and solvents shall be handled under a hood using the proper PPE. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, use a secure transporting devise and/or secondary outer container. Chemical storage is properly segregated and adequately ventilated to reduce the possibility of

hazardous reactions. Chemical storage in work areas shall be kept to a minimum. Storage on bench tops or other work surfaces, except temporary, is not permitted.

Spilled samples, solvents, reagent, and water must be cleaned up from bench tops, instruments and autosampler surfaces immediately. A spill is considered a quantity of hazardous material if it is two times greater than the normal working volume. Concentrated solvents, acids or bases present a moderate to extreme hazard to the skin and mucous membranes. If contact with the skin occurs, immediately flush with large volumes of water. In the case of acidic/basic spills, the *Spill Kit* located in each laboratory shall be utilized before attempting to cleanup the spill. Although procedures are designed to minimize the possibility of an accident, all injuries or accidents, regardless of the nature or severity, are to be reported to the Department Manager Supervisor immediately. If an employee discovers a potentially unsafe condition, this must be reported to the Department Manager Supervisor immediately. No employee should feel compelled to work in a situation where they do not feel entirely informed, trained, or safe.

Analytical instrumentation poses the unique possibility of exposure to high voltages. Other than the *routine* instrument maintenance, as listed in the front of every Instrument Maintenance Logbook, at no time shall an instrument operator attempt to maintenance an instrument alone, or without the proper training, supervision or instruction. Caution must always be used in the presence of moving parts (autosamplers) and hot surfaces (injection ports).

Compressed gas cylinders shall only be moved with the dolly supplied for this specific purpose. The cap must be on the cylinder while it is being moved. The tank must be secured when in its final position. All spent tanks are to be returned in the same manner, and secured until removed by the vendor. Liquid argon or nitrogen represents a potential cryogenic hazard and safe-handling procedures must be used at all times.

7. Equipment and Supplies

7.1 PSA 10.055 MILLENNIUM EXCALIBUR: The PSA Millennium Excalibur Atomic Fluorescence Analyzer equipped with a PC loaded with instrument software and PSA 20.400 autosampler. The instrument is connected to a source of high-purity, Mercury free argon regulated to 50 PSI.

7.2 Santoprene Peristaltic Pump tubing: Green/Green (PSA part number M025T002 or equivalent) for sample, blank and autosampler rinse. Grey/Grey (PSA part number M055T005 or equivalent) for sodium borohydride.

7.3 Compressed Argon

7.4 Air Displacement pipetters: Eppendorf brand or equivalent digital pipettes capable of delivering volumes ranging from 0.1 to 5000 µL with an assortment of high quality disposable pipette tips.

7.5 15-mL plastic AS-91 Autosampler tubes

7.6 Glassware for Digestion: Pre-cleaned 50mL disposable polyethylene screw cap digestion tubes.

7.7 Analytical balance: Capable of accurate measurement to the nearest 0.01 g.

8. Reagents and Standards

Deionized (DI) water is ASTM Type II laboratory reagent grade water or better (*i.e.,* Type I). The Barnstead NANO-pure system provides Type I water used in the preparation of samples and standards. ACS Trace Metal grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

8.1 Hydrochloric acid (HCL), concentrated. ACS reagent grade, Fisher number A508-212, or equivalent. Lots should be checked for purity prior to use and the results stored in a reagent check logbook.

8.2 10% (vol/vol) Hydrochloric acid. Prepare by adding 200 mL of concentrated hydrochloric acid to 2000 mL reagent water in a clean glass bottle.

8.3 20% (vol/vol) Hydrochloric acid. Prepare by adding 200mL of concentrated hydrochloric acid to 1L of reagent water in a plastic bottle.

8.4 5% (wt/vol) Potassium Iodide(KI)/Ascorbic Acid. Prepare by adding 5 g solid KI (Fisher ACS Grade) 5 g solid ascorbic acid (ACS grade) to 100 ml DI water.

8.5 0.7% Sodium Borohydride Sodium Hydroxide. Prepare by adding 2.0 g sodium hydroxide (ACS grade) to 500 ml DI, add 3.5 g sodium borohydride (reagent grade) and mix thoroughly.

8.6 Single element stock solutions: Stock solutions for the following elements are typically maintained in the laboratory or ordered as necessary. These are normally purchased as 1000 mg/L standards from Fisher Scientific or equivalent.

8.6.1 Arsenic

8.6.2 Selenium

8.7 Intermediate Working Standard Solutions

Intermediate Working Standard solutions are prepared from certified vendor Stock Standard solutions and include those analytes listed above. Vendor sources are subject to change and equivalent solutions may be used. Standards can be prepared as multi-element or single element standards as needed.

Stock standards used to prepare the calibration standards and calibration verification standard are typically diluted 1:1000 to prepare an intermediate working standard. These preparations are recorded in the laboratory standards preparation log and noted as below.

8.7.1 Intermediate Calibration Standard C-(Date)-H(element): The intermediate standard used to prepare the calibration standards is identified as C (calibration) – Date (date prepared) – H (hydride) element (As, Se etc.). These standards are typically diluted in the same matrix as the stock standard, i.e. 2% Nitric acid and are prepared approximately every two weeks or as needed. Add 0.05 ml of stock

1000 mg/L standard to 50 ml vial.

8.7.2 Calibration Standards C-(Date)-H(element)#: Calibration standards are prepared from the Intermediate Standard and identified as C (calibration) – Date (date prepared) – H (hydride) element (As, Se etc.) # (The first set of standards prepared from the Intermediate standard would be #1). Calibration standards are prepared with each batch of samples using the same reagents.

8.7.3 Intermediate Calibration Verification Standard I-(Date)-H(element): The intermediate standard used to prepare the calibration standards is identified as I (initial calibration verification) – Date (date prepared) – H (hydride) element (As, Se etc.). These standards are typically diluted in the same matrix as the stock standard, i.e. 2% Nitric acid and are prepared approximately every two weeks or as needed. Add 0.05 ml of stock 1000 mg/L standard to 50 ml vial.

8.7.4 Calibration Verification Standards I-(Date)-H(element)#: Calibration standards are prepared from the Intermediate Standard and identified as I (initial calibration verification) – Date (date prepared) – H (hydride) element (As, Se etc.) # (The first set of standards prepared from the Intermediate standard would be #1). The calibration verification standard is prepared with each batch samples using the same reagents.

8.8 Daily Calibration Standards

An Initial Calibration Curve is prepared for each level and is described below. Typically a four point curve is prepared including a high level standard at or near the linear range of the analysis (STD4), a low level standard that confirms the reporting limit (STD1), and a calibration blank (STD0), is used. See Table II for a list of the initial calibration concentrations. Calibration standards are prepared to matrix match standards to samples.

Standards must be pre-reduced prior to hydride generation analysis to put the analyte in the oxidation state which most readily forms a volatile hydride. These oxidation states are As^{3+} and Se^{4+}. Arsenic requires the KI/Ascorbic acid solution and selenium requires concentrated acid and heating.

For arsenic standards preparation, add 15 ml concentrated hydrochloric acid and 2.5 ml of the KI/Ascorbic acid solution to 50 ml tubes and add standards as stated below. Arsenic may be analyzed after a minimum of four hours standing at room temperature.

For selenium standards preparation, add 5 ml of concentrated acid to 50 ml tubes and add standards as stated below. Heat standards to 90°C for 30 minutes.

8.8.1 STD0 (Calibration Blank): Calibration Blank: Bring reagents to 50 ml with DI water.

8.8.2 STD1 (Reporting Limit Standard): Add 25 µL of intermediate standard to 50 ml digestion tube containing reagents and bring to volume with DI water. The final concentration of each element is 0.5 µg/L.

Note: For a reporting limit standard of 0.1 µg/L, dilute intermediate 1000 µg/L standard 1:10, add 50 µL and brig to final volume of 50 ml.

8.8.3 STD2: Add 100 µL of intermediate standard to 50 ml digestion tube containing reagents and bring to volume with DI water. The final concentration of each element is 2.0 µg/L.

8.8.4 STD3: Add 250 µL of intermediate standard to 50 ml digestion tube containing

reagents and bring to volume with DI water. The final concentration of each element is 5.0 μg/L.

Note: This standard may be used as the high standard in the calibration curve for 0.1 μg/L reporting limit analyses.

8.8.5 STD4: Add 500 µL of intermediate standard to 50 ml digestion tube containing reagents and bring to volume with DI. The final concentration of each element is 10.0 μg/L.

8.9 The Initial calibration verification (ICV) and continuing calibration verification (CCV). The ICV and CCV standards are prepared from sources separate from the initial calibration curve and are used to verify the initial calibration curve throughout the analytical sequence. See Table II for a list of the ICV/CCV concentrations. To prepare this solution add 250 µL of the separate source intermediate standard to 50 ml digestion tube containing reagents and bring to volume with DI water. The final concentration of each element is 5.0 μg/L.

Note: If 5.0 μg/L standard is used for the highest calibration standard, use 0.1 ml of the separate source intermediate standard and bring to a final volume of 50 ml to prepare a 2.0 μg/L ICV.

The Calibration blank (STD0), initial and continuing calibration blanks (ICB and CCB) are the analysis of the matrix diluent and are analyzed prior to the initial calibration curve and after the ICV and CCVs, respectively.

8.10 Calibration blank (STD0), initial and continuing calibration blanks (ICB and CCB): The blanks are the analysis of the matrix diluent and are analyzed prior to the initial calibration curve and after the ICV and CCVs, respectively.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

Quality control (QC) samples are necessary to monitor both the sample digestion and analysis procedures.

9.1 Blank(s)

9.1.1 Method Blank

A method blank must be analyzed once per every 20 samples or per metals digestion batch, whichever is more frequent.

Metal concentrations must not be detectable in the method blank at values greater than the reporting limit.

Corrective Action: May repeat analysis once. Digestion of the method blank and all associated samples must be performed until the method blank for the out of control metal is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Department Manager if the samples associated with an out of control method blank are below the reporting limit for

the affected metal or if the concentrations of the affected metal are greater than 10x the blank level in the samples. In such cases, the sample results are accepted without corrective action for the high method blank result. The client must be notified in the project narrative associated with the sample results.

9.1.2 Initial Calibration Blank (ICB)

An ICB must be analyzed immediately following the ICV.

The ICB concentration must not be greater than the reporting limit.

Corrective Action: May repeat analysis once to see if an analytical error occurred. If the ICB still exceeds the control limits, re-calibrate the instrument and re-analyze a fresh blank.

9.1.3 Continuing Calibration Blank (CCB)

A CCB must be analyzed immediately after every CCV.

The CCB concentration must not be greater than the reporting limit.

Corrective Action: May repeat analysis once to see if an analytical error occurred. If the CCB still exceeds the control limits, re-calibrate and/or re-analyze a fresh blank. All samples associated with the out of control metals in the CCB must be re-analyzed (since the last acceptable CCB). Exceptions may be made with approval of the Department Manager if the samples associated with the out of control method blank are non-detect for the affected metals or if sample concentrations for the affected metals are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high CCB and the client is notified in a project narrative associated with the sample results.

9.2 Laboratory Control Sample (LCS)

The LCS is from a second source to verify the accuracy of the digestion and analytical procedures. The LCS is digested along with the samples. An LCS must be digested and analyzed once per every 20 samples or per metals digestion batch, whichever is more frequent.

The acceptable recovery QC limits are 80%-120% for an aqueous LCS.

Corrective Action: May repeat analysis once to see if an analytical error has occurred. If the LCS recovery is still out of control, re-digest and re-analyze the LCS and all associated samples. Samples cannot be analyzed until an acceptable LCS is obtained. Exceptions may be made with approval of the Department Manager if the samples associated with the out of control LCS are also associated with a matrix spike that is in control. This is an acceptable measure of accuracy of the digestion and analytical procedures. An explanation of this out of control LCS recovery must be included in the project narrative to the client and the sample data reported with the acceptable MS results as batch QC.

9.3 Initial Calibration Verification (ICV)

The initial calibration verification check standard is from a second source to verify the accuracy of the standard curve. The concentration of the ICV is at approximately the mid-level of the calibration curve.

The acceptable recovery QC limits for the ICV is 85-115%.

Corrective Action: May repeat analysis once to see if an analytical error occurred. If the ICV still exceeds the control limits, re-calibrate the instrument.

9.4 Continuing Calibration Verification (CCV)

A CCV must be analyzed at a minimum of every 10 samples and at the close of an analytical sequence. The concentration of the CCV is at approximately the mid-level of the calibration curve. This standard monitors instrument performance throughout the duration of the analytical sequence.

The acceptable recovery QC limits for the CCV is 85-115%.

Corrective Action: May repeat analysis once to see if an analytical error occurred. If the CCV still exceeds the control limits, re-calibrate and re-analyze all samples since the last acceptable CCV.

9.5 Matrix Spike

A matrix spike must be performed once per 20 samples and is from a second source to verify the accuracy of the digestion and analytical procedures. When project specifications dictate, a Matrix Spike Duplicate (MSD) may also need to be performed at the same frequency as the MS.

The acceptable recovery QC limits for a MS/MSD is 75%-125% for both the solid and aqueous MS/MSD pair. Calculate the %RPD as in Section 9.6 when analyzing a MS/MSD pair. The acceptable %RPD is ± 20%.

Corrective Action: May repeat analysis once to see if an analytical error has occurred. If the % recovery or %RPD still exceeds the control limits and the LCS is compliant; include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the affected metals results as evidenced by matrix spike recovery or %RPD outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses (matrix duplicate) must be performed once per 20 samples (5% frequency). Acceptable relative percent difference (RPD) for duplicate analysis is ± 20 % for both aqueous and solid matrices. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

Corrective Action: May repeat analysis once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the affected metals results as evidenced by the matrix duplicate RPD exceedance.

9.7 Method-specific Quality Control Samples

9.7.1 CRI Standard Check Sample (Reporting Limit Standard)

This solution is used to verify instrument sensitivity at the reporting limit. This standard is routinely analyzed as the lowest standard in the initial calibration curve, or STD1, see Section 8.8.2. When included in the initial calibration curve, the curve must meet the criteria for linearity (r = 0.995). When project specifications dictate, the CRI can be analyzed separately from the initial calibration. The concentration of the CRI when analyzed as a separate check from the initial calibration curve, is equivalent to the

reporting limit for each element in STD1, see Table II. See Sections 10.3.1 for preparation details. This check standard, when analyzed, follows the ICV and ICB.

The CRI Check sample, when analyzed per client specifications, must be analyzed at the beginning of an analytical run . This solution verifies the accuracy at the low end of the calibration curve.

Results for the analyte in the CRI solution must be within 50-150% recovery criteria.

Corrective Action: May repeat analysis once to see if an analytical error occurred. If the CRI solutions still exceed the control limits, re-calibrate and/or re-analyze a fresh CRI solution. All samples associated with the out of control metals in the CRI must be re-analyzed (since the last acceptable CRI check sample).

9.8 Method Sequence

The following outlines the general analytical sequence:

Initial calibration curve (Total of 4 standards and a calibration blank)
ICV
ICB
CRI – If required
Method Blank
LCS
Sample analysis (samples 1-6)
CCV
CCB
Sample analysis (7-16)
CCV
CCB
Sample analysis (17-26)
CCV
CCB

10. Procedure

10.1 Equipment Set-up

Samples are prioritized by the Metals Department Manager or Preparation Group Leader for digestion based on hold time and client due date. The analyst must be familiar with instrument software and hardware as described in the user manuals supplied with the instrument, and have a completed IDC on file (Section 13.2) prior to any sample analysis.

The reductant, sample and reagent blank are introduced into the system by the use of a peristaltic pump, which in turn makes up the continuous flow vapor generator. When turning on the instrument, check the tubing for ware, check the pump and the three lines (reductant, sample and blank) for flow.

The Millenium Excalibur should be turned on and allowed to warm up for no less than 20 minutes. Be sure the correct cathode lamp is being used. The peristaltic pumps should be put on idle mode for warm up. Make the appropriate fresh solutions (Sodium Borohydride, 10% HCl, and 20% HCl as needed). In the software, check that the carrier and dryer gases are registering.

At this time, load the autosampler tray with the calibration standards and samples, and create the run sequence. Also check the waste drum, and be sure there is room left for the run's waste. Take the pumps off of idle mode, and light the flame and let the lamp warm up for about 5 minutes.

10.2 Equipment Operation and Sample Processing

Power is turned on to the instrument, autosampler, PC and argon gas supply. Peristaltic pump windings are inspected for wear and replaced if necessary. The winding clips are relaxed in between usage. The instrument manufacturer suggests that the pump tubing be changed once per week.

The sodium borohydride solution is introduced via the grey/grey pump tubing, 10% HCl is attached to the green/green autosampler rinse and the rinse blank line is also a green/green tubing immersed in DI. The autosampler probe line is attached to a green/green pump tubing. The pumps are turned on in the software and flow checked to insure that solutions are delivered to the mixing valve.

After initializing the Millennium software, the instrument is turned on via the software and allowed to warm up for 30 minutes. The analysis method is selected as well as the autosampler template. The hydride methods are set up in the software which specifies delay, read and rinse times as well as pump speeds. Autosampler templates are set up for each method specifying calibration standard, QC standards and positions on the autosamlper rack. Once sample ID's have been typed in, the file is saved as the login number.

A data file is opened and saved as "WGnumber".

Put pumps manually on regular speed, light the lamp and allow to warm up for 5 minutes.

Initiate sample analysis by clicking on the start button on the analysis page. If a sample concentration is greater than the highest standard concentration of the calibration curve, dilute the digestate with the same matrix as was used for the calibration blank, and reanalyze.

Following analysis, the data file is exported to the LIMS on the O:Metals\Millennium drive for data processing. The instrument and autosampler are then rinsed with DI water.

10.3 Initial Calibration

A series of calibration standards are prepared for the initial calibration curve as described in Section 8.8. The preparation date of these standards, the initials of the analyst, the lot number of the source material, stock concentrations, volumes used, final volumes, final concentrations and manufacturer must be recorded in the metals standard preparation logbook.

Calibrate the instrument using the calibration standards listed in Section 8.16. The system must be flushed with a rinse solution and carrier solution (10% HCl) between each standard to protect against potential carry-over. The calibration curve consists of a blank and four standards. Refer to Section 9.8 which outlines the general analytical sequence.

A linear least squares regression calibration curve is selected for the hydride methods.

A four point calibration curve is performed if the CRI standard is not analyzed.

The correlation coefficient must be $\geq$0.995.

Corrective Action: Re-calibrate until criteria are met

10.3.1 Aqueous Sample Preparation

Digests of Sediments (3050B) and Tissues(3051) can be analyzed by this method. Total aqueous, dissolved samples, digests of aqueous and solid samples must be pre-reduced prior to hydride generation analysis to put the analyte in the oxidation state which most readily forms a volatile hydride. These oxidation states are As^{3+} and Se^{4+}. Arsenic requires the KI/Ascorbic acid solution and selenium requires concentrated acid and heating.

For arsenic sample preparation, add 15 ml concentrated hydrochloric acid and 2.5 ml of the KI/Ascorbic acid solution to 50 ml vials, then add 25 ml of sample. This solution must set for a minimum of four hours before bringing to a final volume of 50 ml with DI water for arsenic analysis. If sample dilution is required, cook extra diluent with samples.

Note: If aliquots from acid digestion of soils/sediments,tissues or waters are being analyzed, it is advisable to check the pH of the sample following addition of the sodium borohydride solution to insure that the pH is between 5 and 7.

For selenium sample preparation, add 5 ml of concentrated acid to 50 ml vials and add 45 ml of sample. Heat samples to 90°C for 30 minutes. If sample dilution is required, cook extra diluent with samples.

10.3.1.1 **Preparation Blank:** Add same reagents as sample preparation and add DI water instead of sample. Prepare as sample.

10.3.1.2 **LCS:** Add same reagents as sample preparation and add DI water instead of sample. Add 0.25mL of intermediate standard. Prepare as sample.

10.3.1.3 **Matrix Spike:** Add same reagents as sample preparation and add aliquot of sample. Add 0.25mL of intermediate standard. Prepare as sample.

10.3.2 Sample Analysis

10.3.2.1 Flush the system with the rinse blank. Analyze the CCV and the CCB after each 10 samples and at the end of the analytical sequence. The rinse solution is normally 10% HCl (8.2).

10.3.2.2 The CRI (if required) must be analyzed at the beginning of an analytical run.

The CRI analysis is not performed if a calibration standard is prepared at the reporting limit and included in the initial calibration curve and if not specifically requested by the client. The calibration curve must always meet the criteria for linearity (r = 0.995).

10.3.2.3 Target analytes detected above the calibration range must be diluted and re-analyzed. See Section 12.0 for all Quality Control frequencies and criteria

10.4 Continuing Calibration

Periodically and at he end of the analysis, CCV standards (see 9.4) and CCB standards (see 9.1.3) are analyzed.

10.5 Preventative Maintenance

Inspect pump windings for signs of wear and replace (recommended at least monthly). Clean gas/liquid separator periodically when appears coated with yellow film (rinse with 30% KOH solution and DI). Change dryer tube annually.

11. Data Evaluation, Calculations and Reporting

11.1 Primary and Secondary Review

This analysis review form is used by the analyst and secondary reviewer to note passing/failing QC standards and to record any other comments as communication for reporting sample results.
All metal results are reportable without qualification if analytical holding times are met, preservation (including pH and cooler temperatures) are met, and all QC criteria defined in the table below are met. If any of the below QC parameters are not met, all associated samples must be evaluated for re-analysis. See Section 9 for additional QC discussion including corrective actions for any QC outliers.

QC Parameter	Acceptance Criteria
Initial Calibration Curve	R $\geq$ 0.995
Method Blank	< reporting limit
Laboratory Control Sample	80-120%R for solid and aqueous
Matrix Duplicate	20%RPD for results <5x reporting limit
Matrix Spike	75-125%R for solid and aqueous
Matrix Spike Duplicate (if requested)	75-125%R for solid and aqueous; and 20% RPD
Initial and Continuing Calibration Verification	85-115%R
Initial and Continuing Calibration Blank	< reporting limit
CRI (Reporting Limit Check)	50-150% R (DoD = 80-120%R)

11.2 Calculations

The metal results are calculated as follows:

As:

$$\text{Metal result in } \mu g/L = \frac{C \times B}{A} \times DF$$

where:

A = Initial sample volume in mL, typically 25 mL
B = Digestate final volume in mL, typically 50 mL
C = Concentration of sample from instrument read-out in µg/L
DF = Dilution Factor

Se:

$$\text{Metal result in } \mu g/L = \frac{C \times B}{A} \times DF$$

where:

A = Initial sample volume in mL, typically 45 mL
B = Digestate final volume in mL, typically 50 mL
C = Concentration of sample from instrument read-out in µg/L
DF = Dilution Factor

All metal results must be reported to three significant figures.
For solid samples: C x B x DF divided by grams in dry weight.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

If non-compliant metals results are to be reported, the Department Manager and/or the Laboratory Director must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste and Sample Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/08-05 MDL/LOD/LOQ Generation

SOP/08-12 IDC/DOC Generation

SOP/G-006 Hazardous Waste and Sample Disposal

16. Attachments

Table I: Reporting Limits

Table II: Initial Calibration Levels and ICV/CCV Concentrations

Table III: Hydride Generation AFS: Selenium

Table IV: Hydride Generation AFC: Arsenic

Table I – Reporting Limits

Metal	Aqueous (µg/L)	Solid (mg/Kg)	Tissue (mg/Kg)
Arsenic	1.0	0.05	0.05
Selenium	0.55	0.0275	0.0275

Table II: Initial Calibration Levels and ICV/CCV Concentrations

Analyte	STD1	STD2	STD3	STD4	ICV,CCV
	(µg/L)	(µg/L)	(µg/L)	(µg/L)	(µg/L)
As	0.5	2	5	10	5
Se	0.5	2	5	10	5

Table III

Hydride Generation AFS

Selenium

Stock Standard Preparation

Calibration Stock Standard – Dilute 0.05 ml of 1000 mg/L standard to 50 ml in 2% HNO_3 to make 1000 ug/L standard.

ICV Stock Standard – Dilute 0.05 ml of 1000 mg/L to 50 ml in 2% HNO_3 to make 1000 ug/L standard.

Standards Preparation in 50 ml Digestion Tubes

STD0		add 5 ml Conc. HCl bring to 50 ml final volume with DI
STD1	(0.5 ug/L)	add 5 ml Conc. HCL, **0.025 ml stock standard**, DI to 50 ml
STD2	(2 ug/L)	add 5 ml Conc. HCL, **0.10 ml stock standard**, DI to 50 ml
STD3	(5 ug/L)	add 5 ml Conc. HCL, **0.25 ml stock standard**, DI to 50 ml
STD4	(10 ug/L)	add 5 ml Conc. HCL, **0.50 ml stock standard**, DI to 50 ml
ICV (5 ug/L)		add 5 ml Conc. HCL, **0.25 ml ICV stock**, DI to 50 ml

Sample Preparation in 50 ml Digestion Tubes

Add 45mL of sample to Digestion Tubes

Add 5mL of Concentrated HCL to the Digestion Tubes.

Add 0.25 ml of 1000 ug/L stock to LCS and matrix spike sample

Heat standards and samples to 95°C for 0.5 hr in water bath

Table IV

Hydride Generation AFS
Arsenic

Stock Standard Preparation

Calibration Stock Standard – Dilute 0.05 ml of 1000 mg/L standard to 50 ml in 2% HNO_3 to make 1000 ug/L standard.

ICV Stock Standard – Dilute 0.05 ml of 1000 mg/L to 50 ml in 2% HNO_3 to make 1000 ug/L standard.

STD0 Add 15 ml Conc. HCl, 2.5 ml 5% KI/5% Ascorbic Acid, bring to 50 ml final volume with DI, wait 4 hours minimum.

STD1 (0.5 ug/L) Add 15 ml Conc. HCl, 2.5 ml 5% KI/5% Ascorbic Acid, **0.025 ml stock standard**, bring to 50 ml with DI, wait 4 hours minimum

STD2 (2 ug/L) Add 15 ml Conc. HCl, 2.5 ml 5% KI/5% Ascorbic Acid, **0.10 ml stock standard**, bring to 50 ml with DI, wait 4 hours minimum

STD3 (5 ug/L) Add 15 ml Conc. HCl, 2.5 ml 5% KI/5% Ascorbic Acid, **0.25 ml stock standard**, bring to 50 ml with DI, wait 4 hours minimum

STD4 (10 ug/L) Add 15 ml Conc. HCl, 2.5 ml 5% KI/5% Ascorbic Acid, **0.50 ml stock standard**, bring to 50 ml with DI, wait 4 hours minimum

ICV (5 ug/L) Add 15 ml Conc. HCl, 2.5 ml 5% KI/5% Ascorbic Acid, **0.25 ml ICV stock**, bring to 50 ml with DI, wait 4 hours minimum

Sample Preparation in 10 ml autosampler tubes

Add 15 ml Conc. HCL to Digestion tubes and 2.5 ml 5% KI/5% Ascorbic Acid

Add 0.25 ml of 1000 ug/L stock to LCS and matrix spike sample

Add 25 ml of sample and mix, wait 4 hours minimum, bring up to a 50 mL final volume with DI water.

Ignitability of Solids

Reference Method: **Method 1030**, Rev. 0, December 1996 (Modified)

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1997.

1. Scope and Application

Matrices: Solids including pastes, powders, granular material, soils, clay and solids that can be cut into strips.

Definitions: See Alpha Laboratories Quality Manual Appendix A

This method may be used to meet certain regulatory applications; with respect to the characteristic of ignitability in CFR 261.21, this method may be used, but is not required, to determine whether a solid waste “when ignited, burns so vigorously and persistently that is creates a hazard.” If it is impractical to perform the test because the physical form of the sample, generator knowledge should be used to determine the ignitability hazard posed by the material.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by completing the record of training.

2. Summary of Method

In a preliminary test, the test material is formed into an unbroken strip or powder train 250mm in length. An ignition source is applied to one end of the test material to determine whether combustion will propagate along 200mm of the strip within a specified time period. Materials that propagate burning along a 200mm strip within the specified time period are then subjected to a burning rate test. Materials that do not ignite or propagate combustion as described above do not require further testing.

In the burning rate test, the burning time is measured over a distance of 100mm and the rate of burning is determined. Lab doesn’t perform Burning rate test and will proceed to Flashpoint analysis by Flash test EPA 1010. SOP 2227

2.1 Method Modifications from Reference

A high temperature gas torch is utilized as the flame source. Lab is using modified method. Lab doesn’t proceed to the burning rate test if sample ignites during preliminary test.

3. Reporting Limits

There is no reporting limit for this analysis. Results are reported as either Negative or Positive. Refer to Section 11 for data reporting.

4. Interferences

Particle size of test material can affect not only the burning rate, but also the ignition of the material. Therefore particle size of the test material should be the same for each test run. Report the particle size of the test material in a simple descriptive format (e.g. fine powder, sand, coarse granular).

Temperature of some test material such as sulfur powder affects the burning rate. For reproducible results, all tests must be performed at approximately the same initial temperature (ambient room or laboratory temperature).

All tests must be carried out inside a fume hood with the test apparatus situated perpendicular (90°) to the direction of the airflow. Airflow parallel (0°) to the test apparatus results in non-reproducible burning rates.

The rate of airflow through the fume hood affects the burning rate. Too high an airflow will distort the flame and retard its horizontal propagation. The optimum airflow is within the range of 0.7 – 1 meter per second.

Materials that are moisture sensitive (i.e. readily absorb moisture from the air) should be tested as quickly as possible after removal from the sample container. All materials should be tested as received by the laboratory.

5. Health and Safety

Each sample should be treated as a potential health hazard. From this viewpoint, exposure must be reduced to the lowest possible level by whatever means available. References to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. These practices include, but are not limited to, the use of: laboratory coat, safety glasses, and latex or nitrile gloves, and/or heat resistant gloves. Use caution when operating the high temperature torch. Also use caution in handling the ceramic plate after heating the sample, as it remains hot.

Prior to starting the preliminary test, all sample materials must be tested to determine if that material is explosive or extremely flammable. Use a very small portion of material (1 gram or less). ***If the sample displays explosivity or extreme flammability, do not continue this test.***

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Sample containers should be completely filled and tightly sealed to preserve sample integrity.

6.2 Sample Preservation

No sample preservation is required.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

All samples are tested as received unless requested otherwise. Sample aliquots should be tested as soon as possible after removal from the sample container (i.e. samples must not be allowed to dry or absorb moisture for excessive periods or to lose volatiles). Allow samples to equilibrate to the ambient laboratory temperature in the sample container.

7. Equipment and Supplies

7.1 Ceramic Tile: Low heat conducting, non-combustible and impervious with marks indicating 80mm, 180mm, 200mm and 250mm along the test path. Any material capable to withstand high temperatures that have marks indicating 80mm, 180mm, 200mm and 250mm can be used.

7.2 Powder Train Mold: For molding powdered and granular materials for the burn rate test. The material of construction can be aluminum, brass, stainless steel or plastic. The mold is 250mm in length and has a triangular cross-section, with a width of 20mm, and a depth of 10mm as measured from the bottom of the triangular opening to where the sides meet. On both sides of the mold, in the longitudinal direction, two sheets are mounted as lateral limitations that extend 2mm beyond the upper edge of the triangular cross-section.

7.3 High temperature gas torch: A 6 to 7cm flame, with a minimum diameter of 5mm capable of attaining a temperature of at least 1000 °C. MAPP gas is its fuel source.

7.4 Stop watch.

7.5 Thermocouple: To measure the temperature of the gas flame

7.6 Thermometer: To measure initial temperature of material (i.e. room temperature).

7.7 Anemometer: To measure airflow in the fume hood.

7.8 Fume Hood.

8. Reagents and Standards

No special reagents are required to conduct this test.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Not applicable.

9.2 Laboratory Control Sample (LCS)

Not applicable.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

Not applicable

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Perform preliminary screening test:
  - Prepare test strip or powder train
  - Light flame and measure temperature
    - If it does not burn within specified time, sample is considered a negative, nonflammable solid. Test is complete.
    - If it does burn within specified time, sample is considered Positive for the preliminary screening test. Stop analysis.
- Contact supervisor or team leader; in order to switch method from Ignitability to Flash point.
- Refer to SOP 2227 for Flash point analysis

10.Procedure

10.1 Equipment Set-up

Safety Test: Prior to starting the preliminary test, all sample materials must be tested to determine if that material is explosive or extremely flammable. Use a very small portion of material (1 gram or less). ***If the sample displays explosivity or extreme flammability, do not continue this test.***

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Preliminary Screening Test

10.3.1.1 The preliminary ignitability test is conducted on all waste materials.

10.3.1.2 Place a platform (brick, stone, or other flame-resistant tile) in a fume hood about 20cm (or 8 inches) from the front of the hood in an area of laminar airflow. Position the sample perpendicular to the airflow. The airflow across the perpendicular axis of the sample should be sufficient to prevent fumes from escaping into the laboratory and should not be varied during the test. The air velocity should be approximately 0.7 meters/second. Measure and record the air velocity by using the anemometer (Section 7.7).

10.3.1.3 On the platform, prepare the test material in its “as received” form by forming an unbroken strip or powder train of sample 250mm long by 20mm wide by 10mm high on the platform. Position the sample perpendicular to the airflow. Use the mold (Section 7.3) to form the material as in Section 10.3.2.1 if appropriate.

10.3.1.4 Light the gas torch. Measure and record the temperature of the flame (tip of the flame) by a thermocouple (Section 7.6). The temperature of the flame must be at least 1000 °C.

10.3.1.5 Apply the tip of the flame to one end of the sample strip. Measure the propagation of combustion with the ceramic tile (Section 7.1). The test period will depend on the sample matrix as follows:

10.3.1.5.1 Non-Metallic Waste, Soils, Clays: Hold the flame tip on the sample strip until the sample ignites or for a maximum of 2 minutes. If combustion occurs, begin timing with a stopwatch and note whether the combustion propagates up to the 200mm mark within the 2-minute test period.

10.3.1.5.2 Metal or Alloy Powders: Hold the flame tip on the sample strip until the sample ignites or for a maximum of 5 minutes. If combustion occurs, begin timing with a stopwatch and note whether the combustion propagates up to the 200mm mark within the 20-minute test period.

10.3.1.6 If waste does not ignite and propagate combustion by either burning with open flame or by smoldering along the 200mm of sample strip within the 2 minute test period (or 20 minute test period for metal powders), the waste is not considered flammable and no further testing is required.

10.3.1.7 If the waste propagates burning of 200mm of the test strip within the 2-minute test period (20 minute test period for metals), test should be stopped and analysis should be switched to Flashpoint test (SOP 2227)

10.4 Continuing Calibration

Not applicable.

10.5 Preventive Maintenance

The ceramic plate and train mold must be cleaned after each sample.

11. Data Evaluation, Calculations and Reporting

Open ELN and include the following information for each sample:

Test Material Information:

- Date of test:
- Description of material: e.g. powder or paste, metallic or non-metallic
- Particle size: e.g. fine powder, granular, sand, etc.
- If the preliminary screening test (Section10.3.1) was negative, the result for the sample is Not Ignitable (reported as “NI”) and no further data is required.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the Sample Delivery Group Form and/or Nonconformance Report form. Project manager also have to be notified.

If sample cannot be analyzed using Ignitability method, project manager has to be notified and customer has to be contacted. If matrix of the sample is not appropriate for Ignitability analysis then LIMS product may be changed to Flashpoint product in order to provide customer with accurate data. Project manager/customer have to be notified and project manager has to approve this change.

Supervisor and project manager have to be notified if there is not a sufficient amount of sample provided for analysis.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

This study is not applicable to this method.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

These studies are not applicable to this method.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 MDL/LOD/LOQ Generation

1739 IDC/DOC Generation

1728 Waste Management and Disposal SOP

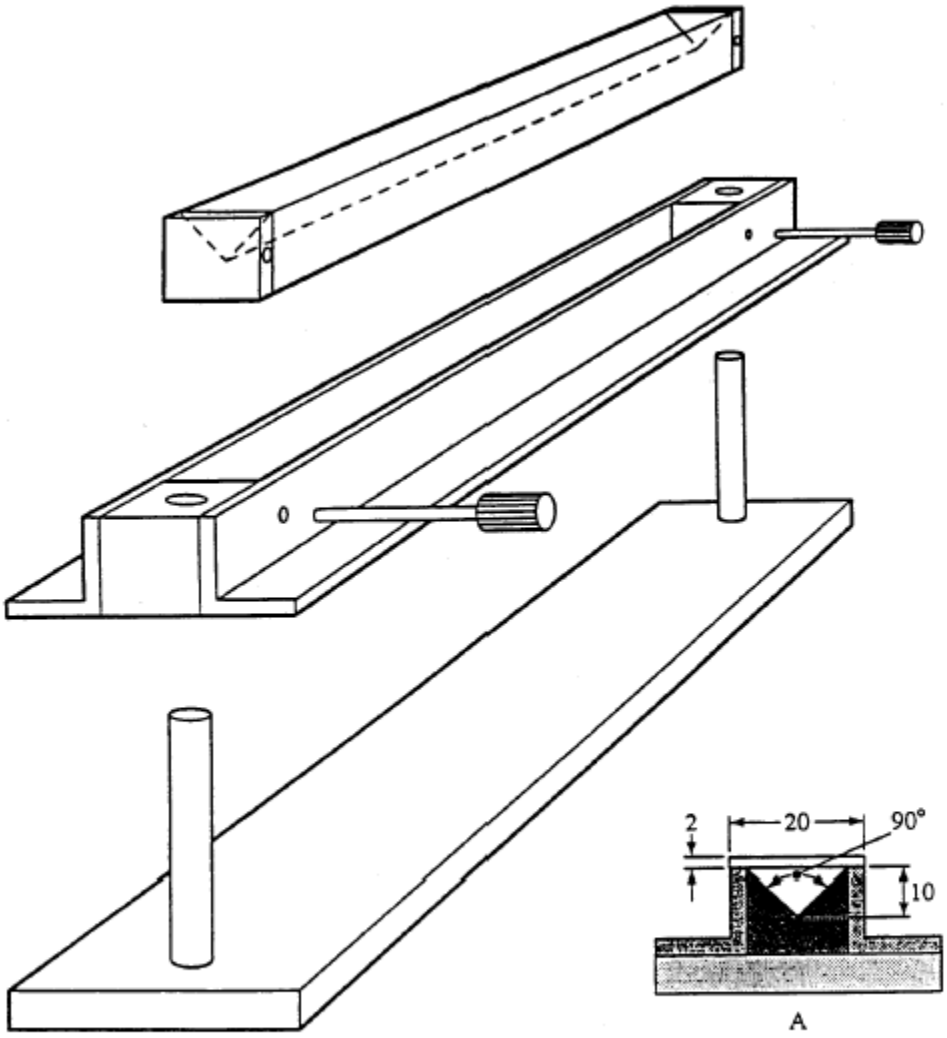
2227 Flashpoint

16. Attachments

- FIGURE 1: Powder Train Mold
- Flowchart

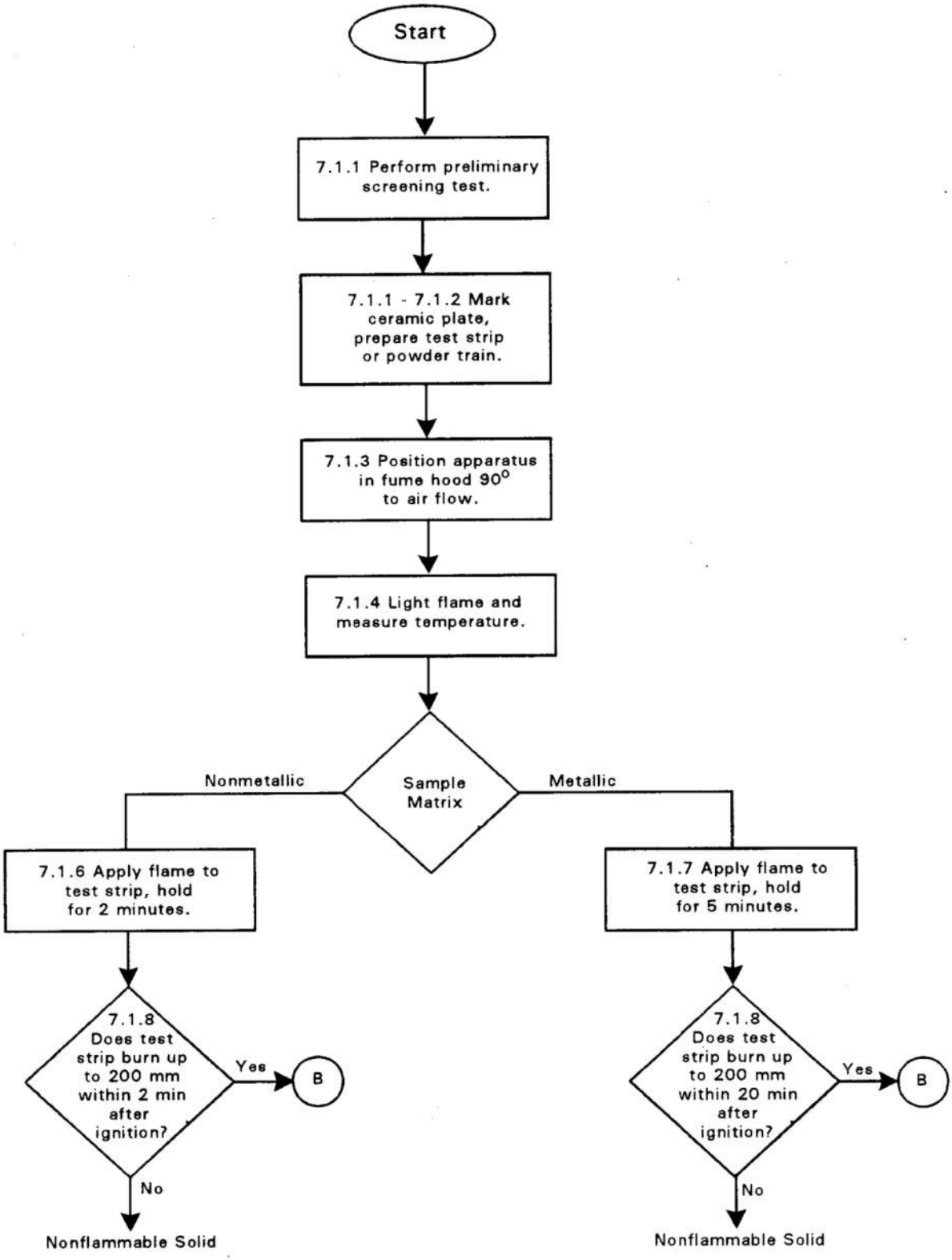
FIGURE 1

Powder Train Mold



(A) Cross-section of 250 mm long mould

Flowchart – Ignitability of Solids Method 1030



Inductively Coupled Plasma - Mass Spectrometry 6020B

Reference: **Method 6020B,** Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision V, July 2014.

1. Scope and Application

Matrices: Groundwaters, aqueous samples, industrial waste, soils, sludges, sediments, and other solid wastes.

Definitions: Refer to Alpha Analytical Quality Manual.

Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub µg/L concentrations of a large number of elements in water samples, waste extracts or digestates.

ICP-MS has been applied to the determination of over 60 elements in various matrices. Elements for which EPA has determined the acceptability of Method 6020 in a mulit-laboratory study on solid wastes are listed below and in Table 1.

If method 6020 is used to determine any analyte not listed in Table 1 below, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

Use of this method is restricted to spectroscopists who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS.

An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended Internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The Lithium internal standard must have an enriched abundance of 6Li, so that interference from Lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant amounts of the recommended internal standards. The internal standard used in this method is as follows: ^{6}Li, ^{45}Sc, ^{74}Ge, ^{103}Rh, ^{115}In, ^{159}Tb, ^{175}Lu, ^{209}Bi.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the ICP-MS and in the interpretation of ICP-MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Table 1

Parameter	CAS		Parameter	CAS
Aluminum	7429-90-5		Magnesium	7439-95-4
Antimony	7440-36-0		Manganese	7439-96-5
Arsenic	7440-38-2		Lithium	7439-93-2
Barium	7440-39-3		Molybdenum	7439-98-7
Beryllium	7440-41-7		Nickel	7440-02-0
Cadmium	7440-43-9		Potassium	7440-09-7
Calcium	7440-70-2		Selenium	7782-49-2
Chromium	7440-47-3		Silver	7440-22-4
Cobalt	7440-48-4		Sodium	7440-23-5
Copper	7440-50-8		Thallium	7440-28-0
Iron	7439-89-6		Vanadium	7440-62-2
Lead	7439-92-1		Zinc	7440-66-6

2. Summary of Method

When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial waste, soils, sludge's, sediments, and other solid wastes for which total (acid-leachable) elements are required.

Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into the mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratio and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate a problem. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

2.1 Method Modifications from Reference

This method is performed in a 1% Nitric Acid matrix for the calibration curve and standards and a 2% Nitric Acid matrix for the rinse.

3. Reporting Limits

Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, instrumentation and operating conditions. In relatively simple matrices, detection limits will generally be below 20 µg/L. Table 2 contains the list of Reporting Limits.

4. Interferences

4.1 Isobaric Elemental Interference

Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. This involves determining the signal for

another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Since commercial ICP-MS instruments nominally provide unit resolution at 10% of the peak height, very high ion currents at adjacent masses can also contribute to ion signals at the mass of interest. Although this type of interference is uncommon, it is not easily corrected, and samples exhibiting a significant problem of this type could require resolution improvement, matrix separation, or analysis using another verified and documented isotope, or use of another method.

4.2 Isobaric Molecular and Doubly Charged Ion Interference

Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature. Examples include $ArCl^+$ ions on the ^{75}As signal and MoO^+ ions on the Cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotope abundances form the literature, the most precise coefficients for an instrument can be determined form the ratio of the net isotope signals observed for a standing solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar^{35}Cl^+$ signal):

Corrected arsenic signal (using natural isotopes abundances for coefficient approximations) = (m/z 75 signal) – (3.13) (m/z 77 signal) + (2.73) (m/z) (82 signal), (where the final term adjust for any selenium contribution at 77 m/z)

Note: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g. $^{81}BrH^+$ from bromine waste)

Similarly, corrected cadmium signal (using natural isotopes abundances for coefficient approximations) = (m/z 114 signal) – (0.027) (m/z 118 signal) – (1.63) (m/z 108 signal), (where the last two terms adjust for any tin or MoO^+ contributions at m/z 114).

> **Note:** Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108, but use of the m/z 111 for Cd is even subject to direct ($^{94}ZrOH^+$) and indirect ($^{90}ZrO^+$) additive interferences when Zr is present
>
> **Note**: As for the arsenic equation above, the coefficients in the Cd equation are for only illustrative purposes. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting precision.

The accuracy of these types of equations is based upon the constancy of the OBSERVED isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the “parent” ion have not been found to be reliable, e.g. oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent –to-oxide ion intensities, the correction must be adjusted for the degree of outside formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferant. This type of correction has been reported for oxide-ion corrections using ThO^+/Th for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas has been shown to greatly reduce molecular interferences. These techniques can be used provided that method detection limits, accuracy, and precision requirements for analysis of the samples can be met.

4.3 Physical Interference

Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for the physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by the matrix change. When the intensity level of an internal standard is less than 30 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

4.4 Memory Interference

Memory interferences can occur when there are large concentration differences between samples or standards which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences which are observed. The rinse period between samples must be long enough to eliminate significant memory interferences.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Only Polyethylene or fluorocarbon (PFA or TFE) containers are recommended. Alpha uses polyethylene bottles. 0.5L is the recommended size.

6.2 Sample Preservation

Samples for total metals are preserved with (1:1) Nitric Acid to a pH<2.

Samples for soluble metals must be preserved with (1:1) Nitric Acid to a pH of <2 **after** filtration through a 0.45 um filter.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Samples that are to be analyzed for soluble metals, and have not been field filtered, must be filtered through a 0.45um filter as soon as possible. Samples are then preserved with 1:1 Nitric Acid to a pH<2, and then held for 18 hours. After the 18 hours the pH must be re-checked. If

after 18 hours the pH is still >2, then the sample must be re-acidified and held again for 18 hours. If the pH is till >2 after this 18 hour period, then the Inorganics Manager must be told.

Samples preserved with Nitric Acid to a pH<2, and are not being analyzed for Mercury, have a hold time of 6 months.

Samples are stored at room temperature

7. Equipment and Supplies

7.1 Thermo Q and RQ ICP-MS:

The MS features dual extraction lenses and a compound ion lens system that ensures a mass range from Li-U (masses 6-240). It also features the enhanced RAPID (Right Angle Positive Ion Deflection) lens technology (90° ion optics done right) for separation of ions and neutrals. Simultaneous analog/PC detector with real time multi-channel analyzer electronics provides >9 orders of dynamic range suitable for both steady state and transient signal analysis. The software is Qtegra which provides integrated plug-ins for autosamplers, autodiluters and one click instrument set up: with a single mouse click, the iCAP Q is taken into operate mode, tested and, where necessary intelligently autotuned and/or calibrated to meet the protocol.

7.2 CETAC ASX-510 Autosampler: Delivers sample and internal standard to the torch

7.3 Thermo X-series II ICP-MS

The Innovative Protective Ion Extraction and Infinity II ion optics, based upon a hexapole design with chicane ion deflector, provides low background specification coupled with a userinterchangeable Xt interface and Xs interface. High-performance quadrupole analyzer is pumped by a novel split flow turbo pump backed by a single rotary. Simultaneous analog/PC detector with real time multi-channel analyzer electronics provides >8 orders of dynamic range suitable for both steady state and transient signal analysis. Instrument and accessories are fully computer controlled by the PlasmaLab software. Peltier spray chamber cooling provides enhanced performance for outstanding signal /background.

7.4 Edwards E2M28 and Sogevac 40 Rotary Pumps

7.5 Neslab M-75 Chiller: Cools the torch and the MS

7.6 Eppendorf pipets: Accurate means to make trace standards

8. Reagents and Standards

8.1 Nitric Acid (HNO_3), Trace metals grade: 18M Concentrated Redistilled

8.2 1% Nitric Acid (v/v): 10mL of 18M HNO_3 diluted to 1L using Type I water

8.3 Type I De-Ionized Water

8.4 Calibration Standard: Multiple Element Standard, purchased (Table 4) and diluted. Store at room temperature. Expires upon manufacturer's specified date. Prepare as needed if confirmed with the ICV solution made fresh daily.

8.5 Internal Standard: Multiple Element Standard, purchased (Table 8). Store at room temperature. Expires upon manufacturer's specified date.

8.6 Tune Stock: Multiple Element Standard, purchased (Table 9). 100.0 ug/L solution of each ^{7}Li ^{9}Be, ^{58}Ce, ^{59}Co, ^{115}In, ^{137}Ba and ^{208}Pb, and ^{238}U.

8.7 ICV: Multiple Element Standard, second source from calibration standard (Section 8.4) purchased and diluted (Table 10). Concentrations are near but not at the mid-point of the calibration Prepare fresh daily.

8.8 CCV: Multiple Element Standard, purchased (Table 11) and diluted. Store at room temperature. Concentrations are at or near the mid-point of the calibration. Expires upon manufacturer's specified date.

8.9 SIC: High-Purity Standards Cat. #ICP-MS-ICS-2 A stock. Dilute 5 mL to 50 mL (Table 12). Store at room temperature. Prepare fresh weekly.

8.10 LDR Check Solution: A multiple element or single element solution run at a point above the highest calibration standard under the same calibration used to quantify the associated sample data. The LDR check must be within +/-10% of the true value of each element of interest and be within +/- 50% of the sample concentration to be consider valid.

8.11 Lower Limit of Quantitation (LLOQ) check standard: The laboratory should establish the LLOQ as the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve. See section 12.16.

8.12 Argon Gas: 0.9995 or better grade. High capacity tank plumbed into the lab.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Three types of blanks are required for the analysis. The calibration blank, which is also the ICB/CCB, is used in establishing the calibration curve. The ICB/CCB is used to monitor carryover, signal noise, and drift. The method blank is used to monitor for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards. See Section 12.3 for Corrective Action.

9.1.1 The Calibration blank, Initial Calibration Blank and the Continuing Calibration Blank consists of the same concentration of the same acid used to prepare the final dilution of the calibrating solutions of the analytes. This is a 1% HNO_3 solution (v/v) in Type I deionized water along with the selected concentrations of internal standard element for each of the analytes. The Calibration Blank is analyzed before the standards are analyzed. The Initial Calibration Blank must follow the Initial Calibration Verification standard (ICV) and the Continuing Calibration Blank must follow the Continuing Calibration Verification standard (CCV). These must be analyzed at a frequency of every 10 or less samples and at the end of the analytical run. The ICB must be no greater than ½ the LLOQ for any analyte; The CCB must be no greater than the LLOQ for any analyte See Section 12.11 for Corrective Action.

9.1.2 The method (or reagent) blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the sample solutions. Method blanks are generally considered to be acceptable if target analyte

concentrations are less than the LLOQ or are less than project-specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e. targets are not present in samples or sample concentrations are ≥10X the blank). Other criteria may be used depending on the needs of the project. See Section 12.8 for Corrective Action.

9.1.3 The rinse blank consists of HNO_3 (1% or 2%) (v/v) in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

9.2 Laboratory Control Sample (LCS)

The Laboratory Control Sample (LCS) must be analyzed for each analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the test samples. One LCS must be prepared and analyzed for each sample batch at a frequency of one LCS for each 20 samples or less. Aqueous LCS recoveries must be 80-120%, and soil LCS recoveries must be within the SRM limits. See Sections 12.9 and 12.10 for Corrective Actions.

9.3 Initial Calibration Verification (ICV)

The ICV must have a recovery that is within ±10% of the true value. The ICV and must contain all of the elements that are calibrated. The ICV is an independent source other than those standards used for the calibration of the instrument. The ICV must be analyzed following the calibration.

9.4 Continuing Calibration Verification (CCV)

The CCV must have a recovery that is within ±10% of the true value. The CCV must be analyzed at a frequency of 10 samples or less and at the end of the analytical run.

9.5 Matrix Spike

Analyze one matrix spike per twenty or less analytical samples. The recovery of the matrix spike must be between 75 – 125%. Calculate percent recovery using Section 11.2.

9.5.1 **Dilution Test:** If the analyte concentration is sufficiently high (minimally, a factor of 25 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution must agree within ± 20% of the original determination. Elements that fail the dilution test are reported as estimated values.

9.5.2 **Post Digestion Spike Addition:** If the sample concentrations are insufficient to perform a dilution test a post digestion spike added to a portion of a prepared sample, or its dilution for the elements failing the matrix spike recoveries must be run, recovery limits equal to 75% to 125% of the known spike value. If the spike is not recovered within the specified limits If the post-digestion recovery fails to meet the acceptance criteria, the sample results must be reported as estimated values

9.6 Laboratory Duplicate

Analyze one duplicate sample for every matrix in a batch at a frequency of one matrix duplicate for every 20 samples. Calculate RPD under section 11.3.

A control limit of 20% RPD must not be exceeded for analyte values greater than 100 times the instrument detection limit (Section 13.3). See Section 12.14 for Corrective Action.

9.7 Method-specific Quality Control Samples

9.7.1 Internal Standards

The intensities of all internal standards must be monitored for every analysis. If the intensity of any internal standard applied to a sample falls below 70% of the intensity of that internal standard in the initial calibration standard, a significant matrix effect must be suspected.

Under these conditions, the established lower limit of quantitation has degraded and the correction ability of the internal standardization technique becomes questionable. The following procedure is followed -- First, make sure the instrument has not drifted by observing the internal standard intensities in the nearest calibration blank.

If the low internal standard intensities are also seen in the nearest calibration blank, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples. If drift has not occurred, matrix effects need to be removed by dilution of the affected sample. The sample should be diluted fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. If the first dilution does not eliminate the problem, this procedure must be repeated until the internal-standard intensities rise to the minimum 70% limit. Reported results must be corrected for all dilutions.

9.7.2 Interference Check Standards

Verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Do this by analyzing the SIC check (8.9). Refer to Section 4.0 for a discussion on interferences and potential solutions to those interferences if additional guidance is needed. SIC must be below 2X LLOQ for all non-spiked elements of interest. See Section 12.8 for Corrective Action

9.7.3 Low Level Verification Standard (LLV)

The low level standard analyzed should quantitate to within 80-120% of the true value.

9.7.4 Mid Level Verification Standard (MLV)

The mid-level standard analyzed should quantitate to within 90-110% of the true value.

9.8 Method Sequence

Performance Check Solution / Tuning Solution

Calibration of instrument

Initial Calibration Verification Standard

Initial Calibration Blank

Low Level Verification Standard

Mid- Level Verification Standard

Spectral Interference Check Solution

Continuing Calibration Verification Standard

Continuing Calibration Blank

Samples (10)

Continuing Calibration Verification Standard

Continuing Calibration Blank
Samples (10)

Continuing Calibration Verification Standard

Continuing Calibration Blank

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation

Prior to analysis, samples which require total (acid-leachable) values must be digested using appropriate sample preparation methods, such as Methods 3005A, 3015, 3051 and 3050B.

10.1.2 Tuning the instrument on from Stand By

- Tighten the peristaltic pump windings for the sample, Internal standard and spray chamber drain.
- Turn on the Argon gas supply and chiller.

Thermo Q and RQ:

- Turn on the computer and monitor. Start the Qtegra software. On the front instrument tab select the ON button, select OK. The fully automatic system will advance to ignition, torch box alignment and start the sample introduction system.
- Allow the instrument to warm up for **30 minutes.**

Thermo X-series II:

- Turn on the computer and monitor. Start the Plasmalab software. On the front instrument tab select the ON button. A message box will ask if to advance from vacuum to start up, select OK. The fully automatic system will advance to ignition, torch box alignment and start the sample introduction system.
- Allow the instrument to warm up for **30 minutes.**

10.1.3 Tuning

After the 30 minute warm up period has passed, a daily tune must be performed and a report generated.

Thermo X-series II and Thermo Q:

Select autotune, introduce the Tune A solution The Instrument stock tune solution (Table 9) is a 10 µg/L, Tune A is a 10 fold dilution. Run the autotune after a minimum 40 second uptake delay.

From the drop down menu select Run Performance Check (x –series), icon (Q) with the sample probe in Tune A solution. After completion of the performance check, select print and the file will be save to the archive. Upon successfully passing the performance check the instrument is ready for calibration.

Typical values of tuning parameters

Parameter	Typical Conditions	Adjustment
RF Power (W)	1300	1200 to 1600
Sampling Depth (mm)	6	4 to 8
Carrier gas (L/min)	1.2	0.8 to 1.3
Makeup gas (L/min)	0	0 to 0.4
Peri-pump 1 (rps)	0.1	0.06 TO 0.15
S/C Temp (°C)	2	Normally used at 2 °C
Extraction	-150	-200 to -100
Extraction	-70	-150 to -10
Einzel 1.3 (V)	-100	-130 to -40
Einzel 2(V)	7	-20 to +70
Omega Bias (V)	-35	-40 to 0
Omega (+)(V)	5	0 to +30
RF Power (W)	1300	1200 to 1600

Typical value of Sensitivity and RSD (Using the normal torch)

Mass	Counts / 10ppb Integ. Time = 0.1 sec	RSD
^{7}Li	>6400	<5%
^{89}Y	>16000	<5%
^{205}Tl	>9600	<5%

10.2 Initial Calibration

Mixed calibration standard solutions are prepared by diluting the stock-standard solutions to levels within the linear range for the instrument in a solvent consisting of 5% HNO_3 (v/v) in reagent water. The calibration standard solutions must contain a suitable concentration of an appropriate internal standard for each analyte. Internal standards are added at the time of analysis using a second channel of the peristaltic pump and an appropriate mixing manifold. Generally, an internal standard must be no more than 50amu removed from the analyte. Alpha employs the following internal standards: ^{6}Li, ^{45}Sc, ^{74}Ge, ^{103}Rh, ^{115}In, ^{159}Tb, ^{175}Lu, ^{209}Bi. Lithium is excluded when analyzing for ^{6}Li/^{7}Li.

Prior to preparing the mixed standards, each stock solution must be analyzed separately to determine possible spectral interferences or the presence of impurities. Care must be taken when preparing the mixed standards that the elements are compatible and stable. Transfer the mixed standard solutions to freshly acid-cleaned HPLC bottles for storage. Fresh mixed standards must be prepared as needed with the realization that concentrations can change upon aging. Calibration standards must be initially verified using a quality control standard (ICV), and monitored daily for stability.

Calibrate the instrument for the analytes of interest (recommended isotopes for the analytes in Table 1 are provided in Table 3), using the calibration blank (Section 9.1.1) and the set of 4 standards (see Table 4.). The analytical range brackets an RDL of 0.2 µg/L to 120 µg/L with the exceptions of Fe, K, Na, Ca and Mg which bracket the range of 10µg/L to 12,000µg/L. All solutions

and standards are prepared in a 1% Nitric Acid matrix. The calibration is defined as the calibration blank and four standards. The only standards that may be discarded in the calibration are the first standard (the low standard) and the fourth standard (the high standard). The only instance when the standard may be discarded is if the linearity of the element in question does not meet the correlation coefficient acceptance criteria of 0.995 or greater. No mid-level standards are ever discarded. The LDR is defined by the LDR standard run within the same calibration and recovered with +/- 10% of the true value of each element and within +/- 50% of the sample concentration, no sample is to exceed 90% of the top LDR standard or a dilution must be performed. All standards, QC samples and samples are to be integrated three times and then averaged. The calibration block is part of the analytical sequence and includes an ICV, ICB, LLV, MLV immediately following the analysis of the calibration standards. The calibration block can also contain a pause so that the analyst can evaluate the calibration prior to sample analysis. The routine calibration sequence is as follows:

Blank #1
Cal blank
0.2 µg/L std (10 µg/L Fe, K, Na, Ca, Mg)
1.0 µg/L std (100 µg/L Fe, K, Na, Ca, Mg)
10.0 µg/L std (1000 µg/L Fe, K, Na, Ca, Mg)
120 µg/L std (12,000 µg/L Fe, K, Na, Ca, Mg)

The quality control standard is the Initial Calibration Verification solution (ICV), which must be prepared in the same acid matrix as the calibration standards. This solution must be an independent standard near but not at the midpoint of the instrument calibration. An independent standard is defined as a standard composed of analytes from a source other than that used for the standards for instrument calibration.

Immediately after the calibration has been established, the calibration must be verified and documented for every analyte by the analysis of the Initial Calibration Verification solution (ICV) (Section 8.7). The calibration is verified if the solution is within the 10% of the true value (Table 10) for each element. See Section 12 for corrective action if the ICV fails. The ICB (9.1.1) is analyzed immediately following the ICV. The ICB must be no greater than the ½ LLOQ for any analyte. See Sections 12 for Corrective Actions.

10.3 Equipment Operation and Sample Processing

10.3.1 Spectral Interference Check Solution

The interference check solution (SIC) is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interference and provide an adequate test of any corrections.

Chloride in the SIC provides a means to evaluate software corrections for chloride-related interferences such as $^{35}Cl^{16}O^{+}$ on $^{51}V^{+}$ and $^{40}Ar^{35}Cl^{+}$ on $^{75}As^{+}$.

Iron is used to demonstrate adequate resolution of the spectrometer for the determination of manganese.

Molybdenum serves to indicate oxide effects on cadmium isotopes.

The other components are present to evaluate the ability of the measurement system to correct for various molecular-ion isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits. The ICSA solution (Section 8.8) contains 20,000mg/L of Cl^{-}, 3,000 mg/L of Ca, 2,500 mg/L of Fe and Na, 2,000 mg/L of C, 1,000 mg/L of Al, K , Mg, P, S and 20 mg/L of Mo and Ti.

The non-target analytes for the SIC should have a recovery less than 2x LLOQ. Narrate nonconformance.

10.3.2 Sample Analysis and Continuing Calibration Verification

The default sample table on the instrument will include the ICV, ICB, LLV, MLV, SIC, samples and CCV and CCB. After the initial Instrument QC has been analyzed and passed, then 10 samples will be analyzed. After the tenth sample has been analyzed, a CCV and a CCB will be analyzed. Analysis of standards and samples must only take place when the instrument has come to equilibrium (typically after 30 minutes after plasma ignition). All masses which could affect data quality must be monitored to determine potential effects from matrix components on the analyte peaks. The recommended isotopes to be monitored are listed in Table 3. When the analysis is ready to start, flush the system with the rinse blank solution (Section 9.1.3) until the signal levels return to the method's level of quantitation (usually about 30 seconds) before the analysis of each sample. Nebulize each sample until a steady-state signal is achieved (usually in about 30 seconds) prior to collecting the data. Analyze the Continuing Calibration Verification solution (CCV) (Section 8.7) and the continuing Calibration blank (Section 9.1.1) at a frequency of at least once per ten analytical samples, and at the end of the analytical run. Dilute and reanalyze samples that are more concentrated than the linear range (or species needed for a correction) or measure an alternate less-abundant isotope if calibrated. The linearity at the alternate mass must be confirmed by appropriate calibration.

10.4 Preventive Maintenance

The scheduled maintenance is automatically tracked by the run time log for the following parameters. A message will appear on the computer screen reminding the analyst that a piece of maintenance must be performed. An electronic log, along with a maintenance notebook is kept. Below is a list what needs to be performed:

1. Check rough pump oil
2. Replace rough pump oil
3. Replace mist filter 4
4. Clean the extraction lenses
5. Clean the skimmer cone
6. Clean the sampling cone
7. Clean the nebulizer

11. Data Evaluation, Calculations and Reporting

The quantitative values are reported in appropriate units directly from the instrument: micrograms per liter (µg/L) for aqueous samples and milligrams per kilograms (mg/Kg) for solid samples. If dilutions were performed, the appropriate corrections must be applied to the sample values.

11.1 It is required that results for solids be reported on a dry weight basis as follows:

A separate determination of percent solids is performed by the Wet Chemistry Department and the result is loaded in the **LIMS**. To retrieve the result in the LIMS, go to **"Status"** and type in the sample ID. Click the sample number that is part of that sample ID, and click the product that says **TS-S**. The percent solids for the sample is there. LIMS will automatically

correct the sample for percent solids after the metals analysis has been **Final Metals Reviewed.** This is the calculation:

$$\text{Concentration (dry weight) (mg/kg)} = \frac{C \times V}{W \times S}$$

Where:
C= Digest Concentration (mg/L)
V= Final volume in Liters after sample preparation
W= Weight in Kg of wet sample
$S = \frac{\%\text{Solids}}{100}$

Calculations must include appropriate interference corrections (see Section 4.1 for examples), internal-standard normalization, and the summation of signals at 206, 207, and 208 m/z for Lead (to compensate for any differences in the abundances of these isotopes between samples and standards).

11.2 Calculate Percent Recovery for the Matrix Spike corrected for concentrations measured in the unfortified sample. Percent recovery is calculated using the following equation:

$$\%\ \text{Recovery} = \frac{(Cm - C)}{s} \times 100$$

Where:
Cm = measured in the fortified sample
C = measured native sample concentration
S = concentration equivalent of spike added to sample

11.3 Calculate the Relative Percent Difference (RPD) for each Duplicate of the initial quantitated concentration (IC) and duplicate quantitated concentration (Dc) using the following formula:

$$RPD = \frac{|(IC - Dc)|}{\{(IC + Dc) / 2\}} \times 100$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedances, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form

12.1 The performance check standard is included in the tuning solution. Ce and Ba are included with the tuning solution and monitor the formation of oxides and the effect of doubly-charged ions

respectively. If the solution fails >3% (Section 10.1.3) then instrument maintenance must be performed and the solution re-run.

12.2 For the Tune to pass, the mass calibration for the three tune elements (Section 10.1.3) must not differ by more than 0.1 amu from the true value and the resolution must be <0.9 amu full width at 10% peak height. Furthermore the RSD for the three tune elements must be <5% If any of these criteria fail then the mass calibration must be adjusted to the correct value. This is done by re-optimizing the instrument conditions and re-tuning.

12.3 The results of the calibration blank (Section 9.1.1) must be less than the LLOQ for each element. If this is not the case, the reason for the out-of-control condition must be found and corrected, and affected samples must be reanalyzed. If the laboratory consistently has concentrations greater than 3 times the LLOQ, the LLOQ may be indicative of an estimated LLOQ and must be re-evaluated.

12.4 Calibration is performed after the tune passes. A five point calibration is performed (Section 9.3). If more than three points are to be used for the calibration, then the resultant curve must have a correlation coefficient (cc) of 0.995 or greater. If the cc is <0.995, then the instrument must be re-optimized and re-calibrated. The calibration must define the working linear range of the curve. The reporting RL must be the low standard, and the upper linear range must be defined by the high standard.

12.5 The ICV (Section 9.3) is performed immediately after an acceptable calibration has been produced. The acceptable range for this standard is 90-110%. If this standard fails then the analysis must be stopped and the instrument re-calibrated, and the ICV re-analyzed. Analysis cannot continue until this standard passes.

12.6 The CCV (Section 9.4) is performed at a frequency of 10 samples or less and at the end of the analytical run. The acceptable range for this standard is 90-110%. If this standard fails it can be rerun once. If it continues to fail the instrument is re-calibrated, and the CCV re-analyzed. Analysis cannot continue until this standard passes. All samples run after the last passing CCV must be rerun for the CCV failures.

12.7 The ICB (Section 9.1.1) must be performed immediately after the ICV. The ICB must be ½ LLOQ. If the ICB fails then the analysis is terminated, sources of contamination are checked for, and the instrument is re-calibrated and the ICV and ICB are re-analyzed.

12.8 The Spectral Interference Check Solution (Section 9.7.2) monitor how well the system is correcting for interference. The target non-spiked elements in SIC must be below 2X LLOQ for those elements in question. If the recovery of this solution is outside of the control limits, the non-compliance must be narrated. There is no corrective action required because instrument corrections are based on natural isotope abundances that cannot be changed. If the IS is in compliance then the data is acceptable.

12.9 The Method (Preparation) Blank (Section 9.1.2) must be less than the LLOQ for all of the elements that are being analyzed. If the element in question is non-detect, and the method blank is positive then the corrective action is a narration on the final report. If the samples associated with the method blank have “hits”, and they are 10x greater than the method blank, then the corrective action is a narration on the final report. If the method blank is not less than the RL, and the associated samples have results that are greater than the RL but less than 10x the method blank, then the samples must be re-digested and re-analyzed.

12.10 If the LCS (Section 9.2) fails, then all samples associated with that batch must be re-digested and re-analyzed. (Massachusetts recognizes that if the MS passes and the LCS fails then the data can be accepted. Narrate then non-compliance. This is only for MCP projects).

12.11 Failure of the CCB (9.1.1) rerun once, if continued failure evaluate the data; if associated sample results are greater than 10x CCB level then the results are acceptable. Otherwise, recalibrate and re-analyze all samples since last compliant CCB.

12.12 If the dilution test (Section 9.5.1) fails, then an interference may be suspected. There is no corrective action to be applied. Report all elements as estimated on the final report if the sample concentration is 25x LLOQ.

12.13 If the Post–Digestion Spike (Section 9.5.2) fails, then matrix interference may be suspected and a dilution should be performed to confirm. Report all elements as estimated on the final report.

12.14 Failure of the duplicate sample (Section 9.6) must be investigated. If the sample is found to be non-homogeneous or less than 5 x the LLOQ then the non-compliance must be narrated on the final report.

12.15 If the Internal Standards (Section 9.7.1) fail the acceptance criteria, then dilute the samples until the IS passes. If the criteria are still not met, then terminate the analysis, re-calibrate, verify the new calibration, and reanalyze all of the affected samples. If IS continues to fail at dilutions, seek an alternate technology to analyze the affected samples.

12.16 The LLOQ is initially verified by the analysis of at least 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be +/- 35% of the true value and RSD should be < 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias.

Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

13. Data Evaluation, Calculations and Reporting

13.1 If dilutions were performed, the appropriate factors must be applied to sample values. All results must be reported with up to three significant figures.

13.2 Soil samples

Soil samples are calculated as follows:

$$A = \frac{\text{Sample weight (grams)}}{\text{Final Volume (mL)}}$$

$$B \text{ (concentration in mg/Kg)} = \frac{\text{Concentration of analyte (mg/L)}}{A}$$

13.2.1 Dry weight correction

The LIMS calculates the dry weight correction, however it is calculated as follows:

$$\text{Final concentration in mg/Kg dry weight} = \frac{B}{\%\ \text{Solids}}$$

13.3 Liquid samples

Liquid samples are calculated as follows:

$$\text{Dilution Factor} = \frac{\text{Final Volume (mL)}}{\text{Sample Volume (mL)}}$$

Final concentration in mg/L = Concentration of analyte (mg/L) x Dilution Factor

13.4 Calculations for Hardness

The method for determining hardness is to compute it from the results of separate determinations of Calcium and Magnesium on aqueous samples.

13.4.1 Total Hardness

Total Hardness, mg equivalent CaCO3/L = [2.497 (Ca, mg/L)] + [4.118 (Mg, mg/L)]

13.4.2 Calcium Hardness

Calcium Hardness, mg equivalent CaCO3/L = [2.497 (Ca, mg/L)]

14. Method Performance

14.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732 unless superseded in the SOP. These studies performed by the laboratory are maintained on file for review

14.2 Demonstration of Capability Studies

14.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

14.2.2 Continuing (DOC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples

14.3 Instrument Detection Limits (IDLs)

Instrument detection limits (IDLs) are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit. It may be helpful to compare the calculated IDLs to the established lower limit of quantitation, however, it should be understood that the lower limit of quantitation needs to be verified.

Instrument Detection limits (in µg/L) can be estimated by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e. each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs should be determined at least once using new equipment, after major instrument maintenance such as changing the detector, and/or at a frequency designated by the project and a record of time and date kept in the instrument maintenance log book.

15. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

16. Referenced Documents

DOC ID 2124 Chemical Hygiene Plan

SOP ID 1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

SOP ID 1739 Demonstration of Capability (DOC) Generation SOP

SOP ID 1728 Hazardous Waste Management and Disposal SOP

17. Attachments

Table 2: Reporting Limits
Table 3: Interference Check Solution Concentrations
Table 4: Calibration Standards
Table 5: Recommended Isotopes for Selected Elements
Table 6: Precision and Accuracy Acceptance Criteria
Table 7: Metals LCS Concentrations
Table 8: Internal Standard
Table 9: Tune Solution
Table 10: ICV Solution
Table 11: CCV Solution
Table 12: Interference Check Solutions
Table 13: Interference Correction Equations

Table 2
Reporting Limits

Element	Atomic Symbol	Mass (m/z)	Aqueous (µg/L)	Soil / Solid (ug/Kg)
Beryllium	Be	9	0.5	20
Sodium	Na	23	100	4000
Magnesium	Mg	24	100	4000
Aluminum	Al	27	10	400
Potassium	K	39	100	4000
Calcium	Ca	44	100	4000
Vanadium	V	51	0.50	20
Chromium	Cr	52	0.50	20
Manganese	Mn	55	0.50	20
Iron	Fe	57	50	2000
Cobalt	Co	59	0.50	20
Nickel	Ni	60	0.50	20
Copper	Cu	65	0.50	20
Zinc	Zn	66	5.0	100
Arsenic	As	75	0.50	20
Selenium	Se	82	0.50	20
Molybdenum	Mo	98	0.50	20
Silver	Ag	107	0.50	20
Cadmium	Cd	111	0.50	20
Antimony	Sb	121	0.50	20
Barium	Ba	137	0.50	20
Thallium	Tl	205	0.50	20
Lead	Pb	208	0.50	20
Lithium[1]	Li	7	N/A	100
Internal Standards				
Lithium	Li	6		
Scandium	Sc	45		
Germanium	Ge	74		
Indium	In	115		
Bismuth	Bi	209		

[1] Lithium is performed only by request for limited client projects with project specific reporting limits. Calculated Method Detection Limits are on file in the QA Department.

Table 3
Interference Check Solution

Solution Component	Solution A Concentration (ug/L)
Al	100,000
Ca	300,000
Fe	250,000
Mg	100,000
Na	250,000
P	100,000
K	100,000
S	100,000
C	200,000
Cl	2,000,000
Mo	2,000
Ti	2,000

Table 4 Calibration Standards

Multi-element standard (Section 8.4) containing the following:

High Range Elements (HR): 1000 mg/L of the following: Fe, K, Ca, Na, Mg, Sr

Low Range Elements (LR): 10 mg/L of the following: Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Th, U

All Calibration Standards are purchased in 5% HNO3, Tr HF (v/v).
Lithium is analyzed with a single standard curve matching the levels listed below for the routine curve.

Standard #1 (0.2 µg/L LR, 20 ug/L HR): Dilute Standard #3: 1.0 mL to 50mL final volume 1% HNO_3 (v/v).

Standard #2 (1.0 µg/L LR, 100 ug/L HR): Dilute Standard #3: 5.0 mL to 50mL final volume 1% HNO_3 (v/v).

Standard #3 (10.0 µg/L LR, 1000 ug/L HR): Dilute 0.05 mL to 50mL final volume 1% HNO_3 (v/v).

Standard #4 (60.0 µg/L LR, 1000 ug/L HR): Dilute 0.3 mL to 50mL final volume 1% HNO_3 (v/v).

Standard #5 (120.0 µg/L LR, 12000 ug/L HR): Dilute 0.6 mL to 50mL final volume 1% HNO_3 (v/v).

Table 5: Recommended Isotopes for Selected Elements

Mass	Element of Interest
27	Aluminum
121, **123**	Antimony
75	Arsenic
138, 137, 136, **135**, 134	Barium
9	Beryllium
209	Bismuth (IS)
114, 112, **111**, 110, 113, 116, 103	Cadmium
42, 43, **44**, 46, 48	Calcium (I)
35, 37, (77, 82)[a]	Chlorine (I)
52, **53**, **50**, 54	Chromium
59	Cobalt
63, **65**	Copper
165	Holmium (IS)
115, 113	Indium (IS)
56, **54**, **57**, 58	Iron (I)
139	Lanthanum (I)
208, **207**, **206**, 204	Lead
6[b], 7	Lithium (IS)
24, **25**, **26**	Magnesium (I)
55	Manganese
98, 96, 92, **97**, 94, (108)[a]	Molybdenum (I)
58, **60**, 62, **61**, 64	Nickel
39	Potassium (I)
103	Rhodium (IS)
45	Scandium (IS)
80, **78**, **82**, **86**, **77**, 74	Selenium
107, **109**	Silver
23	Sodium (I)
159	Terbium (IS)
205, 203	Thallium
51, **50**	Vanadium
120, **118**	Tin (I)
89	Yttrium (IS)
64, **66**, **68**, **67**, 70	Zinc

NOTE: Method 6020 is recommended for only those analytes listed in Table 1. Other elements are included in this Table because they are potential interferents (I) in the determination of recommended analytes, or because they are commonly used internal standards (IS). Isotopes are listed in descending order of natural abundance. The most useful isotopes are underlined and in boldface, although certain matrices may require the use of alternative isotopes.

[a] These masses are also useful for interference correction (Section 4.2)

[b] Internal standard must be enriched in the 6Li isotope. This minimizes interference from indigenous Li

Table 6 Precision and Accuracy Acceptance Criteria *

	Aqueous % Recovery LCS		Soil % Recovery LCS or SRM limits		Duplicate	
Element	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**	**Aqueous %RPD**	**Soil %RPD**
Aluminum	80	120	70	130	20	20
Antimony	80	120	70	130	20	20
Arsenic	80	120	70	130	20	20
Barium	80	120	70	130	20	20
Berylium	80	120	70	130	20	20
Cadmium	80	120	70	130	20	20
Calcium	80	120	70	130	20	20
Chromium	80	120	70	130	20	20
Cobalt	80	120	70	130	20	20
Copper	80	120	70	130	20	20
Iron	80	120	70	130	20	20
Lead	80	120	70	130	20	20
Magnesium	80	120	70	130	20	20
Manganese	80	120	70	130	20	20
Molybdenum	80	120	70	130	20	20
Nickel	80	120	70	130	20	20
Potassium	80	120	70	130	20	20
Selenium	80	120	70	130	20	20
Silver	80	120	70	130	20	20
Sodium	80	120	70	130	20	20
Thallium	80	120	70	130	20	20
Vanadium	80	120	70	130	20	20
Zinc	80	120	70	130	20	20

*These are default limits. The limits are re-evaluated and updated as necessary pending compilation of the minimum number of data points

Table 7 Metals LCS Concentrations

Analyte	Liquid Concentration (mg/L)
Antimony	0.5
Arsenic	0.12
Barium	2.00
Beryllium	0.05
Cadmium	0.051
Chromium	0.20
Copper	0.25
Lead	0.51
Nickel	0.50
Selenium	0.12
Silver	0.05
Thallium	0.12
Zinc	0.50
Iron	1.00
Manganese	0.50
Calcium	10.0
Magnesium	10.0
Potassium	10.0
Sodium	10.0
Aluminum	2.00
Cobalt	0.50
Vanadium	0.50

Table 8 Internal Standard

Multiple Element Standard, purchased (Section 8.5), 100mg/L of ^{6}Li, ^{45}Sc, ^{74}Ge, ^{103}Rh, ^{115}In, ^{159}Tb, ^{175}Lu, ^{209}Bi

Working Internal Standard (IS) Solution (1 mg/L solution) Dilute 10.0 mL to 1L final volume 1% HNO_3 (v/v).

Table 9 Tune Solution

Tune Solution: 100 ug/L multi-element standard, purchased for ^{7}Li ^{9}Be, ^{58}Ce ^{59}Co, ^{115}In, ^{137}Ba and ^{208} Pb, ^{238}U in 1% HNO_3 (v/v) (Section 8.6).

Working Tune Solution: Dilute 1.0 mL of 100 ug/L multi-element standard for ^{7}Li ^{9}Be, ^{58}Ce ^{59}Co, ^{115}In, ^{137}Ba and ^{208}Pb, ^{238}U with 1% HNO_3 (v/v) to 100mL final volume. This will give a final concentration of these metals at 1.0µg/L.

Table 10 ICV Solution

Multiple Element Standard, purchased (Section 8.7):

1000mg/L of K, Na, Ca, Mg, Fe, Sr
10mg/L of Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, An, Th, U

Working ICV Solution: Dilute 1.0 mL with 0.25% HNO_3 (v/v) to 50mL final volume. This will give the following concentrations:

5000µg/L of K, Na, Ca, Mg, Fe, Sr

50µg/L of Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, An, Th, U

Table 11 CCV Solution

Multi-element standard (Section 8.4) containing the following:

High Range Elements (HR): 1000 mg/L of the following: Fe, K, Ca, Na, Mg, Sr

Low Range Elements (LR): 10 mg/L of the following: Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Th, U

All Standards are purchased in 5% HNO3, Tr HF (v/v).

60.0 µg/L LR, **1000 ug/L** HR: Dilute 0.3 mL to 50mL final volume 1% HNO_3 (v/v).

Table 12 Spectral Interference Check Solutions

SIC, High-Purity Standards Cat. #ICP-MS-ICS-2 A (Section 8.8):

20000 mg/L of Cl^- **3000 mg/L Ca**
2500 mg/L Fe, Na
2000 mg/L of C
1000 mg/L of Al, K, Mg, P, S
20 mg/L of Mo, Ti

Working SIC Solution: Dilute 5 mL of High-Purity Standards Cat. #ICP-MS-ICS-2 A, with 1% HNO_3 (v/v) to 50mL final volume. This will give the following concentrations:

2000 mg/L of Cl^-
300 mg/L Ca
250 mg/L Fe, Na
200 mg/L of C
100 mg/L of Al, K, Mg, P, S
2 mg/L of Mo, Ti

Table 13
Interference Correction Equations

Correction equations are in the form: corrected signal for mass X=(signal from mass X) ± (signal from mass Y)*(correction factor).

6= (6) - ((7)*0.082)

44= (44) - ((88)*0.015)

51= (51) - ((53)*3.0)) + ((52)*0.34)

66= (66) - ((69)*0.00141)

75= (75) – ((77)*2.9) + ((82)*2.23) – ((83)*2.23)

82= (82) – (83)

98= (98) – ((99)*0.146)

111= (111) – ((108)*1.073) + ((106)*0.712)

114= (114) – ((118)*0.027)

115= (115) – ((118)*0.016)

208= (208) + (206) + (207)

Inductively Coupled Plasma - Mass Spectrometry 6020B

Reference: **Method 6020B,** Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision V, July 2014.

1. Scope and Application

Matrices: Groundwaters, aqueous samples, industrial waste, soils, sludges, sediments, and other solid wastes.

Definitions: Refer to Alpha Analytical Quality Manual.

Inductively coupled plasma-mass spectrometry (ICP-MS) is applicable to the determination of sub µg/L concentrations of a large number of elements in water samples, waste extracts or digestates.

ICP-MS has been applied to the determination of over 60 elements in various matrices. Elements for which EPA has determined the acceptability of Method 6020 in a mulit-laboratory study on solid wastes are listed below and in Table 1.

If method 6020 is used to determine any analyte not listed in Table 1 below, it is the responsibility of the analyst to demonstrate the accuracy and precision of the method in the waste to be analyzed. The analyst is always required to monitor potential sources of interferences and take appropriate action to ensure data of known quality.

Use of this method is restricted to spectroscopists who are knowledgeable in the recognition and in the correction of spectral, chemical, and physical interferences in ICP-MS.

An appropriate internal standard is required for each analyte determined by ICP-MS. Recommended Internal standards are ^{6}Li, ^{45}Sc, ^{89}Y, ^{103}Rh, ^{115}In, ^{159}Tb, ^{165}Ho, and ^{209}Bi. The Lithium internal standard must have an enriched abundance of 6Li, so that interference from Lithium native to the sample is minimized. Other elements may need to be used as internal standards when samples contain significant amounts of the recommended internal standards. The internal standard used in this method is as follows: ^{6}Li, ^{45}Sc, ^{74}Ge, ^{103}Rh, ^{115}In, ^{159}Tb, ^{175}Lu, ^{209}Bi.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the ICP-MS and in the interpretation of ICP-MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Table 1

Parameter	CAS		Parameter	CAS
Aluminum	7429-90-5		Magnesium	7439-95-4
Antimony	7440-36-0		Manganese	7439-96-5
Arsenic	7440-38-2		Lithium	7439-93-2
Barium	7440-39-3		Molybdenum	7439-98-7
Beryllium	7440-41-7		Nickel	7440-02-0
Cadmium	7440-43-9		Potassium	7440-09-7
Calcium	7440-70-2		Selenium	7782-49-2
Chromium	7440-47-3		Silver	7440-22-4
Cobalt	7440-48-4		Sodium	7440-23-5
Copper	7440-50-8		Thallium	7440-28-0
Iron	7439-89-6		Vanadium	7440-62-2
Lead	7439-92-1		Zinc	7440-66-6

2. Summary of Method

When dissolved constituents are required, samples must be filtered and acid-preserved prior to analysis. No digestion is required prior to analysis for dissolved elements in water samples. Acid digestion prior to filtration and analysis is required for groundwater, aqueous samples, industrial waste, soils, sludge's, sediments, and other solid wastes for which total (acid-leachable) elements are required.

Method 6020 describes the multi-elemental determination of analytes by ICP-MS. The method measures ions produced by a radio-frequency inductively coupled plasma. Analyte species originating in a liquid are nebulized and the resulting aerosol transported by argon gas into the plasma torch. The ions produced are entrained in the plasma gas and introduced, by means of an interface, into the mass spectrometer. The ions produced in the plasma are sorted according to their mass-to-charge ratio and quantified with a channel electron multiplier. Interferences must be assessed and valid corrections applied or the data flagged to indicate a problem. Interference correction must include compensation for background ions contributed by the plasma gas, reagents, and constituents of the sample matrix.

2.1 Method Modifications from Reference

This method is performed in a 1% Nitric Acid matrix for the calibration curve and standards and a 2% Nitric Acid matrix for the rinse.

3. Reporting Limits

Instrument detection limits, sensitivities, and linear ranges will vary with the matrices, instrumentation and operating conditions. In relatively simple matrices, detection limits will generally be below 20 µg/L. Table 2 contains the list of Reporting Limits.

4. Interferences

4.1 Isobaric Elemental Interference

Isobaric elemental interferences in ICP-MS are caused by isotopes of different elements forming atomic ions with the same nominal mass-to-charge ratio (m/z). A data system must be used to correct for these interferences. This involves determining the signal for

another isotope of the interfering element and subtracting the appropriate signal from the analyte isotope signal. Since commercial ICP-MS instruments nominally provide unit resolution at 10% of the peak height, very high ion currents at adjacent masses can also contribute to ion signals at the mass of interest. Although this type of interference is uncommon, it is not easily corrected, and samples exhibiting a significant problem of this type could require resolution improvement, matrix separation, or analysis using another verified and documented isotope, or use of another method.

4.2 Isobaric Molecular and Doubly Charged Ion Interference

Isobaric molecular and doubly-charged ion interferences in ICP-MS are caused by ions consisting of more than one atom or charge, respectively. Most isobaric interferences that could affect ICP-MS determinations have been identified in the literature. Examples include $ArCl^+$ ions on the ^{75}As signal and MoO^+ ions on the Cadmium isotopes. While the approach used to correct for molecular isobaric interferences is demonstrated below using the natural isotope abundances form the literature, the most precise coefficients for an instrument can be determined form the ratio of the net isotope signals observed for a standing solution at a concentration providing suitable (<1 percent) counting statistics. Because the ^{35}Cl natural abundance of 75.77 percent is 3.13 times the ^{37}Cl abundance of 24.23 percent, the chloride correction for arsenic can be calculated (approximately) as follows (where the $^{38}Ar^{37}Cl^+$ contribution at m/z 75 is a negligible 0.06 percent of the $^{40}Ar^{35}Cl^+$ signal):

Corrected arsenic signal (using natural isotopes abundances for coefficient approximations) = (m/z 75 signal) – (3.13) (m/z 77 signal) + (2.73) (m/z) (82 signal), (where the final term adjust for any selenium contribution at 77 m/z)

Note: Arsenic values can be biased high by this type of equation when the net signal at m/z 82 is caused by ions other than $^{82}Se^+$, (e.g. $^{81}BrH^+$ from bromine waste)

Similarly, corrected cadmium signal (using natural isotopes abundances for coefficient approximations) = (m/z 114 signal) – (0.027) (m/z 118 signal) – (1.63) (m/z 108 signal), (where the last two terms adjust for any tin or MoO^+ contributions at m/z 114).

> **Note:** Cadmium values will be biased low by this type of equation when $^{92}ZrO^+$ ions contribute at m/z 108, but use of the m/z 111 for Cd is even subject to direct ($^{94}ZrOH^+$) and indirect ($^{90}ZrO^+$) additive interferences when Zr is present
>
> **Note**: As for the arsenic equation above, the coefficients in the Cd equation are for only illustrative purposes. The most appropriate coefficients for an instrument can be determined from the ratio of the net isotope signals observed for a standard solution at a concentration providing suitable (<1 percent) counting precision.

The accuracy of these types of equations is based upon the constancy of the OBSERVED isotopic ratios for the interfering species. Corrections that presume a constant fraction of a molecular ion relative to the “parent” ion have not been found to be reliable, e.g. oxide levels can vary. If a correction for an oxide ion is based upon the ratio of parent –to-oxide ion intensities, the correction must be adjusted for the degree of outside formation by the use of an appropriate oxide internal standard previously demonstrated to form a similar level of oxide as the interferant. This type of correction has been reported for oxide-ion corrections using ThO^+/Th for the determination of rare earth elements. The use of aerosol desolvation and/or mixed plasmas has been shown to greatly reduce molecular interferences. These techniques can be used provided that method detection limits, accuracy, and precision requirements for analysis of the samples can be met.

4.3 Physical Interference

Physical interferences are associated with the sample nebulization and transport processes as well as with ion-transmission efficiencies. Nebulization and transport processes can be affected if a matrix component causes a change in surface tension or viscosity. Changes in matrix composition can cause significant signal suppression or enhancement. Dissolved solids can deposit on the nebulizer tip of a pneumatic nebulizer and on the interface skimmers (reducing the orifice size and the instrument performance). Total solid levels below 0.2% (2,000mg/L) have been currently recommended to minimize solid deposition. An internal standard can be used to correct for the physical interferences, if it is carefully matched to the analyte so that the two elements are similarly affected by the matrix change. When the intensity level of an internal standard is less than 30 percent or greater than 120 percent of the intensity of the first standard used during calibration, the sample must be reanalyzed after a fivefold (1+4) or greater dilution has been performed.

4.4 Memory Interference

Memory interferences can occur when there are large concentration differences between samples or standards which are analyzed sequentially. Sample deposition on the sampler and skimmer cones, spray chamber design, and the type of nebulizer affect the extent of the memory interferences which are observed. The rinse period between samples must be long enough to eliminate significant memory interferences.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Only Polyethylene or fluorocarbon (PFA or TFE) containers are recommended. Alpha uses polyethylene bottles. 0.5L is the recommended size.

6.2 Sample Preservation

Samples for total metals are preserved with (1:1) Nitric Acid to a pH<2.

Samples for soluble metals must be preserved with (1:1) Nitric Acid to a pH of <2 **after** filtration through a 0.45 um filter.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Samples that are to be analyzed for soluble metals, and have not been field filtered, must be filtered through a 0.45um filter as soon as possible. Samples are then preserved with 1:1 Nitric Acid to a pH<2, and then held for 18 hours. After the 18 hours the pH must be re-checked. If

after 18 hours the pH is still >2, then the sample must be re-acidified and held again for 18 hours. If the pH is till >2 after this 18 hour period, then the Inorganics Manager must be told.

Samples preserved with Nitric Acid to a pH<2, and are not being analyzed for Mercury, have a hold time of 6 months.

Samples are stored at room temperature

7. Equipment and Supplies

7.1 Thermo Q and RQ ICP-MS:

The MS features dual extraction lenses and a compound ion lens system that ensures a mass range from Li-U (masses 6-240). It also features the enhanced RAPID (Right Angle Positive Ion Deflection) lens technology (90° ion optics done right) for separation of ions and neutrals. Simultaneous analog/PC detector with real time multi-channel analyzer electronics provides >9 orders of dynamic range suitable for both steady state and transient signal analysis. The software is Qtegra which provides integrated plug-ins for autosamplers, autodiluters and one click instrument set up: with a single mouse click, the iCAP Q is taken into operate mode, tested and, where necessary intelligently autotuned and/or calibrated to meet the protocol.

7.2 CETAC ASX-510 Autosampler: Delivers sample and internal standard to the torch

7.3 Thermo X-series II ICP-MS

The Innovative Protective Ion Extraction and Infinity II ion optics, based upon a hexapole design with chicane ion deflector, provides low background specification coupled with a userinterchangeable Xt interface and Xs interface. High-performance quadrupole analyzer is pumped by a novel split flow turbo pump backed by a single rotary. Simultaneous analog/PC detector with real time multi-channel analyzer electronics provides >8 orders of dynamic range suitable for both steady state and transient signal analysis. Instrument and accessories are fully computer controlled by the PlasmaLab software. Peltier spray chamber cooling provides enhanced performance for outstanding signal /background.

7.4 Edwards E2M28 and Sogevac 40 Rotary Pumps

7.5 Neslab M-75 Chiller: Cools the torch and the MS

7.6 Eppendorf pipets: Accurate means to make trace standards

8. Reagents and Standards

8.1 Nitric Acid (HNO_3), Trace metals grade: 18M Concentrated Redistilled

8.2 1% Nitric Acid (v/v): 10mL of 18M HNO_3 diluted to 1L using Type I water

8.3 Type I De-Ionized Water

8.4 Calibration Standard: Multiple Element Standard, purchased (Table 4) and diluted. Store at room temperature. Expires upon manufacturer's specified date. Prepare as needed if confirmed with the ICV solution made fresh daily.

8.5 Internal Standard: Multiple Element Standard, purchased (Table 8). Store at room temperature. Expires upon manufacturer's specified date.

8.6 Tune Stock: Multiple Element Standard, purchased (Table 9). 100.0 ug/L solution of each ^{7}Li ^{9}Be, ^{58}Ce, ^{59}Co, ^{115}In, ^{137}Ba and ^{208}Pb, and ^{238}U.

8.7 ICV: Multiple Element Standard, second source from calibration standard (Section 8.4) purchased and diluted (Table 10). Concentrations are near but not at the mid-point of the calibration Prepare fresh daily.

8.8 CCV: Multiple Element Standard, purchased (Table 11) and diluted. Store at room temperature. Concentrations are at or near the mid-point of the calibration. Expires upon manufacturer's specified date.

8.9 SIC: High-Purity Standards Cat. #ICP-MS-ICS-2 A stock. Dilute 5 mL to 50 mL (Table 12). Store at room temperature. Prepare fresh weekly.

8.10 LDR Check Solution: A multiple element or single element solution run at a point above the highest calibration standard under the same calibration used to quantify the associated sample data. The LDR check must be within +/-10% of the true value of each element of interest and be within +/- 50% of the sample concentration to be consider valid.

8.11 Lower Limit of Quantitation (LLOQ) check standard: The laboratory should establish the LLOQ as the lowest point of quantitation which, in most cases, is the lowest concentration in the calibration curve. See section 12.16.

8.12 Argon Gas: 0.9995 or better grade. High capacity tank plumbed into the lab.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Three types of blanks are required for the analysis. The calibration blank, which is also the ICB/CCB, is used in establishing the calibration curve. The ICB/CCB is used to monitor carryover, signal noise, and drift. The method blank is used to monitor for possible contamination resulting from the sample preparation procedure. The rinse blank is used to flush the system between all samples and standards. See Section 12.3 for Corrective Action.

9.1.1 The Calibration blank, Initial Calibration Blank and the Continuing Calibration Blank consists of the same concentration of the same acid used to prepare the final dilution of the calibrating solutions of the analytes. This is a 1% HNO_3 solution (v/v) in Type I deionized water along with the selected concentrations of internal standard element for each of the analytes. The Calibration Blank is analyzed before the standards are analyzed. The Initial Calibration Blank must follow the Initial Calibration Verification standard (ICV) and the Continuing Calibration Blank must follow the Continuing Calibration Verification standard (CCV). These must be analyzed at a frequency of every 10 or less samples and at the end of the analytical run. The ICB must be no greater than ½ the LLOQ for any analyte; The CCB must be no greater than the LLOQ for any analyte See Section 12.11 for Corrective Action.

9.1.2 The method (or reagent) blank must be carried through the complete preparation procedure and contain the same volumes of reagents as the sample solutions. Method blanks are generally considered to be acceptable if target analyte

concentrations are less than the LLOQ or are less than project-specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e. targets are not present in samples or sample concentrations are ≥10X the blank). Other criteria may be used depending on the needs of the project. See Section 12.8 for Corrective Action.

9.1.3 The rinse blank consists of HNO_3 (1% or 2%) (v/v) in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

9.2 Laboratory Control Sample (LCS)

The Laboratory Control Sample (LCS) must be analyzed for each analyzed for each analyte using the same sample preparations, analytical methods and QA/QC procedures employed for the test samples. One LCS must be prepared and analyzed for each sample batch at a frequency of one LCS for each 20 samples or less. Aqueous LCS recoveries must be 80-120%, and soil LCS recoveries must be within the SRM limits. See Sections 12.9 and 12.10 for Corrective Actions.

9.3 Initial Calibration Verification (ICV)

The ICV must have a recovery that is within ±10% of the true value. The ICV and must contain all of the elements that are calibrated. The ICV is an independent source other than those standards used for the calibration of the instrument. The ICV must be analyzed following the calibration.

9.4 Continuing Calibration Verification (CCV)

The CCV must have a recovery that is within ±10% of the true value. The CCV must be analyzed at a frequency of 10 samples or less and at the end of the analytical run.

9.5 Matrix Spike

Analyze one matrix spike per twenty or less analytical samples. The recovery of the matrix spike must be between 75 – 125%. Calculate percent recovery using Section 11.2.

9.5.1 **Dilution Test:** If the analyte concentration is sufficiently high (minimally, a factor of 25 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution must agree within ± 20% of the original determination. Elements that fail the dilution test are reported as estimated values.

9.5.2 **Post Digestion Spike Addition:** If the sample concentrations are insufficient to perform a dilution test a post digestion spike added to a portion of a prepared sample, or its dilution for the elements failing the matrix spike recoveries must be run, recovery limits equal to 75% to 125% of the known spike value. If the spike is not recovered within the specified limits If the post-digestion recovery fails to meet the acceptance criteria, the sample results must be reported as estimated values

9.6 Laboratory Duplicate

Analyze one duplicate sample for every matrix in a batch at a frequency of one matrix duplicate for every 20 samples. Calculate RPD under section 11.3.

A control limit of 20% RPD must not be exceeded for analyte values greater than 100 times the instrument detection limit (Section 13.3). See Section 12.14 for Corrective Action.

9.7 Method-specific Quality Control Samples

9.7.1 Internal Standards

The intensities of all internal standards must be monitored for every analysis. If the intensity of any internal standard applied to a sample falls below 70% of the intensity of that internal standard in the initial calibration standard, a significant matrix effect must be suspected.

Under these conditions, the established lower limit of quantitation has degraded and the correction ability of the internal standardization technique becomes questionable. The following procedure is followed -- First, make sure the instrument has not drifted by observing the internal standard intensities in the nearest calibration blank.

If the low internal standard intensities are also seen in the nearest calibration blank, terminate the analysis, correct the problem, recalibrate, verify the new calibration, and reanalyze the affected samples. If drift has not occurred, matrix effects need to be removed by dilution of the affected sample. The sample should be diluted fivefold (1+4) and reanalyzed with the addition of appropriate amounts of internal standards. If the first dilution does not eliminate the problem, this procedure must be repeated until the internal-standard intensities rise to the minimum 70% limit. Reported results must be corrected for all dilutions.

9.7.2 Interference Check Standards

Verify the magnitude of elemental and molecular-ion isobaric interferences and the adequacy of any corrections at the beginning of an analytical run or once every 12 hours, whichever is more frequent. Do this by analyzing the SIC check (8.9). Refer to Section 4.0 for a discussion on interferences and potential solutions to those interferences if additional guidance is needed. SIC must be below 2X LLOQ for all non-spiked elements of interest. See Section 12.8 for Corrective Action

9.7.3 Low Level Verification Standard (LLV)

The low level standard analyzed should quantitate to within 80-120% of the true value.

9.7.4 Mid Level Verification Standard (MLV)

The mid-level standard analyzed should quantitate to within 90-110% of the true value.

9.8 Method Sequence

Performance Check Solution / Tuning Solution

Calibration of instrument

Initial Calibration Verification Standard

Initial Calibration Blank

Low Level Verification Standard

Mid- Level Verification Standard

Spectral Interference Check Solution

Continuing Calibration Verification Standard

Continuing Calibration Blank

Samples (10)

Continuing Calibration Verification Standard

Continuing Calibration Blank
Samples (10)

Continuing Calibration Verification Standard

Continuing Calibration Blank

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation

Prior to analysis, samples which require total (acid-leachable) values must be digested using appropriate sample preparation methods, such as Methods 3005A, 3015, 3051 and 3050B.

10.1.2 Tuning the instrument on from Stand By

- Tighten the peristaltic pump windings for the sample, Internal standard and spray chamber drain.
- Turn on the Argon gas supply and chiller.

Thermo Q and RQ:

- Turn on the computer and monitor. Start the Qtegra software. On the front instrument tab select the ON button, select OK. The fully automatic system will advance to ignition, torch box alignment and start the sample introduction system.
- Allow the instrument to warm up for **30 minutes.**

Thermo X-series II:

- Turn on the computer and monitor. Start the Plasmalab software. On the front instrument tab select the ON button. A message box will ask if to advance from vacuum to start up, select OK. The fully automatic system will advance to ignition, torch box alignment and start the sample introduction system.
- Allow the instrument to warm up for **30 minutes.**

10.1.3 Tuning

After the 30 minute warm up period has passed, a daily tune must be performed and a report generated.

Thermo X-series II and Thermo Q:

Select autotune, introduce the Tune A solution The Instrument stock tune solution (Table 9) is a 10 µg/L, Tune A is a 10 fold dilution. Run the autotune after a minimum 40 second uptake delay.

From the drop down menu select Run Performance Check (x –series), icon (Q) with the sample probe in Tune A solution. After completion of the performance check, select print and the file will be save to the archive. Upon successfully passing the performance check the instrument is ready for calibration.

Typical values of tuning parameters

Parameter	Typical Conditions	Adjustment
RF Power (W)	1300	1200 to 1600
Sampling Depth (mm)	6	4 to 8
Carrier gas (L/min)	1.2	0.8 to 1.3
Makeup gas (L/min)	0	0 to 0.4
Peri-pump 1 (rps)	0.1	0.06 TO 0.15
S/C Temp (°C)	2	Normally used at 2 °C
Extraction	-150	-200 to -100
Extraction	-70	-150 to -10
Einzel 1.3 (V)	-100	-130 to -40
Einzel 2(V)	7	-20 to +70
Omega Bias (V)	-35	-40 to 0
Omega (+)(V)	5	0 to +30
RF Power (W)	1300	1200 to 1600

Typical value of Sensitivity and RSD (Using the normal torch)

Mass	Counts / 10ppb Integ. Time = 0.1 sec	RSD
^{7}Li	>6400	<5%
^{89}Y	>16000	<5%
^{205}Tl	>9600	<5%

10.2 Initial Calibration

Mixed calibration standard solutions are prepared by diluting the stock-standard solutions to levels within the linear range for the instrument in a solvent consisting of 5% HNO_3 (v/v) in reagent water. The calibration standard solutions must contain a suitable concentration of an appropriate internal standard for each analyte. Internal standards are added at the time of analysis using a second channel of the peristaltic pump and an appropriate mixing manifold. Generally, an internal standard must be no more than 50amu removed from the analyte. Alpha employs the following internal standards: ^{6}Li, ^{45}Sc, ^{74}Ge, ^{103}Rh, ^{115}In, ^{159}Tb, ^{175}Lu, ^{209}Bi. Lithium is excluded when analyzing for ^{6}Li/^{7}Li.

Prior to preparing the mixed standards, each stock solution must be analyzed separately to determine possible spectral interferences or the presence of impurities. Care must be taken when preparing the mixed standards that the elements are compatible and stable. Transfer the mixed standard solutions to freshly acid-cleaned HPLC bottles for storage. Fresh mixed standards must be prepared as needed with the realization that concentrations can change upon aging. Calibration standards must be initially verified using a quality control standard (ICV), and monitored daily for stability.

Calibrate the instrument for the analytes of interest (recommended isotopes for the analytes in Table 1 are provided in Table 3), using the calibration blank (Section 9.1.1) and the set of 4 standards (see Table 4.). The analytical range brackets an RDL of 0.2 µg/L to 120 µg/L with the exceptions of Fe, K, Na, Ca and Mg which bracket the range of 10µg/L to 12,000µg/L. All solutions

and standards are prepared in a 1% Nitric Acid matrix. The calibration is defined as the calibration blank and four standards. The only standards that may be discarded in the calibration are the first standard (the low standard) and the fourth standard (the high standard). The only instance when the standard may be discarded is if the linearity of the element in question does not meet the correlation coefficient acceptance criteria of 0.995 or greater. No mid-level standards are ever discarded. The LDR is defined by the LDR standard run within the same calibration and recovered with +/- 10% of the true value of each element and within +/- 50% of the sample concentration, no sample is to exceed 90% of the top LDR standard or a dilution must be performed. All standards, QC samples and samples are to be integrated three times and then averaged. The calibration block is part of the analytical sequence and includes an ICV, ICB, LLV, MLV immediately following the analysis of the calibration standards. The calibration block can also contain a pause so that the analyst can evaluate the calibration prior to sample analysis. The routine calibration sequence is as follows:

Blank #1
Cal blank
0.2 µg/L std (10 µg/L Fe, K, Na, Ca, Mg)
1.0 µg/L std (100 µg/L Fe, K, Na, Ca, Mg)
10.0 µg/L std (1000 µg/L Fe, K, Na, Ca, Mg)
120 µg/L std (12,000 µg/L Fe, K, Na, Ca, Mg)

The quality control standard is the Initial Calibration Verification solution (ICV), which must be prepared in the same acid matrix as the calibration standards. This solution must be an independent standard near but not at the midpoint of the instrument calibration. An independent standard is defined as a standard composed of analytes from a source other than that used for the standards for instrument calibration.

Immediately after the calibration has been established, the calibration must be verified and documented for every analyte by the analysis of the Initial Calibration Verification solution (ICV) (Section 8.7). The calibration is verified if the solution is within the 10% of the true value (Table 10) for each element. See Section 12 for corrective action if the ICV fails. The ICB (9.1.1) is analyzed immediately following the ICV. The ICB must be no greater than the ½ LLOQ for any analyte. See Sections 12 for Corrective Actions.

10.3 Equipment Operation and Sample Processing

10.3.1 Spectral Interference Check Solution

The interference check solution (SIC) is prepared to contain known concentrations of interfering elements that will demonstrate the magnitude of interference and provide an adequate test of any corrections.

Chloride in the SIC provides a means to evaluate software corrections for chloride-related interferences such as $^{35}Cl^{16}O^{+}$ on $^{51}V^{+}$ and $^{40}Ar^{35}Cl^{+}$ on $^{75}As^{+}$.

Iron is used to demonstrate adequate resolution of the spectrometer for the determination of manganese.

Molybdenum serves to indicate oxide effects on cadmium isotopes.

The other components are present to evaluate the ability of the measurement system to correct for various molecular-ion isobaric interferences. The ICS is used to verify that the interference levels are corrected by the data system within quality control limits. The ICSA solution (Section 8.8) contains 20,000mg/L of Cl^{-}, 3,000 mg/L of Ca, 2,500 mg/L of Fe and Na, 2,000 mg/L of C, 1,000 mg/L of Al, K , Mg, P, S and 20 mg/L of Mo and Ti.

The non-target analytes for the SIC should have a recovery less than 2x LLOQ. Narrate nonconformance.

10.3.2 Sample Analysis and Continuing Calibration Verification

The default sample table on the instrument will include the ICV, ICB, LLV, MLV, SIC, samples and CCV and CCB. After the initial Instrument QC has been analyzed and passed, then 10 samples will be analyzed. After the tenth sample has been analyzed, a CCV and a CCB will be analyzed. Analysis of standards and samples must only take place when the instrument has come to equilibrium (typically after 30 minutes after plasma ignition). All masses which could affect data quality must be monitored to determine potential effects from matrix components on the analyte peaks. The recommended isotopes to be monitored are listed in Table 3. When the analysis is ready to start, flush the system with the rinse blank solution (Section 9.1.3) until the signal levels return to the method's level of quantitation (usually about 30 seconds) before the analysis of each sample. Nebulize each sample until a steady-state signal is achieved (usually in about 30 seconds) prior to collecting the data. Analyze the Continuing Calibration Verification solution (CCV) (Section 8.7) and the continuing Calibration blank (Section 9.1.1) at a frequency of at least once per ten analytical samples, and at the end of the analytical run. Dilute and reanalyze samples that are more concentrated than the linear range (or species needed for a correction) or measure an alternate less-abundant isotope if calibrated. The linearity at the alternate mass must be confirmed by appropriate calibration.

10.4 Preventive Maintenance

The scheduled maintenance is automatically tracked by the run time log for the following parameters. A message will appear on the computer screen reminding the analyst that a piece of maintenance must be performed. An electronic log, along with a maintenance notebook is kept. Below is a list what needs to be performed:

1. Check rough pump oil
2. Replace rough pump oil
3. Replace mist filter 4
4. Clean the extraction lenses
5. Clean the skimmer cone
6. Clean the sampling cone
7. Clean the nebulizer

11. Data Evaluation, Calculations and Reporting

The quantitative values are reported in appropriate units directly from the instrument: micrograms per liter (µg/L) for aqueous samples and milligrams per kilograms (mg/Kg) for solid samples. If dilutions were performed, the appropriate corrections must be applied to the sample values.

11.1 It is required that results for solids be reported on a dry weight basis as follows:

A separate determination of percent solids is performed by the Wet Chemistry Department and the result is loaded in the **LIMS**. To retrieve the result in the LIMS, go to **"Status"** and type in the sample ID. Click the sample number that is part of that sample ID, and click the product that says **TS-S**. The percent solids for the sample is there. LIMS will automatically

correct the sample for percent solids after the metals analysis has been **Final Metals Reviewed.** This is the calculation:

$$\text{Concentration (dry weight) (mg/kg)} = \frac{C \times V}{W \times S}$$

Where:
C= Digest Concentration (mg/L)
V= Final volume in Liters after sample preparation
W= Weight in Kg of wet sample
$S = \frac{\%\text{Solids}}{100}$

Calculations must include appropriate interference corrections (see Section 4.1 for examples), internal-standard normalization, and the summation of signals at 206, 207, and 208 m/z for Lead (to compensate for any differences in the abundances of these isotopes between samples and standards).

11.2 Calculate Percent Recovery for the Matrix Spike corrected for concentrations measured in the unfortified sample. Percent recovery is calculated using the following equation:

$$\%\ \text{Recovery} = \frac{(Cm - C)}{s} \times 100$$

Where:
Cm = measured in the fortified sample
C = measured native sample concentration
S = concentration equivalent of spike added to sample

11.3 Calculate the Relative Percent Difference (RPD) for each Duplicate of the initial quantitated concentration (IC) and duplicate quantitated concentration (Dc) using the following formula:

$$RPD = \frac{|(IC - Dc)|}{\{(IC + Dc) / 2\}} \times 100$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedances, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form

12.1 The performance check standard is included in the tuning solution. Ce and Ba are included with the tuning solution and monitor the formation of oxides and the effect of doubly-charged ions

respectively. If the solution fails >3% (Section 10.1.3) then instrument maintenance must be performed and the solution re-run.

12.2 For the Tune to pass, the mass calibration for the three tune elements (Section 10.1.3) must not differ by more than 0.1 amu from the true value and the resolution must be <0.9 amu full width at 10% peak height. Furthermore the RSD for the three tune elements must be <5% If any of these criteria fail then the mass calibration must be adjusted to the correct value. This is done by re-optimizing the instrument conditions and re-tuning.

12.3 The results of the calibration blank (Section 9.1.1) must be less than the LLOQ for each element. If this is not the case, the reason for the out-of-control condition must be found and corrected, and affected samples must be reanalyzed. If the laboratory consistently has concentrations greater than 3 times the LLOQ, the LLOQ may be indicative of an estimated LLOQ and must be re-evaluated.

12.4 Calibration is performed after the tune passes. A five point calibration is performed (Section 9.3). If more than three points are to be used for the calibration, then the resultant curve must have a correlation coefficient (cc) of 0.995 or greater. If the cc is <0.995, then the instrument must be re-optimized and re-calibrated. The calibration must define the working linear range of the curve. The reporting RL must be the low standard, and the upper linear range must be defined by the high standard.

12.5 The ICV (Section 9.3) is performed immediately after an acceptable calibration has been produced. The acceptable range for this standard is 90-110%. If this standard fails then the analysis must be stopped and the instrument re-calibrated, and the ICV re-analyzed. Analysis cannot continue until this standard passes.

12.6 The CCV (Section 9.4) is performed at a frequency of 10 samples or less and at the end of the analytical run. The acceptable range for this standard is 90-110%. If this standard fails it can be rerun once. If it continues to fail the instrument is re-calibrated, and the CCV re-analyzed. Analysis cannot continue until this standard passes. All samples run after the last passing CCV must be rerun for the CCV failures.

12.7 The ICB (Section 9.1.1) must be performed immediately after the ICV. The ICB must be ½ LLOQ. If the ICB fails then the analysis is terminated, sources of contamination are checked for, and the instrument is re-calibrated and the ICV and ICB are re-analyzed.

12.8 The Spectral Interference Check Solution (Section 9.7.2) monitor how well the system is correcting for interference. The target non-spiked elements in SIC must be below 2X LLOQ for those elements in question. If the recovery of this solution is outside of the control limits, the non-compliance must be narrated. There is no corrective action required because instrument corrections are based on natural isotope abundances that cannot be changed. If the IS is in compliance then the data is acceptable.

12.9 The Method (Preparation) Blank (Section 9.1.2) must be less than the LLOQ for all of the elements that are being analyzed. If the element in question is non-detect, and the method blank is positive then the corrective action is a narration on the final report. If the samples associated with the method blank have “hits”, and they are 10x greater than the method blank, then the corrective action is a narration on the final report. If the method blank is not less than the RL, and the associated samples have results that are greater than the RL but less than 10x the method blank, then the samples must be re-digested and re-analyzed.

12.10 If the LCS (Section 9.2) fails, then all samples associated with that batch must be re-digested and re-analyzed. (Massachusetts recognizes that if the MS passes and the LCS fails then the data can be accepted. Narrate then non-compliance. This is only for MCP projects).

12.11 Failure of the CCB (9.1.1) rerun once, if continued failure evaluate the data; if associated sample results are greater than 10x CCB level then the results are acceptable. Otherwise, recalibrate and re-analyze all samples since last compliant CCB.

12.12 If the dilution test (Section 9.5.1) fails, then an interference may be suspected. There is no corrective action to be applied. Report all elements as estimated on the final report if the sample concentration is 25x LLOQ.

12.13 If the Post–Digestion Spike (Section 9.5.2) fails, then matrix interference may be suspected and a dilution should be performed to confirm. Report all elements as estimated on the final report.

12.14 Failure of the duplicate sample (Section 9.6) must be investigated. If the sample is found to be non-homogeneous or less than 5 x the LLOQ then the non-compliance must be narrated on the final report.

12.15 If the Internal Standards (Section 9.7.1) fail the acceptance criteria, then dilute the samples until the IS passes. If the criteria are still not met, then terminate the analysis, re-calibrate, verify the new calibration, and reanalyze all of the affected samples. If IS continues to fail at dilutions, seek an alternate technology to analyze the affected samples.

12.16 The LLOQ is initially verified by the analysis of at least 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be +/- 35% of the true value and RSD should be < 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias.

Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

13. Data Evaluation, Calculations and Reporting

13.1 If dilutions were performed, the appropriate factors must be applied to sample values. All results must be reported with up to three significant figures.

13.2 Soil samples

Soil samples are calculated as follows:

$$A = \frac{\text{Sample weight (grams)}}{\text{Final Volume (mL)}}$$

$$B \text{ (concentration in mg/Kg)} = \frac{\text{Concentration of analyte (mg/L)}}{A}$$

13.2.1 Dry weight correction

The LIMS calculates the dry weight correction, however it is calculated as follows:

$$\text{Final concentration in mg/Kg dry weight} = \frac{B}{\%\ \text{Solids}}$$

13.3 Liquid samples

Liquid samples are calculated as follows:

$$\text{Dilution Factor} = \frac{\text{Final Volume (mL)}}{\text{Sample Volume (mL)}}$$

Final concentration in mg/L = Concentration of analyte (mg/L) x Dilution Factor

13.4 Calculations for Hardness

The method for determining hardness is to compute it from the results of separate determinations of Calcium and Magnesium on aqueous samples.

13.4.1 Total Hardness

Total Hardness, mg equivalent CaCO3/L = [2.497 (Ca, mg/L)] + [4.118 (Mg, mg/L)]

13.4.2 Calcium Hardness

Calcium Hardness, mg equivalent CaCO3/L = [2.497 (Ca, mg/L)]

14. Method Performance

14.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732 unless superseded in the SOP. These studies performed by the laboratory are maintained on file for review

14.2 Demonstration of Capability Studies

14.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

14.2.2 Continuing (DOC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples

14.3 Instrument Detection Limits (IDLs)

Instrument detection limits (IDLs) are useful means to evaluate the instrument noise level and response changes over time for each analyte from a series of reagent blank analyses to obtain a calculated concentration. They are not to be confused with the lower limit of quantitation, nor should they be used in establishing this limit. It may be helpful to compare the calculated IDLs to the established lower limit of quantitation, however, it should be understood that the lower limit of quantitation needs to be verified.

Instrument Detection limits (in µg/L) can be estimated by calculating the average of the standard deviations of the three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day. Each measurement must be performed as though it were a separate analytical sample (i.e. each measurement must be followed by a rinse and/or any other procedure normally performed between the analysis of separate samples). IDLs should be determined at least once using new equipment, after major instrument maintenance such as changing the detector, and/or at a frequency designated by the project and a record of time and date kept in the instrument maintenance log book.

15. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

16. Referenced Documents

DOC ID 2124 Chemical Hygiene Plan

SOP ID 1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

SOP ID 1739 Demonstration of Capability (DOC) Generation SOP

SOP ID 1728 Hazardous Waste Management and Disposal SOP

17. Attachments

Table 2: Reporting Limits
Table 3: Interference Check Solution Concentrations
Table 4: Calibration Standards
Table 5: Recommended Isotopes for Selected Elements
Table 6: Precision and Accuracy Acceptance Criteria
Table 7: Metals LCS Concentrations
Table 8: Internal Standard
Table 9: Tune Solution
Table 10: ICV Solution
Table 11: CCV Solution
Table 12: Interference Check Solutions
Table 13: Interference Correction Equations

Table 2
Reporting Limits

Element	Atomic Symbol	Mass (m/z)	Aqueous (µg/L)	Soil / Solid (ug/Kg)
Beryllium	Be	9	0.5	20
Sodium	Na	23	100	4000
Magnesium	Mg	24	100	4000
Aluminum	Al	27	10	400
Potassium	K	39	100	4000
Calcium	Ca	44	100	4000
Vanadium	V	51	0.50	20
Chromium	Cr	52	0.50	20
Manganese	Mn	55	0.50	20
Iron	Fe	57	50	2000
Cobalt	Co	59	0.50	20
Nickel	Ni	60	0.50	20
Copper	Cu	65	0.50	20
Zinc	Zn	66	5.0	100
Arsenic	As	75	0.50	20
Selenium	Se	82	0.50	20
Molybdenum	Mo	98	0.50	20
Silver	Ag	107	0.50	20
Cadmium	Cd	111	0.50	20
Antimony	Sb	121	0.50	20
Barium	Ba	137	0.50	20
Thallium	Tl	205	0.50	20
Lead	Pb	208	0.50	20
Lithium[1]	Li	7	N/A	100
Internal Standards				
Lithium	Li	6		
Scandium	Sc	45		
Germanium	Ge	74		
Indium	In	115		
Bismuth	Bi	209		

[1] Lithium is performed only by request for limited client projects with project specific reporting limits. Calculated Method Detection Limits are on file in the QA Department.

Table 3
Interference Check Solution

Solution Component	Solution A Concentration (ug/L)
Al	100,000
Ca	300,000
Fe	250,000
Mg	100,000
Na	250,000
P	100,000
K	100,000
S	100,000
C	200,000
Cl	2,000,000
Mo	2,000
Ti	2,000

Table 4 Calibration Standards

Multi-element standard (Section 8.4) containing the following:

High Range Elements (HR): 1000 mg/L of the following: Fe, K, Ca, Na, Mg, Sr

Low Range Elements (LR): 10 mg/L of the following: Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Th, U

All Calibration Standards are purchased in 5% HNO3, Tr HF (v/v).
Lithium is analyzed with a single standard curve matching the levels listed below for the routine curve.

Standard #1 (0.2 µg/L LR, 20 ug/L HR): Dilute Standard #3: 1.0 mL to 50mL final volume 1% HNO_3 (v/v).

Standard #2 (1.0 µg/L LR, 100 ug/L HR): Dilute Standard #3: 5.0 mL to 50mL final volume 1% HNO_3 (v/v).

Standard #3 (10.0 µg/L LR, 1000 ug/L HR): Dilute 0.05 mL to 50mL final volume 1% HNO_3 (v/v).

Standard #4 (60.0 µg/L LR, 1000 ug/L HR): Dilute 0.3 mL to 50mL final volume 1% HNO_3 (v/v).

Standard #5 (120.0 µg/L LR, 12000 ug/L HR): Dilute 0.6 mL to 50mL final volume 1% HNO_3 (v/v).

Table 5: Recommended Isotopes for Selected Elements

Mass	Element of Interest
27	Aluminum
121, **123**	Antimony
75	Arsenic
138, 137, 136, **135**, 134	Barium
9	Beryllium
209	Bismuth (IS)
114, 112, **111**, 110, 113, 116, 103	Cadmium
42, 43, **44**, 46, 48	Calcium (I)
35, 37, (77, 82)[a]	Chlorine (I)
52, **53**, **50**, 54	Chromium
59	Cobalt
63, **65**	Copper
165	Holmium (IS)
115, 113	Indium (IS)
56, **54**, **57**, 58	Iron (I)
139	Lanthanum (I)
208, **207**, **206**, 204	Lead
6[b], 7	Lithium (IS)
24, **25**, **26**	Magnesium (I)
55	Manganese
98, 96, 92, **97**, 94, (108)[a]	Molybdenum (I)
58, **60**, 62, **61**, 64	Nickel
39	Potassium (I)
103	Rhodium (IS)
45	Scandium (IS)
80, **78**, **82**, **86**, **77**, 74	Selenium
107, **109**	Silver
23	Sodium (I)
159	Terbium (IS)
205, 203	Thallium
51, **50**	Vanadium
120, **118**	Tin (I)
89	Yttrium (IS)
64, **66**, **68**, **67**, 70	Zinc

NOTE: Method 6020 is recommended for only those analytes listed in Table 1. Other elements are included in this Table because they are potential interferents (I) in the determination of recommended analytes, or because they are commonly used internal standards (IS). Isotopes are listed in descending order of natural abundance. The most useful isotopes are underlined and in boldface, although certain matrices may require the use of alternative isotopes.

[a] These masses are also useful for interference correction (Section 4.2)

[b] Internal standard must be enriched in the 6Li isotope. This minimizes interference from indigenous Li

Table 6 Precision and Accuracy Acceptance Criteria *

	Aqueous % Recovery LCS		Soil % Recovery LCS or SRM limits		Duplicate	
Element	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**	**Aqueous %RPD**	**Soil %RPD**
Aluminum	80	120	70	130	20	20
Antimony	80	120	70	130	20	20
Arsenic	80	120	70	130	20	20
Barium	80	120	70	130	20	20
Berylium	80	120	70	130	20	20
Cadmium	80	120	70	130	20	20
Calcium	80	120	70	130	20	20
Chromium	80	120	70	130	20	20
Cobalt	80	120	70	130	20	20
Copper	80	120	70	130	20	20
Iron	80	120	70	130	20	20
Lead	80	120	70	130	20	20
Magnesium	80	120	70	130	20	20
Manganese	80	120	70	130	20	20
Molybdenum	80	120	70	130	20	20
Nickel	80	120	70	130	20	20
Potassium	80	120	70	130	20	20
Selenium	80	120	70	130	20	20
Silver	80	120	70	130	20	20
Sodium	80	120	70	130	20	20
Thallium	80	120	70	130	20	20
Vanadium	80	120	70	130	20	20
Zinc	80	120	70	130	20	20

*These are default limits. The limits are re-evaluated and updated as necessary pending compilation of the minimum number of data points

Table 7 Metals LCS Concentrations

Analyte	Liquid Concentration (mg/L)
Antimony	0.5
Arsenic	0.12
Barium	2.00
Beryllium	0.05
Cadmium	0.051
Chromium	0.20
Copper	0.25
Lead	0.51
Nickel	0.50
Selenium	0.12
Silver	0.05
Thallium	0.12
Zinc	0.50
Iron	1.00
Manganese	0.50
Calcium	10.0
Magnesium	10.0
Potassium	10.0
Sodium	10.0
Aluminum	2.00
Cobalt	0.50
Vanadium	0.50

Table 8 Internal Standard

Multiple Element Standard, purchased (Section 8.5), 100mg/L of ^{6}Li, ^{45}Sc, ^{74}Ge, ^{103}Rh, ^{115}In, ^{159}Tb, ^{175}Lu, ^{209}Bi

Working Internal Standard (IS) Solution (1 mg/L solution) Dilute 10.0 mL to 1L final volume 1% HNO_3 (v/v).

Table 9 Tune Solution

Tune Solution: 100 ug/L multi-element standard, purchased for ^{7}Li ^{9}Be, ^{58}Ce ^{59}Co, ^{115}In, ^{137}Ba and ^{208} Pb, ^{238}U in 1% HNO_3 (v/v) (Section 8.6).

Working Tune Solution: Dilute 1.0 mL of 100 ug/L multi-element standard for ^{7}Li ^{9}Be, ^{58}Ce ^{59}Co, ^{115}In, ^{137}Ba and ^{208}Pb, ^{238}U with 1% HNO_3 (v/v) to 100mL final volume. This will give a final concentration of these metals at 1.0µg/L.

Table 10 ICV Solution

Multiple Element Standard, purchased (Section 8.7):

1000mg/L of K, Na, Ca, Mg, Fe, Sr
10mg/L of Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, An, Th, U

Working ICV Solution: Dilute 1.0 mL with 0.25% HNO_3 (v/v) to 50mL final volume. This will give the following concentrations:

5000µg/L of K, Na, Ca, Mg, Fe, Sr

50µg/L of Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, An, Th, U

Table 11 CCV Solution

Multi-element standard (Section 8.4) containing the following:

High Range Elements (HR): 1000 mg/L of the following: Fe, K, Ca, Na, Mg, Sr

Low Range Elements (LR): 10 mg/L of the following: Ag, Al, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Sb, Se, Tl, V, Zn, Th, U

All Standards are purchased in 5% HNO3, Tr HF (v/v).

60.0 µg/L LR, **1000 ug/L** HR: Dilute 0.3 mL to 50mL final volume 1% HNO_3 (v/v).

Table 12 Spectral Interference Check Solutions

SIC, High-Purity Standards Cat. #ICP-MS-ICS-2 A (Section 8.8):

20000 mg/L of Cl^- **3000 mg/L Ca**
2500 mg/L Fe, Na
2000 mg/L of C
1000 mg/L of Al, K, Mg, P, S
20 mg/L of Mo, Ti

Working SIC Solution: Dilute 5 mL of High-Purity Standards Cat. #ICP-MS-ICS-2 A, with 1% HNO_3 (v/v) to 50mL final volume. This will give the following concentrations:

2000 mg/L of Cl^-
300 mg/L Ca
250 mg/L Fe, Na
200 mg/L of C
100 mg/L of Al, K, Mg, P, S
2 mg/L of Mo, Ti

Table 13
Interference Correction Equations

Correction equations are in the form: corrected signal for mass X=(signal from mass X) ± (signal from mass Y)*(correction factor).

6= (6) - ((7)*0.082)

44= (44) - ((88)*0.015)

51= (51) - ((53)*3.0)) + ((52)*0.34)

66= (66) - ((69)*0.00141)

75= (75) – ((77)*2.9) + ((82)*2.23) – ((83)*2.23)

82= (82) – (83)

98= (98) – ((99)*0.146)

111= (111) – ((108)*1.073) + ((106)*0.712)

114= (114) – ((118)*0.027)

115= (115) – ((118)*0.016)

208= (208) + (206) + (207)

Low Level Mercury Determination by Purge and Trap Cold Vapor Atomic Fluorescence Technique (CVAF) Using CETAC M-8000

References: **Method 1631, Rev. E,** Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Fluorescence Spectrometry. U.S. EPA Office of Water, Office of Science and Technology. August 2002. EPA-821-R-02-019.

Appendix to Method 1631, Total Mercury in Tissue, Sludge, Sediment, and Soil by Acid Digestion and BrCl Oxidation, January 2001. EPA-821-R-01-013

Determination of Mercury in Seawater at Sub-Nanogram per Liter Levels. Bloom, N.S. and E.A. Crecilius, Marine Chemistry, 14, 49-59. 1983.

1. Scope and Application

Matrices: The method is applicable to total and dissolved mercury in aqueous samples, tissue, sludge, sediment and soil.

Definitions: Refer to Alpha Analytical Quality Manual.

Three procedures for the analysis of mercury by cold vapor atomic fluorescence spectrometry are discussed in this SOP.

Method 1631 for aqueous samples

This procedure utilizes a bromine chloride digestion. Samples are then decolorized using hydroxylamine hydrochloride (optional) and analyzed following reduction with stannous chloride. Elemental mercury is then purged from solution and collected on a gold trap. The trap is heated to desorb mercury and analyzed by atomic fluorescence spectrometry. The reporting limit is 0.5 ng/L and the linear range of the analysis is 100 ng/L which may be extended with dilution.

Method 1631 for tissue (digestion II as outlined in Appendix to 1631)

This procedure is preferred for matrices containing organic materials, such as sludge and plant and animal tissues. An aliquot of sample is digested with HNO_3/H_2SO_4. The digestate is diluted with BrCl solution to destroy any remaining organic material. The reporting limit is 0.001 mg/Kg assuming 0.5g sample and 100% solids.

Method 1631 for sediment and soil (digestion I as outlined in Appendix to 1631)

This procedure is preferred for geological materials and is capable of rapid and compete dissolution of inorganic mercury. A sample aliquot is digested with HCl/HNO3 followed by addition of BrCl to destroy any organic material. The reporting limit is 0.001 mg/Kg assuming 0.5g sample and 100% solids.

Parameter	CAS
Mercury (Hg)	7439-97-6

2. Summary of Method

Mercury is determined by cold vapor atomic fluorescence spectrometry following digestion with an acidic bromine chloride solution. Bromine chloride is prepared by mixing potassium bromide with potassium bromate solutions with hydrochloric acid producing a yellow solution. Bromine chloride is known to oxidize inorganic and organomercury compounds.

If a yellow color does not persist after addition of bromine chloride then additional solution must be added until a yellow color persists. The same volume of bromine chloride solution is added to all samples, the method blank, LCS and standards. After digestion is complete, 12% hydroxylamine hydrochloride solution may be added (not required) to the sample, the sample shaken and allowed to stand for 5 minutes. As samples are diluted 1:10 at the instrument, there is adequate stannous chloride to reduce both the BrCl and mercury to Hg^0.

Samples and standards are placed in the autosampler rack and analyzed by cold vapor atomic fluorescence spectrometry at a wavelength of 253.7 nm.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2.1 Method Modifications from Reference

New perfluoropolymer lined 250 mL bottles are used for aqueous sample collection for Method 1631E. These bottles are rinsed two times with Type I water, filled with DI water and 1 mL 0.2N BrCl and leached overnight. The bottles are rinsed two more times and placed in a new plastic bag for use. This cleaning procedure has been determined to be adequate through analysis of bottles blanks prepared for at least each lot of bottles.

3. Reporting Limits

The mercury aqueous is 0.0005 μg/L Method 1631.

The soil/sediment/tissue RL is 0.001 mg/Kg.

4. Interferences

4.1 Gold, silver and iodide are known interferences. An iodide test is performed on all samples suspected or known to contain iodide. Samples positive for iodide are pre-reduced with stannous chloride to clarify the brown color, analysis should proceed immediately.

4.1.1 Place about 15 to 20 drops of the sample solution into cup and add 8 to 10 drops of the 1 mol/L silver nitrate solution. The formation of a yellow precipitate represents a positive test for iodide.

4.1.2 Prepare a starch solution by dissolving 1g of soluble starch in 100mL of distilled water.

4.1.3 Place about 20 drops of the sample solution in a test tube and add 1 to 2 drops of sodium hypochlorite (household bleach). The sample should turn brownish-red if iodide is present.

4.1.4 Shake the test tube and add 4 or 5 drops of starch solution. A dark blue coloration upon addition of the starch confirms the presence of iodide.

It may be necessary to clean the analytical system with 4N HCl following analysis of a sample with high iodide concentrations (typically greater than 30 mg/L).

4.2 Sulfide concentrations up to 24 mg/L produce no interference problems.

4.3 High concentrations of gold and silver in a sample may suppress mercury reduction.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. Eating, drinking, smoking, or the application of cosmetics is not permitted in the laboratory area. Horseplay of any kind is prohibited. Pipetting by mouth is not permitted. All Personal Protective Equipment (PPE) must be removed before leaving the laboratory area and before entering the employee lounge or eating area. Always wash your hands before leaving the laboratory. All relevant Material Safety Data Sheets (MSDSs) are kept alphabetically in the centrally located file storage, in the common area outside of the Information Technology (IT) offices.

Approved PPE, which includes Safety Glasses, Gloves and Lab Coats, must be worn at ***all*** times when handling samples, reagents, chemicals, or when in the vicinity of others handling these items, so that dermal contact is avoided. All standards, reagents and solvents shall be handled under a hood using the proper PPE. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, use a secure transporting devise and/or secondary outer container. Chemical storage is properly segregated and adequately ventilated to reduce the possibility of hazardous reactions. Chemical storage in work areas shall be kept to a minimum. Storage on bench tops or other work surfaces, except temporary, is not permitted.

Spilled samples, solvents, reagents, and water must be cleaned up from bench tops, instruments and autosampler surfaces immediately. A spill is considered a quantity of hazardous material if it is two times greater than the normal working volume. Concentrated solvents, acids or bases present a moderate to extreme hazard to the skin and mucous membranes. If contact with the skin occurs, immediately flush with large volumes of water. In the case of acidic/basic spills, the *Spill Kit* located in each laboratory shall be utilized before attempting to cleanup the spill. Although procedures are

designed to minimize the possibility of an accident, all injuries or accidents, regardless of the nature or severity, are to be reported to the Department Manager Supervisor immediately. If an employee discovers a potentially unsafe condition, this must be reported to the Department Manager Supervisor immediately. No employee should feel compelled to work in a situation where they do not feel entirely informed, trained, or safe.

Analytical instrumentation poses the unique possibility of exposure to high voltages. Other than the *routine* instrument maintenance, as listed in the front of every Instrument Maintenance Logbook, at no time shall an instrument operator attempt to maintenance an instrument alone, or without the proper training, supervision or instruction. Caution must always be used in the presence of moving parts (autosamplers) and hot surfaces (injection ports).

Compressed gas cylinders shall only be moved with the dolly supplied for this specific purpose. The cap must be on the cylinder while it is being moved. The tank must be secured when in its final position. All spent tanks are to be returned in the same manner, and secured until removed by the vendor. Liquid argon or nitrogen represents a potential cryogenic hazard and safe-handling procedures must be used at all times.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples collected for Methods 1631 are collected in pre-cleaned fluoropolymer bottles with tightly fitting fluoropolymer caps. Atmospheric mercury is known to diffuse through polyethylene bottles at levels below 0.05 µg/L.

Fluoropolymer bottles supplied by the laboratory for sample collection by Methods 1631 must be tested prior to sampling. 5% of a new lot of bottles are tested by adding DI water and 0.25 mL of BrCl and letting stand overnight. Mercury concentrations in the bottles must be less than 0.5 ng/L.

Sediments and tissues are collected in amber glass jars.

6.2 Sample Preservation

Samples collected for Methods 1631 may be preserved with 5 mL/L of 12 N HCl or 5 mL/L of BrCl solution (Section 8.8) if only total Hg is being analyzed. Sample preservation at the laboratory is recorded in the Sample Preservation/Filtration Log Book in the Metals Laboratory.

Samples collected and preserved under these conditions are stable for 90 days for Method 1631E.

6.3 Sample Shipping

No special sample shipping requirements. Typical shipping procedures may be found in the Alpha Sample Receipt & Login SOP (08-01).

6.4 Sample Handling

Samples may be stored at room temperature in the laboratory. It is suggested that the sample containers for samples being analyzed by Methods 1631 be stored in new polyethylene bags until analysis to prevent possible contamination.

7. Equipment and Supplies

7.1 Instrument: CETAC M-8000 equipped with a PC loaded with instrumental software,. The instrument is connected to a source of high-purity, mercury free argon regulated to 15 PSI.

7.2 Santoprene Peristaltic Pump Tubing: Various tubing sizes for reagent pump and autosampler rinse pump.

7.3 Glassware for Digestion: Pre-cleaned 50mL disposable polyethylene screw cap digestion tubes.

7.4 Other: Adjustable Eppendorf pipettes and replacement tips.

8. Reagents and Standards

ACS Trace Metal grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

Solutions below expire yearly unless noted and are stored at room temperature.

8.1 Deionized (DI) water: The Barnstead NANO-pure system provides Type I water used in the preparation of samples and standards.

8.2 Hydrochloric acid, concentrated (HCl): ACS reagent grade, Fisher #A508-212, or equivalent. Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.3 Nitric acid, concentrated (HNO_3): ACS reagent grade, Fisher #A509-212, or equivalent. Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.3.1 2% HCl/2%HNO_3: Add 20mL concentrated HCl and 20mL concentrated HNO_3 to approximately 500mL DI and bring to 1L with DI. Larger volumes are prepared in a similar manner.

8.3.2 10% HNO_3: Add 100 concentrated HNO_3 to approximately 500mL DI and bring to 1L with DI. Larger volumes are prepared in a similar manner.

8.4 Hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$): ACS reagent grade, VWR #HX0770-1, or equivalent. Reagent expires 4 years from date received.

8.4.1 12% Hydroxylamine Hydrochloride solution: Add 12g Hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$) per 100 mL of DI. Prepare in 125 mL fluorocarbon lined bottle. Prepare every 6 months or as needed.

8.5 Stannous Chloride ($SnCl_2{\cdot}H_2O$): ACS reagent grade, Fisher #T142-500, or equivalent. Reagent expires 4 years from date received.

8.5.1 10% Stannous Chloride/7% HCl Solution: Add 100g $SnCl_2$ and 70 mL concentrated HCl to 500mL DI, dilute to 1 L with DI. Solution is stable for 3 days. Solution is purged with argon at 2L/minute for 30 minutes to remove Hg impurities if necessary.

8.6 Potassium Bromate ($KBrO_3$): ACS grade, Fisher #P207-250. Bake in muffle furnace at 250°C for 8 hours to remove mercury impurities if necessary. Reagent expires 4 years from date received.

8.7 Potassium Bromide (KBr): ACS grade, Fisher #P205-500. Bake in muffle furnace at 250°C for 8 hours to remove mercury impurities if necessary. Reagent expires 5 years from date received.

8.8 Bromine Chloride (0.2N BrCl) Solution: Prepare in BOD bottle or fluoropolymer bottle under fume hood by adding 2.7g KBr and 3.8g $KBrO_3$ to 250mL concentrated HCl and mix. Solution will turn yellow. **Free Halogens are generated from this solution. Seal bottle when taking out of fume hood.** Prepare every 6 months or as needed.

8.9 0.07N Bromine Chloride (BrCl) Solution: Prepare in BOD bottle or fluoropolymer bottle under fume hood by diluting 300 mL of 0.2N BrCl to 1000mL DI. **Seal bottle when taking out of fume hood.** Prepare every 6 months or as needed.

8.10 0.02N Bromine Chloride (BrCl) Solution: Prepare in BOD bottle or PEF bottle under fume hood by diluting 100 mL of 0.2N BrCl to 1000mL DI. **Seal bottle when taking out of fume hood.** Prepare every 6 months or as needed.

8.11 HNO_3/H_2SO_4 Solution: In a hood, slowly add 300 mL of concentrated H2SO4 to 700 mL of concentrated HNO3 in a fluoropolymer bottle. Prepare every 6 months or as needed.

8.12 Stock and Working Standards: Store standards at room temperature out of direct light. All stock and working calibration standards expiration dates are based on manufacturer expiration date or one year from date received.

8.12.1 1000 mg/L Stock Calibration Mercury Standard: Ultra Scientific #ICP-080, or equivalent.

8.12.1.1 1000µg/L Working Standard: Prepare by adding 0.1 mL of 1000 mg/L stock to a 100 mL volumetric flask containing 2% HCl. Bring to volume with DI water and pour into brown bottle. This standard is identified as **C-date-V** in the Mercury Standards Preparation Log.

8.12.1.2 100 µg/L Intermediate Standard: Prepare by adding 1 mL of the 1000 µg/L working standard to 15 mL graduated centrifuge tube containing 2% HCl. Bring to 10 ml final volume with DI water. This intermediate standard is used for calibration standards preparation and then discarded.

8.12.1.3 5 µg/L Working Standard: Prepare by adding 0.25 mL of 1000 µg/L working standard to a 50 mL graduated screw cap centrifuge tube containing 2% HCl. Bring to 50 mL final volume with DI water. This standard is identified as **C-date-VS** in the Mercury Standards Preparation Log.

8.12.2 1000 mg/L Stock ICV Mercury Standard: Inorganic Ventures #CGHG-1, or equivalent.

8.12.2.1 1000 µg/L ICV Working Standard: Prepare by adding 0.1 mL of 1000 mg/L ICV stock to a 100 mL volumetric flask containing 2% HCl. Bring to volume with DI water and pour into brown bottle. This standard is identified as **I-date-V** in the Mercury Standards Preparation Log.

8.12.2.2 5 µg/L ICV Working Standard: Prepare by adding 0.25 mL of 1000 µg/L working standard to a 50 mL graduated screw cap centrifuge tube containing 2% HCl. Bring to 50 mL final volume with DI water. This standard is identified as **I-date-VS** in the Mercury Standards Preparation Log.

8.13 Calibration and Calibration Check Standards: Store standards at room temperature out of direct light.

Calibration and calibration check standards are prepared in 50 ml polypropylene (or equivalent) screw cap digestion tubes containing about 50 mL of DI water and 0.5 mL of BrCl(Section 8.8) solution. The STD0 consists of 50mL DI and 0.5mL BrCl solution.

After addition of the working standard, calibration standards are brought to 50mL final volume with DI water and optionally decolorized with 0.1 mL of hydroxylamine hydrochloride solution (Section 8.4.1). If sample preparation required additional quantities of BrCl to maintain a yellow color, then the same amount of BrCl is added to calibration and calibration check standards.

8.13.1 Standards Preparation for Method 1631

Standard ID	Vol. 5 µg/L Standard (Section 8.12.1.3) (mL)	Vol. STD5 (mL)	Final Concentration (µg/L)
STD0	-	-	0.0
STD1	-	0.25	0.0005
STD2	0.05	-	0.005
STD3	0.25	-	0.025
STD4	0.5	-	0.050
STD5	1.0	-	0.100
ICV,CCV,MS Section 8.12.2.2)	0.05	-	0.005

8.14 Argon Gas: Ultra-high purity

8.15 1M Silver Nitrate solution: purchased

8.16 Soluble starch

8.17 Chlorine Bleach: Clorox brand or equivalent (6% sodium hypochlorite)

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blanks

9.1.1 Method Blank

A method blank must be prepared once per every 20 samples or per digestion batch, whichever is more frequent. Prepare with same reagents as in the sample digests where applicable.

Mercury must not be detectable in the method blank at a concentration greater than the reporting limit.

For samples analyzed by Method 1631E, The mercury concentration in the Method Blank must be less than 0.5 ng/L or not to exceed one third of the regulatory limit if the data are to be used for regulatory purposes.

Corrective Action: May reanalyze once to determine if analytical error has occurred. Digestion of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control method blank are non-detect for the elements of interest, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high method blank and the client is notified in a project narrative associated with the sample results.

9.1.2 Initial Calibration Blank (ICB)

An initial calibration blank must be analyzed immediately following the ICV(See 9.4).

An initial calibration blank may be analyzed immediately following calibration for Method 1631E.

Mercury must not be detectable in the ICB at a concentration greater than the reporting limit.

Corrective Action: May reanalyze once to determine if analytical error has occurred. If the concentration still exceeds the acceptance limit, stop and recalibrate the instrument. Samples cannot be analyzed until an acceptable ICB analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control blank are non-detect for mercury, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high blank and the client is notified in a project narrative associated with the sample results.

Note: The ICB is equivalent to a Reagent Blank for Method 1631E. The mercury concentration in the Reagent Blank is not to exceed 0.2 ng/L or one third of the regulatory limit.

9.1.3 Continuing Calibration Blank

A continuing calibration blank must be analyzed immediately following the CCV (see 9.5).

Mercury must not be detectable in the CCB at a concentration greater than the reporting limit.

Corrective Action: May reanalyze once to determine if analytical error has occurred. If the concentration still exceeds the acceptance limit, stop, recalibrate the instrument and reanalyze all associated samples. Samples cannot be analyzed until an acceptable CCB analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control blank are non-detect for the elements of interest, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high

method blank and the client is notified in a project narrative associated with the sample results.

Note: The CCB is equivalent to a Method Blank for Method 1631E. Three Method Blanks must be analyzed with each batch.

9.2 Initial Precision and Recovery (IPR) for Soil/Sediment and Tissue

IPR is a one time initial demonstration of performance. Four aliquots of the appropriate reference matrix(Teflon chips for soil/sediment and chicken breast for tissue) spiked with 4.0 ng of Hg. The average % recovery must be 75-125% and the RSD between the replicates <20%.

9.3 LCS/Ongoing Precision and Recovery (OPR) for Soil/Sediment and Tissue

OPR/LCS is a test of the analytical system including prep. Analyze one aliquot of appropriate reference matrix spiked with 4.0 ng of Hg. Recovery must be 70-130%.

LCS/OPR for aqueous samples, recovery must be 77-123%

Corrective Action: Analysis according to the method may be repeated once to see if an analytical error has occurred. If the concentration still exceeds the acceptance limit, stop and recalibrate the instrument. Samples cannot be analyzed until an acceptable OPR analysis is obtained.

9.4 Initial Calibration Verification (ICV)

An initial calibration verification standard from a source different from that used to calibrate the instrument must be analyzed immediately following calibration as an independent check of instrument performance. Nominal acceptance limits are 85% - 115% of the true value.

Corrective Action: Analysis according to the method may be repeated once to see if an analytical error has occurred. If the concentration still exceeds the acceptance limit, stop and recalibrate the instrument. Samples cannot be analyzed until an acceptable ICV analysis is obtained.

9.5 Continuing Calibration Verification (CCV)

A continuing calibration verification standard must be analyzed after analysis of no more than 10 samples as an on-going check of instrument performance. Nominal acceptance limits are 85% - 115% of the true value.

Corrective Action: Analysis according to the method may be repeated once to see if an analytical error has occurred. If the concentration still exceeds the acceptance limit, stop, recalibrate the instrument and reanalyze all associated samples. Results cannot be accepted until an acceptable CCV analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with a high recovery CCV are non-detect. In such cases, the sample results are accepted without corrective action for the high CCV recovery and the client is notified in a project narrative associated with the sample results.

9.6 Matrix Spike/Matrix Spike Duplicate

An MS/MSD pair must be analyzed at a frequency of 1 per 10 samples. Spike samples at level in IPR/OPR (4.0 ng Hg for sediment/tissue, 0.005ug/L for aqueous). MS/MSD recovery criteria

70-130% and RPD <30% for sediment/tissue, 71-125% and RPD 24% for aqueous samples.

Corrective Action: For sediment/tissue, if MS/MSD fail and sample concentration is > ½ the spike concentration, the MS/MSD must be spiked at 2-5X the sample concentration and reanalyzed.

If the %RPD between the MS and MSD is not acceptable, a narrative is submitted with the data for inclusion on the final report.

9.7 Laboratory Duplicate

Duplicate analyses (matrix or sample duplicate) must be performed once per 20 samples 5% frequency), or per digestion batch, whichever is more frequent.

Acceptable relative percent differences (RPD) of duplicates is ≤20%. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate the RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:

R1 = sample Replicate #1
R2 = sample Replicate #2

Corrective Action: Include a project narrative.

9.8 Standard Reference Materials

Digest and analyze an appropriate reference material for the matrix, (animal tissue/plant tissue/soil/sediment). Recommended SRMs:

Soil/solid – ERA D076 (Metals in Soil) or equivalent

Sediment – NIST 2702 (Marine Sediment)

Plant tissue – NIST 1515 (Apple Leaves)

Animal tissue – NIST 2976 (Mussel tissue) or Tort-2 (Lobster tissue)

One SRM per batch must be analyzed and pass acceptance criteria

Corrective Action: Analysis according to the method may be repeated once to see if an analytical error has occurred. If the concentration still exceeds the acceptance limit, stop, recalibrate the instrument and reanalyze all associated samples. Results cannot be accepted until an acceptable SRM analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with a high recovery SRM are non-detect. In such cases, the sample results are accepted without corrective action for the high SRM recovery and the client is notified in a project narrative associated with the sample results.

9.9 Method-specific Quality Control Samples

Method 1631 requires that three calibration or system blanks be analyzed with the mean blank concentration used to blank correct all Data.

The average blank must be less than one third of the regulatory limit or 0.5 ng/L with the RSD less than <0.1 ng/L if the data is to be used for regulatory purposes.

Method 1631 requires that the recovery of mercury from the low calibration standard be within the range of 75% -125%.

The RSD of the mean calibration factor must be less than or equal to 15%.

9.10 Method Sequence

- Initial Calibration
- ICV
- ICB
- Method Blank
- IPR (LCS)
- SRM (sediment/tissue)
- Matrix Sample 1
- Matrix Duplicate
- Matrix Spike
- Matrix Spike Duplicate
- Samples 2-4
- CCV
- CCB
- Samples 5-14
- CCV
- CCB

10. Procedure

10.1 Equipment Set-up

Samples are prioritized by the Metals Department Manager or Preparation Group Leader for digestion based on hold time and client due date. The analyst must be familiar with instrument software and hardware as described in the user manuals supplied with the instrument, and have a completed IDC on file (Section 13.2) prior to any sample analysis.

10.1.1 Digestion I (Tissue/Sludge)

To a 50mL digestion tube, weigh 0.5 to 1.5 grams of material. Use less material if high organic content is expected.

Spike the OPR and MS/MSD samples with 0.08 mL of 5 ug/L intermediate (Section 8.12.2.2)

To each sample, add 10.0 mL of HNO3/H2SO4 (Section 8.11). Loosely cap with a watch glass. Carbohydrate based samples may react violently, allow these type of samples to react at room temperature for 4 hours prior to heating.

Place samples into digestion block. Monitor samples and remove from heat if excessive foaming occurs. Reflux for 2-3 hours.

After digestion, dilute samples with 0.02N BrCl (Section 8.10) and mix thoroughly. Allow samples to oxidize organic mercury for 4 hours prior to analysis.

Diluted digestates may be stored up to one year in glass or fluoropolymer containers.

10.1.2 Digestion II (Soil/Sediment)

To a 50mL digestion tube, weigh 0.5-1.5 grams of sample. Samples may be sieved if necessary to assure homogeneity.

Spike the OPR and MS/MSD samples with 0.08 mL of 5 ug/L intermediate (Section 8.12.2.2).

In a hood, add 8.0 mL concentrated HCl, then add 2.0 mL concentrated HNO_3. Allow to digest at room temperature for at least 4 hours, preferably overnight.

Dilute the digestate to 50 mL with 0.07N BrCl and shake thoroughly. Samples may be filtered through a 0.45µm filter or centrifuged for 20 minutes at 3000 RPM.

Diluted digestates may be stored up to one year in glass or fluoropolymer containers.

10.1.3 Aqueous Samples

To a 50mL digestion tube, add 50mL of sample and 0.25 mL of BrCl solution (Section 8.8). The sample should remain a yellow color. If not, add more BrCl solution to all samples, the Method Blank, LCS and all samples until yellow color persists.

Spike the MS/MSD and LCS with 0.05 L of 5ug/L Working Standard (Section 8.12.2.2).

Let stand for 12 hours and decolorize(optional) with 0.05mL of 12% hydroxylamine solution (Section 8.4.1).

10.2 Initial Calibration

The instrument is calibrated prior to sample analysis and at least once every 24 hours. Refer to Section 8.13 for the preparation of calibration standards for each determinative method.

For Method 1631, all standards and sample signals are blank corrected using the mean of the three calibration blanks analyzed at the beginning of the run. The instrument software calculates a mean calibration factor, calibration factor RSD and a recovery of the low calibration standard. The RSD of the mean calibration factor must be ≤ 15% and the recovery of the low calibration standard must be within the 75% - 125% range.

10.3 Equipment Operation and Sample Processing

Power is turned on to the instrument, autosampler, PC and argon gas supply. Peristaltic pump tubing is inspected for wear and replaced if necessary.

10.3.1 STARTUP

Instrument startup (cold start).

Power up M-8000 and autosampler and open the QuickTrace software. If system is to be used for analysis in the ppb range a minimum of 15 minutes warm-up time is required. If system is to be run in the ppt range a minimum of 30 minutes warm-up time is required.

Instrument startup (warm start). *The instrument and auto-sampler are on and stable. It is ok to leave the units powered up with the lamp and gas off.*
Place the auto-sampler rinse tubing into the rinse bottle(2%HCl/2%HNO_3)(Section 8.3.2). Turn on the lamp and carrier gas. A minimum 15-minute warm-up time is required.

Verify that the sample capillary (inlet insert) is 0.5mm above the Gas/liquid separator (GLS) center post.

Open vents on waste container.

Inspect peristaltic pump tubing for flat spots replace if necessary. Place the peristaltic pump tubing in their appropriate shoes and bridge stops. Do not lock shoe clamps at this time.

Open instrument controls and start auto-sampler rinse pump if not already operating (click pump on and probe down).

Place the SnCl2 capillary in the 10%$SnCl_3$/7% HCl (Section 8.5.1). and start the peristaltic pump at desired pump speed, which will usually match the method speed.

Lock down the peristaltic shoe clamps.

Inspect liquid flows. The GLS drain should be flowing smoothly with no build up or pulsing of liquid. The waste line from the peristaltic pump to the waste container should be liquid/gas etc… with no vibration. If this is not the case upon inspection, stop immediately and change GLS drain line and or waste line. The autosampler rinse station should have a convex liquid bubble adhering to the sample probe. If this is not the case, check that the rinse reservoir supply tubes are immersed in the rinse. If they are then check the rinse pump and pump tubing. Replace the tubing if necessary.

Wet the GLS center post. In instrument controls set the GLS gas flow to high and pump speed to 100%, pinch the drain line prior to the tee on the peristaltic pump drain tubing inlet side, let two or three bubbles go to the top of the GLS center post. Release the drain line and allow the liquid level to restore itself to normal operating levels before continuing to the next step. If the liquid does not bubble and the over flow sensor activates then clear the overflow and retry.

Connect the GLS exhaust tube (Nafion) or soda lime tubing to the GLS.

Place reagent capillaries in the reagent bottles.

Open the appropriate worksheet and verify that the gas flow and pump speed in the worksheet matches what is listed in >instrument>M-8000 controls, if the flow and speed are not the same make the necessary change in instrument controls or click the auto set icon on the menu bar. This will stabilize the instrument before auto zeroing and running a peak profile.

Zero the M-8000 using the auto zero.

If major maintenance was performed since the last start up perform the following check: Peak profile a standard with a minimum peak area response of 200K and verify the baseline is stable and the sample peak falls within the method integration times. Perform the necessary maintenance to meet this before proceeding with calibration and analysis.

Initiate sample analysis by clicking on the start button on the analysis page. If a sample concentration is greater than the highest standard concentration of the calibration curve, dilute the digestate and reanalyze.

10.3.2 SHUTTING DOWN:

Place the SnCl2 capillary in a beaker of 10% HNO3 and cap the SnCl2 bottle.

Rinse the system for a minimum of ten minutes with acid flowing.

Place the reagent capillaries in a beaker of DI water and rinse the system for one minute.

Raise sample probe via >instrument>auto-sampler controls (click probe up and pump off).

Remove reagent capillaries from DI water.

Allow the drain and waste lines to run completely dry.

Turn off peristaltic pump via >instrument>M-8000>pump off.

Release peristaltic shoe clamps and release the pump tubing from their bridge stops, release and relax peristaltic pump tubing.

Close vents on waste container.

Disconnect the GLS exhaust line (Nafion) or soda lime tubing from the GLS.

Turn off gas and lamp >instrument>M-8000>controls (note: you may want to turn off main supply gas at the Dewar or cylinder source).

If you are going to use the instrument the next day or in the near future, leave the software on the page >instrument>M-8000 controls>auto-sampler. It will then be ready for a warm start.

If you are not going to be using the instrument in the near future then exit the QuickTrace software and turn off the autodiluter, autosampler and M-8000 and pump.

The analysis method is selected and the autosampler template is selected. Once sample ID's have been typed in, the file is saved as the LIMS workgroup number.

Following analysis, the data file is exported to the LIMS on the O:Metals\CETAC drive for data processing.

10.4 Continuing Calibration

Periodically and at the end of the analysis, CCV standards (Section 9.5) and CCB standards (Section 9.1.3) are analyzed.

10.5 Preventive Maintenance

Inspect pump tubing for signs of wear and replace (recommended at least monthly). Clean gas/liquid separator as needed. Change dryer tube annually.

11. Data Evaluation, Calculations and Reporting

All results for the metals elements of interests are reportable without qualification if digestion and analytical holding times are met, preservation (including cooler temperatures) are met, all QC criteria defined in the table below are met, and matrix interference is not suspected during digestion or analysis of the samples

Sample concentrations for Method 1631 using the CETAC M-8000 are calculated based on peak area.

Sample concentrations are calculated as follows:

Sample Concentration = $((A_s - A_{mcb}) * (V_f/V_i) * DF)/CFm$

Where:

A_s = sample peak height or area
A_{mcb} = Mean calibration blank peak height or area
V_f = Final volume from preparation
V_i = Initial volume from preparation
DF = dilution factor
CFm = mean calibration factor for the calibration curve

Note: The final volume from the sample preparation would reflect any changes in volume due to BrCl added to sample.

QC Parameter	Acceptance Criteria
Method Blank	< reporting limit
OPR/LCS (sediment/tissue)	70-130% Recovery
OPR/LCS (aqueous)	77-123% Recovery
SRM (sediment/tissue)	% Recovery limits are specific to each SRM
Matrix Duplicate	≤ 20% RPD
Matrix Spike	70-130% Recovery(sediment/tissue) 71-125% Recovery (aqueous)
Matrix Spike Duplicate	≤ 30% RPD, 70-130% Recovery (sediment/tissue) ≤ 24% RPD, 71-125% Recovery (aqueous)

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9 outlines sample batch QC acceptance criteria. If non-compliant results are to be reported, the Metals Department Manager and/or the Laboratory Director must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client for approval, or other corrective action, such as re-sampling and re-analysis. The instrument analyst or Department Manager performing the secondary analytical review initiates a project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

13.3 Instrument Detection Limits

The instrument detection limit (IDL) is the smallest signal above the background noise that an instrument can detect. The IDL can be calculated taking the average of the standard deviations for three measurements of a reagent blank solution analyzed on three analytical runs on three non-consecutive days. Seven consecutive measurements must be taken per

day. The IDL samples are not required to go through sample digestion. Each measurement must be performed as though it were a separate sample.

14. Pollution Prevention and Waste Management

The *Hazardous Waste and Sample Disposal* SOP (1797), must be referenced for disposal of used standards, solvents, acids, reagents or other chemicals.

Once sample batches have completed digestion, the sample containers are stored in the metals lab and held for 30 days. If there is no sample remaining in the sample collection bottle, it may be rinsed and thrown away.

Once the samples have been held for 30 days, any aqueous sample remaining must be disposed in a 55-gallon drum labeled “Corrosive Liquid”.

Once satisfactory inorganic element results have been generated, the digestates are held for 30 days, or longer if specified by a client contract, then discarded into a 55-gallon drum labeled “Corrosive Liquid”.

All reagent waste generated during digestion must be stored in satellite containers in the metals preparation laboratory.

Once the reagent waste satellite containers are full, they must be emptied into 55-gallon drums marked “Corrosive Liquid”.

Refer to Alpha’s Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP 1559 Sample Receipt & Log-In

SOP 1732 MDL/LOD/LOQ Generation

SOP 1739 IDC/DOC Generation

SOP 1797 Waste Management and Disposal SOP

16. Attachments

None.

Mercury Determination in Aqueous Samples by Cold Vapor Atomic Fluorescence Technique (CVAF)

References: **Method 7474,** Mercury in Sediment and Tissues by Atomic Flourescence Spectrometry. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 0, February 2007.

1. Scope and Application

Matrices: The method is applicable to total and dissolved mercury in aqueous samples.

Definitions: Refer to Alpha Analytical Quality Manual.

Three procedures for the analysis of mercury by cold vapor atomic fluorescence spectrometry are discussed in this SOP.

Lab Method 7474 (modified)

This method utilizes a bromine chloride digestion. Samples are then decolorized using hydroxylamine hydrochloride and analyzed following reduction with stannous chloride. Elemental mercury is then purged from solution and analyzed by atomic fluorescence spectrometry. The reporting limit is 0.1 µg/L and the linear range of the analysis is 5 µg/L.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Mercury Analyzer and in the interpretation of Mercury data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Parameter	CAS
Mercury (Hg)	7439-97-6

2. Summary of Method

Mercury in aqueous samples is determined by cold vapor atomic fluorescence spectrometry following digestion with an acidic bromine chloride solution. Bromine chloride is prepared by mixing potassium bromide with potassium bromate solutions with hydrochloric acid producing a yellow solution. Bromine chloride is known to oxidize inorganic and organomercury compounds. Samples are digested by adding 5 ml of bromine chloride per liter of sample. Samples are allowed to digest for a minimum of 15 minutes.

If a yellow color does not persist after addition of bromine chloride then additional solution must be added until a yellow color persists. The same volume of bromine chloride solution is added to all samples, the method blank, LCS and standards. After digestion is complete, 12% hydroxyl amine

hydrochloride solution is added to the sample, the sample shaken and allowed to stand for 5 minutes. Additional hydroxyl amine hydrochloride is added if the yellow color from the bromine chloride solution persists.

Following stannous chloride reduction, samples and standards are placed in the autosampler rack and analyzed by cold vapor atomic fluorescence spectrometry at a wavelength of 253.7 nm.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability, analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the Quality Assurance Officer and/or Laboratory Director on a case-by-case basis.

2.1 Method Modifications from Reference

Method 7474 is a test method for the analysis of mercury in Soil/Sediments and tissues. A reduced gain setting is used for decreased instrumental sensitivity for samples not requiring low level reporting limits (<0.1 µg/L).

The procedure used to prepare the bromine chloride solution has been modified from the method reference to that used in EPA method 1631E, 2002. There is no impact to the data as indicated by the performance of PT samples.

3. Reporting Limits

The mercury aqueous RL is 0.05 µg/L by Method 7474 Modified.

4. Interferences

4.1 Gold, silver and iodide are known interferences. Samples with iodide concentrations greater than 3 mg/L are pre-reduced with stannous chloride to clarify the brown color. It may be necessary to clean the analytical system with 4N HCl following analysis of a sample with iodide concentrations greater than 30 mg/L.

4.2 Sulfide concentrations up to 24 mg/L produce no interference problems.

4.3 High concentrations of gold and silver in a sample may suppress mercury reduction.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. Eating, drinking, smoking, or the application of cosmetics is not permitted in the laboratory area. Horseplay of any kind is prohibited. Pipetting by mouth is not permitted. All Personal Protective Equipment (PPE) must be removed before leaving the laboratory area and before entering the employee lounge or eating area. Always wash your hands before leaving the laboratory. All relevant Material Safety Data Sheets (MSDSs) are kept alphabetically in the centrally located file storage, in the common area outside of the Information Technology (IT) offices.

Approved PPE, which includes Safety Glasses, Gloves and Lab Coats, must be worn at ***all*** times when handling samples, reagents, chemicals, or when in the vicinity of others handling these items, so that dermal contact is avoided. All standards, reagents and solvents shall be handled under a hood using the proper PPE. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, use a secure transporting devise and/or secondary outer container. Chemical storage is properly segregated and adequately ventilated to reduce the possibility of hazardous reactions. Chemical storage in work areas shall be kept to a minimum. Storage on bench tops or other work surfaces, except temporary, is not permitted.

Spilled samples, solvents, reagents, and water must be cleaned up from bench tops, instruments and autosampler surfaces immediately. A spill is considered a quantity of hazardous material if it is two times greater than the normal working volume. Concentrated solvents, acids or bases present a moderate to extreme hazard to the skin and mucous membranes. If contact with the skin occurs, immediately flush with large volumes of water. In the case of acidic/basic spills, the *Spill Kit* located in each laboratory shall be utilized before attempting to cleanup the spill. Although procedures are designed to minimize the possibility of an accident, all injuries or accidents, regardless of the nature or severity, are to be reported to the Department Manager Supervisor immediately. If an employee discovers a potentially unsafe condition, this must be reported to the Department Manager Supervisor immediately. No employee should feel compelled to work in a situation where they do not feel entirely informed, trained, or safe.

Analytical instrumentation poses the unique possibility of exposure to high voltages. Other than the *routine* instrument maintenance, as listed in the front of every Instrument Maintenance Logbook, at no time shall an instrument operator attempt to maintenance an instrument alone, or without the proper training, supervision or instruction. Caution must always be used in the presence of moving parts (autosamplers) and hot surfaces (injection ports).

Compressed gas cylinders shall only be moved with the dolly supplied for this specific purpose. The cap must be on the cylinder while it is being moved. The tank must be secured when in its final position. All spent tanks are to be returned in the same manner, and secured until removed by the

vendor. Liquid argon or nitrogen represents a potential cryogenic hazard and safe-handling procedures must be used at all times.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples collected for Method 7474 Modified are collected in polyethylene bottles. Bottle size will vary with project requirements.

6.2 Sample Preservation

Aqueous samples collected for Method 7474 Modified are preserved at pH ≤ 2 with 2 mL/L of 1:1 HNO_3.

Samples collected and preserved under these conditions are stable for 28 days for Method 7474.

6.3 Sample Shipping

No special sample shipping requirements. Typical shipping procedures may be found in the Alpha Sample Receipt & Login SOP (1559).

6.4 Sample Handling

Samples may be stored at room temperature in the laboratory.

7. Equipment and Supplies

7.1 Instrument: The PSA Millennium Merlin Atomic Fluorescence Analyzer equipped with a PC loaded with instrumental software, and PSA 20.400 autosampler. The instrument is connected to a source of high-purity, mercury free argon regulated to 50 PSI.

7.2 Santoprene Peristaltic Pump Tubing: Green/Green (PSA part number M025T002 or equivalent) for sample, blank and autosampler rinse, Grey/Grey (PSA part number M055T005 or equivalent) for stannous chloride.

7.3 Glassware for Digestion: Pre-cleaned 50mL disposable polyethylene screw cap digestion tubes. Rinse two times with DI water before use.

7.4 Other: Adjustable Eppendorf pipettes and replacement tips.

8. Reagents and Standards

ACS Trace Metal grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening

the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

Solutions below expire yearly unless noted and are stored at room temperature.

8.1 Deionized (DI) water: The Barnstead NANO-pure system provides Type I water used in the preparation of samples and standards.

8.2 Hydrochloric acid, concentrated (HCl): ACS reagent grade.

8.2.1 10% HCl: Add 100mL concentrated HCl to approximately 500mL DI and bring to 1L with DI. Larger volumes are prepared in a similar manner.

8.3 Nitric acid, concentrated (HNO_3): ACS reagent grade.

8.4 Hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$): ACS reagent grade. Reagent expires 4 years from date received.

8.4.1 12% Hydroxylamine Hydrochloride solution: Add 12g Hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$) per 100 mL of DI. Prepare in 125 mL fluorocarbon lined bottle.

8.5 Stannous Chloride ($SnCl_2{\cdot}H_20$): ACS reagent grade. Reagent expires 4 years from date received.

8.5.1 2% Stannous Chloride Solution: Add 20g to 1L of 10% HCl. Prepare daily. Solution is purged with argon at 2L/minute for 30 minutes to remove Hg impurities if necessary.

8.6 Potassium Bromate ($KBrO_3$): ACS grade. Bake in muffle furnace at 150°C for 8 hours to remove mercury impurities if necessary. Reagent expires 4 years from date received.

8.7 Potassium Bromide (KBr): ACS grade. Bake in muffle furnace at 150°C for 8 hours to remove mercury impurities if necessary. Reagent expires 5 years from date received.

8.8 Bromine Chloride (BrCl) Solution: Prepare in BOD bottle under fume hood by adding 2.7g KBr and 3.8g $KBrO_3$ to 250mL of concentrated HCl and mix. Solution will turn yellow. **Free Halogens are generated from this solution. Seal bottle when taking out of fume hood.** Prepare every 6 months or as needed.

8.9 Stock and Working Standards: Store standards at room temperature out of direct light. All stock and working calibration standards expiration dates are based on manufacturer expiration date or one year from date received.

8.9.1 1000 mg/L Stock Calibration Mercury Standard: Ultra Scientific #ICP-080, or equivalent.

8.9.1.1 1000µg/L Working Standard: Prepare by adding 0.1 mL of 1000 mg/L stock to a 100 mL volumetric flask containing 2% HCl. Bring to volume with DI water and pour into brown bottle. This standard is identified as **C-date-V** in the Mercury Standards Preparation Log.

8.9.1.2 100 µg/L Intermediate Standard: Prepare by adding 1 mL of the 1000 µg/L working standard to 15 mL graduated centrifuge tube containing 2% HCl. Bring to 10 ml final volume with DI water. This intermediate standard is used for calibration standards

preparation and then discarded.

8.9.1.3 5 µg/L Working Standard: Prepare by adding 0.25 mL of 1000 µg/L working standard to a 50 mL graduated screw cap centrifuge tube containing 2% HCl. Bring to 50 mL final volume with DI water. This standard is identified as **C-date-VS** in the Mercury Standards Preparation Log.

8.9.2 1000 mg/L Stock ICV Mercury Standard: Inorganic Ventures #CGHG-1, or equivalent.

8.9.2.1 1000 µg/L ICV Working Standard: Prepare by adding 0.1 mL of 1000 mg/L ICV stock to a 100 mL volumetric flask containing 2% HCl. Bring to volume with DI water and pour into brown bottle. This standard is identified as **I-date-V** in the Mercury Standards Preparation Log.

8.9.2.2 5 µg/L ICV Working Standard: Prepare by adding 0.25 mL of 1000 µg/L working standard to a 50 mL graduated screw cap centrifuge tube containing 2% HCl. Bring to 50 mL final volume with DI water. This standard is identified as **I-date-VS** in the Mercury Standards Preparation Log.

8.10 Calibration and Calibration Check Standards: Store standards at room temperature out of direct light.

Calibration and calibration check standards are prepared in 50 ml polypropylene (or equivalent) screw cap digestion tubes containing approximately 25 mL of DI water and 0.25 mL of BrCl solution. The STD0 consists of 50mL DI and 0.25mL BrCl solution.

After addition of the working standard, calibration standards are brought to 50mL final volume with DI water, wait 15 minutes and decolorized with 0.050 mL of hydroxylamine hydrochloride solution (Section 8.4). If sample preparation required additional quantities of BrCl to maintain a yellow color, then the same amount of BrCl is added to calibration and calibration check standards (Section 10.3).

8.10.1 Standards Preparation for Method 7474 Modified

Standard ID	Vol. 1000 µg/L Standard (Section 8.9.1.1) (mL)	Vol. 100 µg/L Standard (Section 8.9.1.2) (mL)	Final Concentration (µg/L)
STD0	-	-	0.0
STD1	-	0.025	0.05
STD2	-	0.05	0.1
STD3	-	0.25	0.5
STD4	0.1	-	2.0
STD5	0.25	-	5.0
ICV,CCV,MS **(Section 8.9.2.1)**	0.125	-	2.5

8.11 Argon Gas: Ultra-high purity

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blanks

9.1.1 Method Blank

A method blank must be prepared once per every 20 samples or per digestion batch, whichever is more frequent.

Mercury must not be detectable in the method blank at a concentration greater than the reporting limit.

Corrective Action: May reanalyze once to determine if analytical error has occurred. Digestion of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control method blank are non-detect for the elements of interest, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high method blank and the client is notified in a project narrative associated with the sample results.

9.1.2 Initial Calibration Blank (ICB)

An initial calibration blank must be analyzed immediately following the ICV (see 9.3).

Mercury must not be detectable in the ICB at a concentration greater than the reporting limit.

Corrective Action: May reanalyze once to determine if analytical error has occurred. If the concentration still exceeds the acceptance limit, stop and recalibrate the instrument. Samples cannot be analyzed until an acceptable ICB analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control blank are non-detect for mercury, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high blank and the client is notified in a project narrative associated with the sample results.

9.1.3 Continuing Calibration Blank

A continuing calibration blank must be analyzed immediately following the CCV (see 9.4).

Mercury must not be detectable in the CCB at a concentration greater than the reporting limit.

Corrective Action: May reanalyze once to determine if analytical error has occurred. If the concentration still exceeds the acceptance limit, stop, recalibrate the instrument and reanalyze all associated samples. Samples cannot be analyzed until an acceptable CCB analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control blank are non-detect for the elements

of interest, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high method blank and the client is notified in a project narrative associated with the sample results.

9.2 Laboratory Control Sample (LCS)

Laboratory control sample (LCS) must be prepared once per every 20 samples or per digestion batch, whichever is more frequent, and spiked with a solution prepared from a second source or lot number, other than the source used to verify the accuracy of the standard curve for the determinative analytical method. The LCS contains the target element, and is digested along with the samples as verification of the accuracy of the entire digestion procedure.

The LCS must be recovered within 80-120% of the true value.

Corrective Action: Analysis according to the method may be repeated once to see if an analytical error has occurred. If the LCS recovery is still out of control, re-prepare and re-analyze the LCS and all associated samples. Samples cannot be reported until an acceptable LCS is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control LCS are also associated with a matrix spike that is in control. This is an acceptable measure of accuracy of the digestion and analytical procedures. An explanation of the out of control LCS recovery must be included in the project narrative to the client and the sample data reported noting the acceptable MS results as batch QC.

9.3 Initial Calibration Verification (ICV)

An initial calibration verification standard from a source different from that used to calibrate the instrument (see 8.9.2) must be analyzed immediately following calibration as an independent check of instrument performance. Nominal acceptance limits are 90% - 110% for Method 7474.

Corrective Action: Analysis according to the method may be repeated once to see if an analytical error has occurred. If the concentration still exceeds the acceptance limit, stop and recalibrate the instrument. Samples cannot be analyzed until an acceptable ICV analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with a high recovery ICV are non-detect. In such cases, the sample results are accepted without corrective action for the high ICV recovery and the client is notified in a project narrative associated with the sample results.

9.4 Continuing Calibration Verification (CCV)

A continuing calibration verification standard must be analyzed after analysis of no more than 10 samples as an on-going check of instrument performance. Nominal acceptance limits are 90% - 110% for Method 7474.

Corrective Action: Analysis according to the method may be repeated once to see if an analytical error has occurred. If the concentration still exceeds the acceptance limit, stop, recalibrate the instrument and reanalyze all associated samples. Results cannot be accepted until an acceptable CCV analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with a high recovery CCV are non-detect. In such cases, the sample results are accepted without corrective

action for the high CCV recovery and the client is notified in a project narrative associated with the sample results.

9.5 Matrix Spike

A matrix spike (MS) sample must be performed once per 20 samples (5% frequency), or per digestion batch, whichever is more frequent. The MS contains the target element.

The MS must be recovered within 80%-120%.

A matrix spike duplicate (MSD) is performed upon client request and must meet the same recovery criteria as the MS. The %RPD between the MS and MSD must be ≤20%.

Corrective Action: Analysis according to the appropriate method may be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits and the LCS is compliant; include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy of the reported results as evidenced by MS recovery outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses (matrix or sample duplicate) must be performed once per 20 samples 5% frequency), or per digestion batch, whichever is more frequent.

Acceptable relative percent differences (RPD) of duplicates is ≤20%. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate the RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:

R1 = sample Replicate #1
R2 = sample Replicate #2

Corrective Action: Analysis may be repeated once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the reported metals results as evidenced by the matrix duplicate %RPD exceedance.

9.7 Method Sequence

- Initial Calibration
- ICV
- ICB
- Method Blank
- Laboratory Control Sample
- Matrix QC Sample

- Matrix Duplicate
- Matrix Spike
- Samples 2-5
- CCV
- CCB
- Samples 6-15
- CCV
- CCB

10. Procedure

10.1 Equipment Set-up

Samples are prioritized by the Metals Department Manager or Preparation Group Leader for digestion based on hold time and client due date. The analyst must be familiar with instrument software and hardware as described in the user manuals supplied with the instrument, and have a completed IDC on file (Section 13.2) prior to any sample analysis.

10.1.1 Method 7474 (Modified) Sample Preparation

To a 50mL digestion tube, add 50mL of sample and 0.25 mL of BrCl solution (Section 8.8). The sample should remain a yellow color. If not, add more BrCl solution to all samples, the Method Blank, LCS and all samples until yellow color persists.

Spike the MS and LCS with 0.125mL of 5ug/L Working Standard (Section 8.9.1.3).

Let stand for 15 minutes and decolorize with 0.05mL of 12% hydroxylamine solution (Section 8.4.1).

10.2 Initial Calibration

The instrument is calibrated prior to sample analysis and at least once every 24 hours. Refer to Section 8.10 for the preparation of calibration standards for each determinative method. The peak height mode is selected in the method for calibration purposes. Peak area mode is also an option in the method for determining sample concentration and may be used if the analyst determines that it is necessary.

A linear least squares regression calibration curve is selected for methods 7474.

In all cases, a correlation coefficient ≥ 0.995 is required before the analysis of samples can begin.

An ICV (see 9.3) and ICB (see 9.1.2) are analyzed after calibration

10.3 Equipment Operation and Sample Processing

Power is turned on to the instrument, autosampler, PC and argon gas supply. Peristaltic pump windings are inspected for wear and replaced if necessary. The winding clips are relaxed in between usage. The instrument manufacturer suggests that the pump tubing be changed once per week.

The stannous chloride solution (Section 8.5) is attached to the grey/grey pump tubing, 10% HCl (Section 8.2.1) is attached to the green/green autosampler rinse and DI is attached to the green/green rinse blank. The autosampler probe line is attached to a green/green pump tubing. The pumps are turned on in the software and flow checked to insure that solutions are delivered to the mixing valve.

After initializing the Millennium software, the instrument is turned on via the software and allowed to warm up for 30 minutes. The analysis method is selected and the autosampler template is selected. Methods 7474 is set up in the software which specifies delay, read and rinse times as well as pump speeds. Autosampler templates are set up for each method specifying calibration standard, QC standards and positions on the autosampler rack. Once sample ID's have been typed in, the file is saved as the ETR number.

A data file is opened and saved as "WGnumber".

Initiate sample analysis by clicking on the start button on the analysis page. If a sample concentration is greater than the highest standard concentration of the calibration curve, dilute the digestate with the same matrix as was used for the calibration blank, and reanalyze.

Following analysis, the data file is exported to the LIMS on the O:Metals\Millennium drive for data processing. The instrument and autosampler are then rinsed with DI water.

10.4 Continuing Calibration

Periodically and at the end of the analysis, CCV standards (see 9.4) and CCB standards (see 9.1.3) are analyzed.

10.5 Preventive Maintenance

Inspect pump windings for signs of wear and replace (recommended at least monthly). Clean gas/liquid separator periodically when appears coated with yellow film (rinse with 30% KOH solution and DI). Change dryer tube annually.

11. Data Evaluation, Calculations and Reporting

All results for the metals elements of interests are reportable without qualification if digestion and analytical holding times are met, preservation (including cooler temperatures) are met, all QC criteria defined in the table below are met, and matrix interference is not suspected during digestion or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

The instrument software calculates sample concentrations based on the signal peak height, the calibration curve. Sample concentrations for Method 7474 are calculated as follows:

Sample Concentration = $(A_s / Cf) * (V_f / V_i) * DF$

Where:

A_s = sample peak height or area
V_f = Final volume from preparation
V_i = Initial volume from preparation
DF = dilution factor
Cf = calibration factor for the calibration curve

Note: The final volume from the sample preparation would reflect any changes in volume due to BrCl added.

QC Parameter	Acceptance Criteria
Method Blank	< reporting limit
Laboratory Control Sample	80%-120% Recovery
Matrix Duplicate	≤ 20% RPD
Matrix Spike	75% - 125% Recovery 80%- 120% for Method 7474
Matrix Spike Duplicate (if needed)	≤ 20% RPD, 75% - 125% Recovery 80% - 120% for Method 7474

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9 outlines sample batch QC acceptance criteria. If non-compliant results are to be reported, the Metals Department Manager and/or the Laboratory Director must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client for approval, or other corrective action, such as re-sampling and re-analysis. The instrument analyst or Department Manager performing the secondary analytical review initiates a project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

13.3 Instrument Detection Limits

The instrument detection limit (IDL) is the smallest signal above the background noise that an instrument can detect. The IDL can be calculated taking the average of the standard deviations for three measurements of a reagent blank solution analyzed on three analytical

runs on three non-consecutive days. Seven consecutive measurements must be taken per day. The IDL samples are not required to go through sample digestion. Each measurement must be performed as though it were a separate sample.

14. Pollution Prevention and Waste Management

The *Hazardous Waste and Sample Disposal* SOP (1797), must be referenced for disposal of used standards, solvents, acids, reagents or other chemicals.

Once sample batches have completed digestion, the sample containers are stored in the metals lab and held for 30 days. If there is no sample remaining in the sample collection bottle, it may be rinsed and thrown away. It must be noted in the *Internal COC* that there is no sample remaining in the bottle, and the bottle was discarded.

Once the samples have been held for 30 days, any aqueous sample remaining must be disposed in a 55-gallon drum labeled “Corrosive Liquid”.

Once satisfactory inorganic element results have been generated, the digestates are held for 30 days, or longer if specified by a client contract, then discarded into a 55-gallon drum labeled “Corrosive Liquid”.

All reagent waste generated during digestion must be stored in satellite containers in the metals preparation laboratory.

Once the reagent waste satellite containers are full, they must be emptied into 55-gallon drums marked “Corrosive Liquid”.

Refer to Alpha’s Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

SOP # 2124 Chemical Hygiene Plan

SOP/1559 Sample Receipt & Log-In

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1797 Waste Management and Disposal SOP

16. Attachments

None.

Mercury in Liquid Waste

(Automated Cold-Vapor Technique)

Reference Method No.: **EPA 7470A**

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update II, September 1994.

1. Scope and Application

Matrices: Method 7470 is a cold-vapor atomic absorption procedure approved for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. (Method 7470 can also be used for analyzing certain solid and sludge-type wastes; however, Method 7471 is usually the method of choice for these waste types.) All samples must be subjected to an appropriate dissolution step prior to analysis.

Definitions: See Alpha Laboratories Quality Manual Appendix A.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Mercury Analyzer and in the interpretation of Mercury data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Prior to analysis, the liquid samples must be prepared according to the procedure discussed in this method.

Method 7470, a cold-vapor atomic absorption technique, is based on the absorption of radiation at 253.7-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.1 Method Modifications from Reference

2.1.1 A smaller sample sized is prepared, and therefore proportionately less reagent volumes are used.

2.1.2 The original method does not address the automated instrument procedure.

3. Reporting Limits

The typical reporting limit for Mercury is 0.0002mg/L. This satisfies Massachusetts, GW1 and GW 2 criteria. Connecticut mobility criteria for SPLP is 0.0004mg/L, Rhode Island is 0.002mg/L, and the Drinking Water reporting limit is 0.0002mg/L.

4. Interferences

Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253.7 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL). Both inorganic and organic mercury spikes have been quantitatively recovered from seawater by using this technique.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Analysis is conducted under a laboratory exhaust hood. The analyst must wear chemical resistant gloves when handling concentrated mercury standards.

The acidification of samples containing reactive materials may result in the release of toxic gases, such as cyanides or sulfides. Therefore, the acidification of samples is to be conducted under a laboratory exhaust hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in either glass or plastic containers.

6.2 Sample Preservation

Samples are preserved with HNO_3 to a pH of <2.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are stored under refrigeration at 4 ± 2°C and analyzed as soon as possible after collection. The samples have a 28-day holding time from the time of collection.

7. Equipment and Supplies

Instrumentation:

Perkin Elmer FIMS 100 Atomic absorption spectrophotometer: Use instrument settings recommended by the manufacturer. The PE FIMS is designed specifically for the measurement of mercury using the cold-vapor technique with BOC (background offset correction) performed by a survey scan prior to each sample introduction. PE S10 autosampler is coupled to the instrument.

Cetac M-6100 Atomic absorption spectrophotometer: Use instrument settings recommended by the manufacturer. This instrument employs a reference cell off-set correction and full automation through the CETAC software. A Cetac ASX-260 autosampler is coupled to the instrument.

Nippon Instrument model# RA-4300A analyzer with integrated 80 position autosampler:

The instrument adds a stannous chloride (II) solution to the sample post digestion, the divalent mercury ion (Hg2+) is reduced to zero-valent metallic mercury and turns into mercury gas by bubbling. Hg2++SnCl2 → Hg0 ↑
After removing the acid mist and water vapor generated by bubbling with an electronic cooling unit, the instrument measures the absorbance of mercury at 253.7 nm absorption wavelength. It measures the known mercury amount, creates a calibration curve, and then calculates the mercury amount from the absorbance.

7.1 Graduated cylinder: Rinse once with 50% HNO_3 and then rinse with reagent water prior to use.

7.2 Volumetric Flasks, Class A, various volumes: Rinse once with 50% HNO_3 and then rinse with reagent water prior to use.

7.3 Heating Block: Environmental Express HotBlock, 48 position capacity, able to maintain 95˚C +/-3.

7.4 50 mL Digestion Tubes: Polypropylene, graduated.

7.5 50 mL Digestion Tube Rack: 48 position, racklock

7.6 Pump tubing: Environmental Express, three stop and two in various IDs.

7.7 PTFE membranes: Whatman TE37 disks.

7.8 Dilution vials: 20mL capacity, used to prepare analytical dilutions.

7.9 Low Dust Laboratory Wipes

7.10 Compressed Air

7.11 Whatman 41 filter paper or equivalent

8. Reagents and Standards

8.1 Reagent Water: Reagent water is DI water shown to be interference free. All references to water in this method will refer to reagent water unless otherwise specified.

8.2 Sulfuric acid (H_2SO_4), concentrated: Reagent grade. Store at room temperature in an appropriately designated acid cabinet.

8.3 Hydrochloric acid, concentrated: Trace Metal grade. Store at room temperature in an appropriately designated acid cabinet.

8.4 Carrier, Hydrochloric Acid, 3%: This is the ***carrier*** for the Instrument. In a 1L volumetric flask, add 30mL concentrated trace grade HCl (Section 8.3). Bring to volume with reagent water. Store at room temperature, prepare daily as needed.

8.5 Reductant, Stannous Chloride in 3% HCl: This is the ***reductant*** for the Instrument. In a 1L volumetric flask, add 30mL concentrated trace grade HCl and 11g $SnCl_2 \cdot 2H_20$. Mix to dissolve the solid and bring to volume with reagent water. Store at room temperature, prepare daily as needed.

8.6 Nitric acid (HNO_3), concentrated: Trace metal grade of low mercury content. If a high reagent blank is obtained, it may be necessary to distill the nitric acid. Store at room temperature in an appropriately designated acid cabinet.

8.7 Sodium chloride-hydroxylamine hydrochloride solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride in reagent water and dilute to 100mL. Store at room temperature. Expires one month from date of preparation.

8.8 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 5 g of potassium permanganate in 100 mL of reagent water. Store at room temperature. Expires one month from date of preparation.

8.9 Potassium persulfate, 5% solution (w/v): Dissolve 5 g of potassium persulfate in 100 mL of reagent water. Store at room temperature. Expires one month from date of preparation.

8.10 Mercury Stock Standard, 1000ppm: Purchased from a commercial source with a certificate of analysis. Purchase three different sources. Store at room temperature. Expires upon manufacturer's specification.

8.11 Mercury Stock Calibration Standard, 10ppm: To a 100mL volumetric flask, add 5mL of concentrated HNO_3, 2.5mL of concentrated H_2SO_4 and 1000ppm Mercury Stock Standard (Section 8.10, use one source). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.

8.12 Mercury Working Calibration Standard / Matrix Spike Solution, 0.1ppm: To a 100mL volumetric flask, add 5mL of concentrated HNO_3, 2.5mL of concentrated H_2SO_4 and 1mL of 10ppm Mercury Stock Standard (Section 8.11). Bring to volume with reagent water. Store at room temperature. Make fresh daily.

8.13 Mercury Calibration Standards: All calibration standards are prepared daily.

8.13.1 0 ppm Calibration Standard: Add 10 mL of reagent water to a polypropylene digestion vessel. This aliquot may be used for the CCB. Another separate aliquot is prepared in the same manner for use as the ICB and diluent for sample dilutions.

8.13.2 **0.0002ppm Calibration Standard:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 0.05 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.

8.13.3 **0.001ppm Calibration Standard:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 0.25 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.

8.13.4 **0.002ppm Calibration Standard** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 0.5 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.

8.13.5 **0.005ppm Calibration Standard/Continuing Calibration Verification Standard:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 1.25 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.

8.13.6 **0.010ppm Calibration Standard / Post Analytical Spike Solution:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 2.5 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.

8.13.7 **0.020ppm Calibration Standard / Post Analytical Spike Solution:** Add 10 mL of reagent water to a polypropylene digestion vessel. Pipet 5.0 mL of 0.1ppm Mercury Working Stock (Section 8.12) to the digestion vessel. Bring to a final volume of 25 mL.

8.14 Mercury Stock LCS Standard, 10ppm: To a 100mL volumetric flask add 25mL of reagent water and 5mL of concentrated HNO_3 (Section 8.6). Add 1mL of 1000ppm Mercury Stock Standard (Section 8.10). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.

8.15 Mercury Working LCS Standard, 0.1ppm: To a 100mL volumetric flask add 25mL of reagent water and 5mL concentrated HNO_3 (Section 8.6). Add 1mL of 10ppm Stock LCS Standard (Section 8.14). Bring to volume with reagent water. Store at room temperature. Expires one week from date of preparation.

8.16 Mercury LCS Standard, 0.001ppm: Prepare daily with each batch of samples. To a 50mL digestion vessel add 10mL of reagent water Add 0.25 mL of 0.1ppm Working LCS Standard (Section 8.15). Bring to a final volume of 25mL and carry through entire digestion process as in Section 10.1.1.

8.17 Mercury Stock ICV Standard, 10ppm: To a 100mL volumetric flask add 25mL of reagent water and 5mL of concentrated HNO_3 (Section 8.6). Add 1mL of 1000ppm Mercury Stock Standard (Section 8.10-use alternate source than that used for both the calibration standards and the LCS Stock Standard). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.

8.18 Mercury Working ICV Standard, 0.3ppm: To a 100mL volumetric flask add 25mL of reagent water and 5mL of concentrated HNO_3 (Section 8.6). Add 3mL of 10ppm Stock ICV Standard (Section 8.5). Bring to volume with reagent water. Store at room temperature. Expires one week from date of preparation.

8.19 Mercury ICV Standard, 0.003ppm: Prepare daily with each batch of samples. To a 25mL digestion vessel add 10mL of reagent water. Add 0.25mL of 0.3ppm Working ICV Standard (Section 8.18). Bring to a 25mL final volume with reagent water and carry through entire digestion process as in Section 10.1.1..

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

The ICB, CCB, and Method Blank: A 25mL aliquot of 0ppm calibration standard brought through the preparation procedure as outlined in Section 10.1.1 . Blank results must be <RL. See Section 12.1 for corrective action. An ICB is analyzed after the initial calibration or re-calibration. The CCB is analyzed at every 10 sample injection interval. Method Blank is analyzed once per batch of samples; batch consists of 20 samples.

9.2 Laboratory Control Samples (LCS)

The LCS Standard consists of a 0.001ppm Mercury LCS Standard (Section 8.16). This standard is brought through the preparation procedure as outlined in Section 10.1.1. The LCS Standard must be recovered within ± 20% of the true value. See Section 12.3 for corrective action. The LCS Standard is analyzed once per batch of samples. A batch consists of 20 samples.

9.3 Initial Calibration verification (ICV)

The ICV Standard consists of a 25mL aliquot of 0.003ppm Mercury ICV Standard (Section 8.19). The ICV must be recovered within 10% of the true value. See Section 12.2 for corrective action.

9.4 Continuing Calibration Verification (CCV)

The CCV Standard consists of a 0.010ppm calibration standard (Section 8.13.3). The CCV must be recovered within 20% of the true value. See Section 12.2 for corrective action.

9.5 Matrix Spike

A matrix spike is analyzed once per batch of samples. A batch consists of 20 samples for monitoring wells, surface waters, influents and effluents. Prepare the matrix spike at 0.005ppm by adding 1.25mL of 0.1ppm Mercury Stock Standard (Section 8.3) to 25mL of the selected QC sample. The final concentration of the matrix spike is 0.005mg/L.

The matrix spike sample is brought through the preparation procedure as outlined in Section 10.1. A matrix spike is analyzed once per batch of samples. A batch consists of 20 samples for monitoring wells and surface waters. The recovery of the matrix spike must be between 75 – 125% (using the calculation in Section 11.2).

If the recovery of the matrix spike is out of range, a post-analytical spike is analyzed. Prepare the post analytical spike by adding 5mL of 0.010ppm Calibration Standard / Post Analytical Spike Solution (Section 8.1.6) and 5mL of the sample digestate to a 50mL centrifuge tube for a final concentration of 0.005mg/L. Analyze the post spike as outlined in Section 10.3.5.

Calculate the post spike concentration as follows:

Post Analytical Spike Sample Concentration (mg/L) =

[Sample Concentration (mg/L) x (0.5)] + 0.005mg/L

The percent recovery of the post-analytical spike must be between 75 – 125%. See Section 12.4 for corrective action.

9.6 Laboratory Duplicate

A sample is analyzed in duplicate once per batch of samples. A batch consists of 20 samples for monitoring wells and surface waters. The RPD between the sample and its duplicate must be 20% or less (using the calculation in Section 11.3), See Section 12.5 for corrective action.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Calibration Blank
- 0.0002 ppm Calibration Standard
- 0.0005 ppm Calibration Standard
- 0.001 ppm Calibration Standard
- 0.002 ppm Calibration Standard
- 0.010 ppm Calibration Standard
- 0.020 ppm Calibration Standard
- ICV
- ICB
- Ten analytical samples
- CCV
- CCB
- Ten analytical samples
- CCV
- CCB
- Etc.

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation and Digestion

Samples, Standards and All Batch QC

Transfer 25mL of well-homogenized sample (or an aliquot of the sample diluted to 25mL with reagent water) or standards (Sections 8.13.1 through 8.13.7, 8.16 and 8.19) to a 50mL centrifuge tube.

Add 1.25mL of concentrated H_2SO_4 (Section 8.2), 0.625mL of concentrated HNO_3 (Section 8.6), Add 3.75mL of Potassium Permanganate Solution, shake and add additional portions of potassium permanganate solution (if necessary) to all samples and QC, until the purple color persists for at least 15 min. (Section 8.8). Add 2mL of Potassium Persulfate Solution (Section 8.9), and heat samples for 2 hours in a 95°C +/-3

heating block. Cool, and add 1.5mL of Sodium Chloride-Hydroxylamine hydrochloride solution (Section 8.7).
Filter the sample if needed to remove any sediment or particulate.

Analyze samples and the digested calibration standards (Sections 8.13.1 through 8.13.7) are used in Section 9.2 to generate a calibration curve.

10.2 Initial Calibration

Construct a calibration curve by plotting the absorbances of prepared standards (Section 10.1.1) versus micrograms of mercury. Determine the peak height of the unknown from the absorbance maxima on the spectrometer, and read the mercury value from the standard curve. (See Section 11.)

The curve correlation coefficient (cc) must be greater than or equal to 0.995 in order for the curve to be linear. If the correlation coefficient is less than 0.995, find and correct the problem. When the problem has been corrected, re-analyze either the previous standards or new standards. When the curve has generated an acceptable cc then the analysis can continue with the ICV/ICB.

10.3 Equipment Operation and Sample Processing

Sample and standard analysis:

10.3.1 Instrument Setup

10.3.1.1 Turn the instrument on. The autosampler will initialize itself.

10.3.1.2 Choose the instrument software from the desktop menu. The autosampler will initialize again.

FIMS 100 NOTE: The instrument must be turned on before the application is started. Otherwise, an error message will result.

10.3.1.3 Enter the appropriate fields for sample identification, and data storage.

10.3.1.4 Fill the carrier and reductant bottles.

10.3.1.4.1 The Carrier is 3% HCl (Section 8.6).

10.3.1.4.2 The Reductant is 1.1% $SnCl_2$ in 3% HCl (Section 8.5).

10.3.1.5 Allow the instrument to warm up while clearing samples. Samples that are cloudy or with particulate present after clearing must be filtered through Whatman 41 filter paper (Section 7.11) before analysis.

10.3.1.6 Place carrier uptake line and reductant uptake line.

10.3.1.7 Load carrier and reductant lines into pump magazines

10.3.1.8 Load the two waste lines into the pump magazines below the roller.

10.3.1.9 Lock the magazines into place.

FIMS100 only:

10.3.1.10 Remove the cap from the liquid/vapor separator and wipe dry with a Lab Wipe (Section 7.9). Compressed air (Section 7.10) through the vapor transfer line to dry it out.

10.3.1.11 Place a PTFE membrane (Section 7.7), rough side up, in the liquid/vapor separator; replace the cap and reattach the vapor transfer line to the sample absorption cell.

10.3.1.12 Adjust the gas flow by turning the black knob below the air flow meter to obtain a reading of just over 50.

10.3.2 Calibration and Sample Analysis

10.3.2.1 The instrument will now run the calibration standards; verify a CC of 0.995 or better before proceeding with the ICV and ICB. Ten analytical samples, a CCV and CCB, ten analytical samples, CCV, CCB, etc. The CCBs and CCVs must be recovered within the proper ranges (Sections 9.4 and 9.1.3) for analysis to continue.

10.3.2.2 If the sample result is beyond 90% of the concentration of the highest point on the calibration curve or LDR study used to establish the linear range, dilute the sample extract with a portion of one of the prepared blanks (ICB, CCB or PBS) to produce an analytical result that is within the range.

10.3.3 Instrument Shut Down

10.3.3.1 When analysis is complete place reagent uptake lines in a beaker of reagent water.

10.3.3.1.1 Continue to run the pumps for several minutes to flush reagents out of the lines.

10.3.3.1.2 Continue to run the pumps for several minutes to flush reagents out of the lines.

10.3.3.2 Pull the reagent uptake lines out of the reagent water beaker to allow the pump to draw air through the lines.

10.3.3.3 Unlock the top and bottom pump magazines and remove tubing from the magazines.

10.3.3.4 Exit from the software application.

10.3.3.4.1 Dump the samples and instrument waste in the Metals/WetChem waste drum located in the transfer room.

10.4 Continuing Calibration

Continuing calibration verification samples are analyzed after every 10 samples in the sample run, as outlined in Section 10.3.5.

10.5 Preventative Maintenance

Preventative maintenance is conducted per the manufacturer's instructions. All preventative maintenance is recorded in the Instrument Maintenance Logbook.

11. Data Evaluation, Calculations and Reporting

11.1 Calculate Mercury concentration

Calculate Mercury concentration from the daily calibration curve. The curve is generated utilizing a straight-line equation defined as:

$$A = k_1 + k_2C$$

Where:

A = Average peak height of the sample/standard integrations
C = Sample/Standard Concentration, µg/L
k_1 = y-intercept
k2 = slope

The instrument will plot peak height against concentration (µg/L). The result is generated in µg/L . This value is divided by 1000 to convert the units to mg/L. If the sample is diluted (DF), the result is multiplied by the DF to generate the final result.

Result, mg/L = (concentration, µg/L) x (1mg/1000µg) x (DF)

11.2 Matrix Spike Calculation

Calculate percent recovery for the Matrix Spike corrected for concentrations measured in the unfortified sample. Percent recovery is calculated using the following equation:

$$\% \text{ Recovery} = \frac{(Cm - C)}{s} \times 100$$

Where:

Cm = measured Mercury in the fortified sample
C = measured native mercury sample concentration
S = concentration equivalent of spike added to sample

11.3 Relative Percent Difference (RPD) Calculation

Calculate the Relative Percent Difference (RPD) for each Duplicate of the initial quantitated concentration (IC) and duplicate quantitated concentration (Dc) using the following formula:

$$RPD = \frac{|(IC - Dc)|}{\{(IC + Dc) / 2\}} \times 100$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 Method Blank Failure: When a prep blank mercury level constitutes 10% or more of analyte level determined for any sample in the batch, or is greater than 2.2x the MDL value (whichever is greater), the associated samples in the batch must be redigested (Section 10.1).

For method blanks that have concentrations greater than the RL, the data is rejected and the associated samples sent back for redigestion unless the associated sample concentrations are greater than 10x the blank concentration. In this case the blank is narrated and the results are reported without qualification.

12.2 ICV / CCV Failure: If the ICV %Recovery is outside of acceptance criteria, the ICV is reinjected. If the %Recovery is outside the acceptance criteria, the analysis is terminated until the problem is found and corrected. If the CCV %Recovery is outside of acceptance criteria, the CCV is reinjected. If the % Recovery is still outside the acceptance criteria, all samples analyzed since the last acceptable CCV must be reanalyzed following correction of the problem.

12.3 LCS Failure: If the LCS is not recovered within acceptance criteria, the LCS is reinjected. If the % Recovery is still outside the acceptance criteria, either recalibrate and rerun or the associated batch and another LCS must be redigested (Section 10.1).

12.4 Matrix Spike / Post Digestion Spike Failure: If the recovery of the matrix spike is outside of the acceptance criteria, a post digestion spike is performed (Section 9.54). If the post digestion spike is outside of 75 – 125%, a narrative must be included with the data. (Section 10.1).

12.5 Duplicate Failure: If the RPD between the sample and its duplicate is greater than 20%, visually evaluate the sample matrix. If the matrix appears problematic, the sample digestate may be diluted and reanalyzed, or a narrative included with the data to explain the matrix problem.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

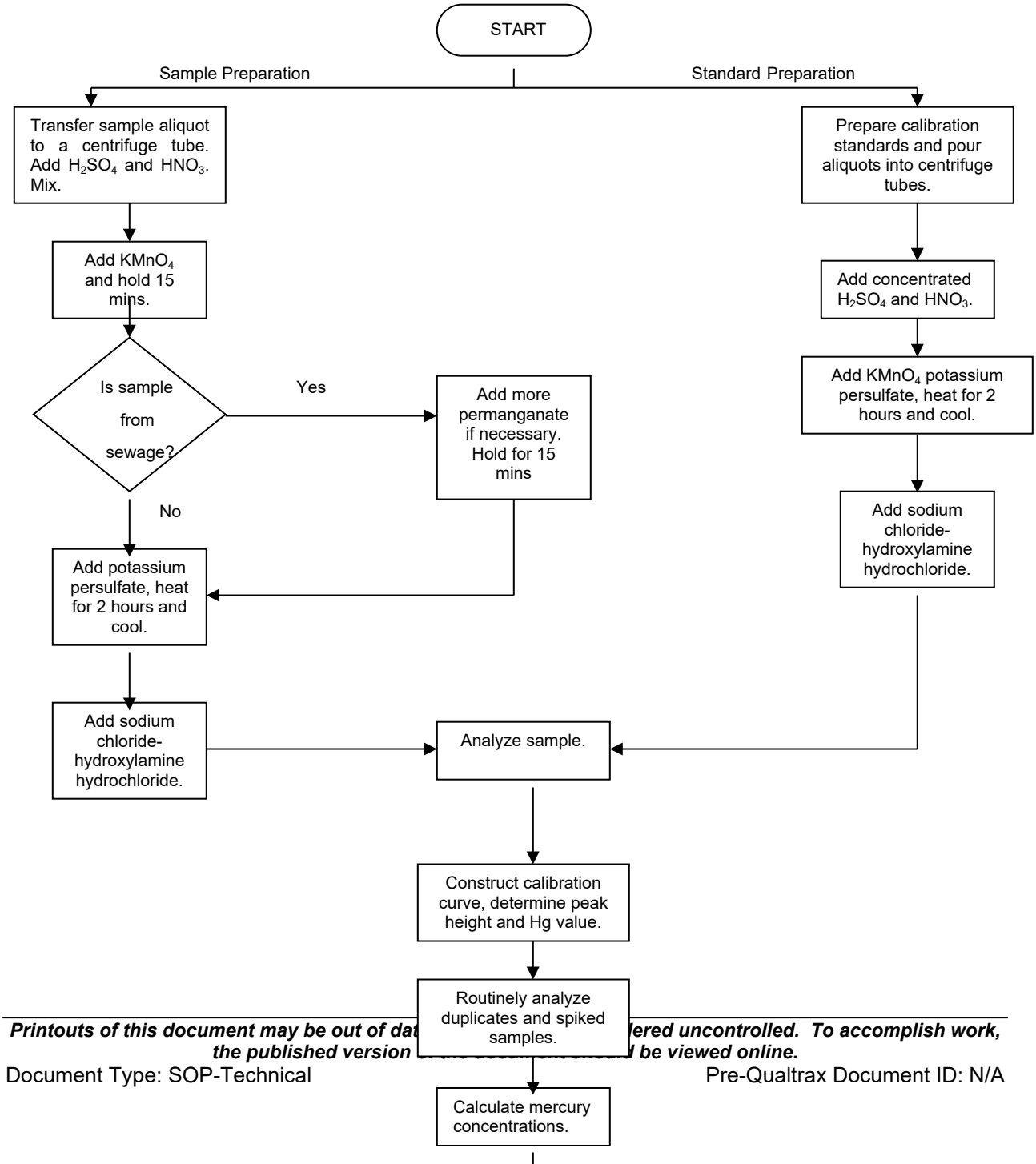
SOP/1797 Waste Management and Disposal SOP

16. Attachments

Figure 1: Method 7470A Flow Chart

Figure 1

Method 7470A Flow Chart



END

Mercury Determination in Tissue and Soil/Sediment Samples by Cold Vapor Atomic Fluorescence Technique (CVAF)

References: **Method 7474,** Mercury in Sediment and Tissues by Atomic Fluorescence Spectrometry. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 0, 2007.

Method 7471B, Mercury in Solid or Semisolid Waste (Manual Cold Vapor Technique). SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 2, February 2007.

1. Scope and Application

Matrices: The method is applicable to total mercury in tissue and soil/sediment samples.

Definitions: Refer to Alpha Analytical Quality Manual.

Two procedures for the analysis of mercury by cold vapor atomic fluorescence spectrometry are discussed in this SOP.

Mercury in Tissues - This method utilizes a microwave digestion followed by bromine chloride oxidation. Samples are then decolorized using hydroxylamine hydrochloride and analyzed following reduction with stannous chloride. Elemental mercury is then purged from solution and analyzed by atomic fluorescence spectrometry.

Mercury in Soil/Sediments - This method utilizes a hot plate digestion with aqua regia followed by bromine chloride oxidation. Samples are then decolorized using hydroxylamine hydrochloride and analyzed following reduction with stannous chloride. Elemental mercury is then purged from solution and analyzed by atomic fluorescence spectrometry.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Mercury analyzer and in the interpretation of Mercury data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Parameter	CAS
Mercury	7439-97-6

2. Summary of Method

Mercury in tissue samples is determined by cold vapor atomic fluorescence spectrometry from an aliquot of a microwave digestion (see SOP 2150; method 3051). A 10 mL aliquot of the digest is

added to a 50 mL screw cap polyethylene digestion tube along with 2 mL of an acidic bromine chloride solution. Bromine chloride is prepared by mixing potassium bromide with potassium bromate solutions with hydrochloric acid producing a yellow solution. Bromine chloride is known to oxidize inorganic and organomercury compounds. If a yellow color does not persist after addition of bromine chloride, then additional solution must be added until a yellow color persists. The same volume of bromine chloride solution is added to all samples and standards. This solution is allowed to stand for at least 15 minutes. After digestion is complete, bring to 50 mL, add 0.4 mL of 12% hydroxyl amine hydrochloride solution and shake. Additional hydroxyl amine hydrochloride is added if the yellow color from the bromine chloride solution persists.

Samples and standards are placed in the autosampler rack for analysis following stannous chloride reduction and analysis by cold vapor atomic fluorescence spectrometry at a wavelength of 253.7 nm.

Mercury in soil/sediment samples is determined by cold vapor atomic fluorescence spectrometry from a hot plate digestion utilizing aqua regia. A 0.5 – 1 g wet sediment aliquot is added to a 50 mL screw cap polyethylene digestion tube along with 5 mL of DI water, 3.75 mL concentrated HCl and 1.25 mL of concentrated HNO_3. Samples are heated on the hot block for 2 minutes and allowed to cool. 25 mL of Di water are then added, the samples placed back on the hot block and heated for 30 minutes. The samples are then cooled and brought to 50 mL with DI water. A 10 mL aliquot of the digest is added to a 50 mL digestion tube along with 2 mL of an acidic bromine chloride solution. If a yellow color does not persist after addition of bromine chloride then additional solution must be added until a yellow color persists. The same volume of bromine chloride solution is added to all samples and standards. This solution is allowed to stand for at least 15 minutes. After digestion is complete, bring to 50 mL, add 0.4 mL of 12% hydroxyl amine hydrochloride solution and shake. Additional hydroxyl amine hydrochloride is added if the yellow color from the bromine chloride solution persists.

Samples and standards are placed in the autosampler rack for analysis following stannous chloride reduction and analyzed by cold vapor atomic fluorescence spectrometry at a wavelength of 253.7 nm.

2.1 Method Modifications from Reference

Method 7474 calls for a microwave digestion for both tissues and soils/sediments with nitric and hydrochloric acids. The method has been modified for tissues to use an aliquot of a microwave digest utilizing nitric acid only. The method has been modified for soils/sediments to use a hot block digestion similar to that used for Method 7471B but without the addition of potassium permanganate. The change allows for more rapid preparation of samples and yields equivalent data to the reference method as indicated by the performance of PT samples.

Bromate/Bromide solution has been modified from the method reference.

3. Reporting Limit

The Reporting limit for mercury in tissues is 0.05 µg/L (0.0125 mg/kg) and the linear range of the analysis is 5 µg/L (1.25 mg/kg).

The Reporting limit for mercury in soil/sediment is 0.05 µg/L (0.0125 mg/kg) and the linear range of the analysis is 5 µg/L (1.25 mg/kg).

4. Interferences

4.1 Gold, silver and iodide are known interference.

4.1.1 High concentrations of gold and silver in a sample may suppress mercury reduction.

4.1.2 Samples with iodide concentrations greater than 3 mg/L should be pre-reduced with stannous chloride to clarify the brown color. It may be necessary to clean the analytical system with 4N HCl following analysis of a sample with iodide concentrations greater than 30 mg/L.

4.1.3 Sulfide concentrations up to 24 mg/L produce no interference problems.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. Eating, drinking, smoking, or the application of cosmetics is not permitted in the laboratory area. Horseplay of any kind is prohibited. Pipetting by mouth is not permitted. All Personal Protective Equipment (PPE) must be removed before leaving the laboratory area and before entering the employee lounge or eating area. Always wash your hands before leaving the laboratory. All relevant Material Safety Data Sheets (MSDSs) are kept alphabetically in a centrally located file storage.

Approved PPE, which includes Safety Glasses, Gloves and Lab Coats, must be worn at ***all*** times when handling samples, reagents, chemicals, or when in the vicinity of others handling these items, so that dermal contact is avoided. All standards, reagents and solvents shall be handled under a hood using the proper PPE. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, use a secure transporting device and/or secondary outer container. Chemical storage is properly segregated and adequately ventilated to reduce the possibility of hazardous reactions. Chemical storage in work areas shall be kept to a minimum. Storage on bench tops or other work surfaces, except temporary, is not permitted.

Spilled samples, solvents, reagents, and water must be cleaned up from bench tops, instruments and autosampler surfaces immediately. A spill is considered a quantity of hazardous material if it is moderate to extreme hazard to the skin and mucous membranes. If contact with the skin occurs, immediately flush with large volumes of water. In the case of acidic/basic spills, the *Spill Kit* located in each laboratory shall be utilized before attempting to cleanup the spill. Although procedures are designed to minimize the possibility of an accident, all injuries or accidents, regardless of the nature or severity, are to be reported to the Department Manager Supervisor immediately. If an employee discovers a potentially unsafe condition, this must be reported to the Department Manager Supervisor immediately. No employee should feel compelled to work in a situation where they do not feel entirely informed, trained, or safe.

Analytical instrumentation poses the unique possibility of exposure to high voltages. Other than the *routine* instrument maintenance, as listed in the front of every Instrument Maintenance Logbook, at no time shall an instrument operator attempt to maintenance an instrument alone, or without the proper training, supervision or instruction. Caution must always be used in the presence of moving parts (autosamplers) and hot surfaces (injection ports).

Compressed gas cylinders shall only be moved with the dolly supplied for this specific purpose. The cap must be on the cylinder while it is being moved. The tank must be secured when in its final position. All spent tanks are to be returned in the same manner, and secured until removed by the vendor. Liquid argon or nitrogen represents a potential cryogenic hazard and safe-handling procedures must be used at all times.

All additional company safety practices shall be followed at all times as written in the *Chemical Hygiene Plan.*

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

A minimum of 10g of sample must be collected in a pre-cleaned 2oz or 4oz glass jar with a Teflon lined screw cap.

6.2 Sample Preservation

No preservatives are used. See Section 6.4.

6.3 Sample Shipping

No special sample shipping requirements.

6.4 Sample Handling

Samples are stored at 4°C $\pm$ 2°C. The hold time for soil/sediment and tissue samples is 28 days. Sediment and tissue samples may be stored frozen at -20°C $\pm$ 10°C. The hold time for solid samples stored frozen may be extended unless specifically noted by project or client requirements.

7. Equipment and Supplies

Instrument:

7.1 PSA Millennium Merlin Atomic Fluorescence Analyzer equipped with a PC loaded with instrumental software, and PSA 20.400 autosampler. The instrument is connected to a source of high-purity, mercury free argon regulated to 50 PSI.

7.2 Santoprene Peristaltic Pump Tubing: Green/Green (PSA part number M025T002 or equivalent) for blank and autosampler rinse, Grey/Grey (PSA part number M055T005 or equivalent) for sample and stannous chloride.

7.3 Electric Hot Plate: Adjustable and capable of maintaining a temperature of 90-95°C equipped with graphite carbon blocks that each have 36 positions to hold the sample tubes.

NOTE: Hotplate/Block temperatures are monitored regularly using NIST calibrated and traceable thermometers. If any thermometer is suspected to not be reading the temperatures correctly, it is replaced with another certified thermometer.

7.4 CEM Mars 6 Xpress Microwave Oven: Equipped with temperature control sensors and high pressure perfluoropolymer vessels.

7.5 Digestion Tubes: 50mL disposable polyethylene screw cap digestion tubes.

7.6 Other: Adjustable Eppendorf pipettes and replacement tips

8. Reagents and Standards

ACS Trace Metal grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

Solutions below expire six months from preparation unless noted.

Stock standard solutions are stored in a cabinet, out of direct light

8.1 Deionized (DI) water: The Barnstead NANO-pure system provides Type I water used in the preparation of samples and standards.

8.2 Hydrochloric acid, concentrated (HCl): ACS reagent grade Hydrochloric acid (1:1 HCl) - Add 500mL concentrated HCl to 400mL DI water and dilute to 1L. 10% HCl – Add 100 mL concentrated HCl to approximately 500 mL DI and bring to 1 L with DI. Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.3 Nitric acid, concentrated (HNO_3): ACS reagent grade Nitric Acid (1:1 HNO_3) - Add 500mL concentrated HNO_3 to 400mL DI water and dilute to 1L. Prepare 1% Nitric Acid by diluting 1mL of concentrated HNO_3 up to 100mL in DI water. Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.4 Hydroxylamine hydrochloride ($NH_2OH{\cdot}HCl$): ACS reagent grade. Prepare 12% solution by adding 12 g per 100 mL of DI.

8.5 Stannous Chloride ($SnCl_2{\cdot}H_20$): ACS reagent grade. Prepare 2% solution by adding 20 g to 1L of 10% HCl. Prepare daily. Solution may be purged with argon at 2 L/minute for 30 minutes to remove Hg impurities.

8.6 Potassium Bromate ($KBrO_3$): ACS grade. May be baked in muffle furnace at 250°C for 8 hours to remove mercury impurities.

8.7 Potassium Bromide (KBr): ACS grade. May be baked in muffle furnace at 250°C for 8 hours to remove mercury impurities.

8.8 Bromine Chloride (BrCl): Prepare in a BOD bottle under a fume hood by adding 5.4 g KBr and 7.6 g $KBrO_3$ to 500mL of concentrated HCl and mix. Final concentration is 0.2N. Solution will turn yellow. **Free Halogens are generated from this solution. Seal the bottle when taking out of fume hood.**

8.9 Stock and Working Standards:

The 1000 µg/L working standards must be prepared in the same manner as samples. These standards are then diluted to make calibration and calibration check standards. After preparation, the 1000 µg/L working standards can be used for one week.

8.9.1 1000 mg/L Stock Calibration Mercury Standard. Purchased.

8.9.1.1 1000 µg/L Working Standard for Tissues: Prepare by adding 0.05mL of 1000 mg/L stock to a microwave digestion vessel containing 10mL of concentrated HNO_3. This standard is digested along with samples according to Method 3051 (SOP 2150). The standard is then brought to a 50mL final volume with DI water in a screw cap digestion tube.

8.9.1.2 1000 µg/L Working Standard for Soils/Sediments: Prepare by adding 0.05 mL of 1000 mg/L stock (Section 8.9.1) to a 50 mL screw cap digestion tube containing 5 mL of DI water, 1.25 mL of concentrated HNO_3 and 3.75 mL of concentrated HCl. This standard is digested along with samples. The standard is then brought to a 50 mL final volume with DI water.

8.9.1.3 100 µg/L Intermediate Standard: Prepare by adding 0.1 mL of the 1000 µg/L working standard (Section 8.9.1.1 or 8.9.1.2) to 0.9 mL DI water. This intermediate standard is used for calibration standards preparation and then discarded.

8.9.1.4 10 µg/L Intermediate Standard: Prepare by adding 0.1 mL of the 100 µg/L working standard (Section 8.9.1.3) to 0.9 mL DI. This intermediate standard is used for calibration standards preparation and then discarded.

8.9.1.5 5 µg/L Intermediate Standard: Prepare by adding 0.25 mL of the 1000 µg/L working standard (Section 8.9.1.1 or 8.9.1.2) to 50 mL graduated digestion tube containing 2% HCl. Bring to 50 mL final volume.

8.9.2 1000 mg/L Stock ICV Mercury Standard: Purchased.

8.9.2.1 1000 µg/L ICV Working Standard for Tissues: Prepare by adding 0.05 mL of 1000 mg/L stock to a microwave digestion vessel containing 10 mL of concentrated HNO_3. This standard is digested along with samples according to Method 3051 (SOP-2150). The standard is then brought to a 50 mL final volume with DI water in a screw cap digestion tube.

8.9.2.2 1000 µg/L ICV Working Standard for Soils/Sediments: Prepare by adding 0.05 mL of 1000 mg/L stock to a 50 mL screw cap digestion tube containing 5 mL of DI water, 1.25 mL of concentrated HNO_3 and 3.75 mL of concentrated HCl. This standard is digested along with samples. The standard is then brought to a 50 mL final volume with DI water.

8.9.2.3 5 µg/L Intermediate Standard: Prepare by adding 0.25 mL of the 1000 µg/L working standard (Section 8.9.2.1 or 8.9.2.2) to 50 mL graduated digestion tube containing 2% HCl. Bring to 50 mL final volume.

8.9.2.4 1000 µg/L LCS and Matrix Standard for Tissues and Soils/Sediments: Prepare by adding 0.1 mL of 1000 mg/L stock (8.9.2) to a 100 mL volumetric flask containing 2% HCL.

8.10 Calibration and Calibration Check Standards

8.10.1 Standards Preparation for Method 7474 Tissues

To 50 mL screw cap digestion tubes, add 10 mL of DI water and 2 mL of concentrated HNO3. Add the volumes of each standard as indicated in the table below, 2 mL of BrCl solution and bring to 50 mL. The same volume of the 1000 µg/L ICV stock is added to the preparation batch LCS and matrix spike sample.

If more than 2 mL of BrCl solution was added to samples to maintain the yellow color, add the same amount to standards. 0.2 mL of 12% hydroxyl amine hydrochloride is added to each vial and shaken. The yellow color should disappear.

8.10.2 Standards Preparation for Method 7474 Soils/Sediments

To 50 mL screw cap digestion tubes, add 10 mL of DI water, 0.75 mL of concentrated HCl and 0.25 mL of concentrated HNO_3. Add the volumes of each standard as indicated in the table below, 2 mL of BrCl solution and bring to 50 mL. The same volume of the 1000 µg/L ICV stock is added to the preparation batch LCS and matrix spike sample.

If more than 2 mL of BrCl solution was added to samples to maintain the yellow color, add the same amount to standards. 0.2 mL of 12% hydroxyl amine hydrochloride is added to each vial and shaken. The yellow color should disappear.

8.10.3 Calibration, Calibration Check Standards, LCS and Matrix Spike

Calibration and calibration check standards are prepared by adding standard volumes to vials prepared for tissues and soils/sediments as designated in the following table:

Standard ID	Vol. 1000 µg/L (mL) (Sec. 8.9.1.1 or 8.9.1.2)	Vol. 100 µg/L (mL) (Sec. 8.9.1.3)	Vol. 10 µg/L (mL) (Sec. 8.9.1.4)	Final Concentration (µg/L)
STD0	-	-	-	0.0
STD1	-	-	0.25	0.05
STD2	-	0.05	-	0.1
STD3	-	0.25	-	0.5
STD4	0.1	-	-	2.0
STD5	0.25	-	-	5.0
ICV, CCV	0.125 (Sec. 8.9.2.1 or 8.9.2.2)			2.5
LCS, MS	0.125 (Sec. 8.9.2.4)			2.5

Lower detection limits and analytical reporting limits may be required at times for some samples. Lower limits are attained by increasing the instrumental gain from 10 to 100 and by calibrating using lower concentration standards. Calibration standards and the LCS and MS are prepared as in the following table:

Standard ID	Vol. 5 µg/L Standard (Section 8.9.1.1 through .5) (mL)	Final Concentration (µg/L)
STD0	-	0.000
STD1	0.05	0.005
STD2	0.10	0.010
STD3	0.20	0.020
STD4	0.50	0.050
STD5	1.0	0.100
STD6	2.0	0.200
ICV,CCV,LCS, MS	1.0	0.100

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blanks

9.1.1 Method Blank

A method blank must be prepared once per every 20 samples or per digestion batch, whichever is more frequent.

Mercury must not be detectable in the method blank at a concentration greater than the reporting limit.

Corrective Action: Analysis according to the appropriate analytical SOP may be repeated once to see if an analytical error has occurred. Digestion of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control method blank are non-detect for the elements of interest, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high method blank and the client is notified in a project narrative associated with the sample results.

9.1.2 Initial Calibration Blank (ICB)

An initial calibration blank must be analyzed immediately following the ICV (see 9.3).

Mercury must not be detectable in the ICB at a concentration greater than the reporting limit.

Corrective Action: May reanalyze the standard once. If the concentration still exceeds the acceptance limit, stop and recalibrate the instrument. Samples cannot be analyzed until

an acceptable ICB analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control blank are non-detect for mercury, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high blank and the client is notified in a project narrative associated with the sample results.

9.1.3 Continuing Calibration Blank

A continuing calibration blank must be analyzed immediately following the CCV (see 9.4).

Mercury must not be detectable in the CCB at a concentration greater than the reporting limit.

Corrective Action: May reanalyze the standard once. If the concentration still exceeds the acceptance limit, stop, recalibrate the instrument and reanalyze all associated samples. Samples cannot be analyzed until an acceptable CCB analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control blank are non-detect for the elements of interest, or if sample concentrations are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high method blank and the client is notified in a project narrative associated with the sample results.

9.2 Laboratory Control Sample (LCS)

Laboratory control sample (LCS) must be prepared once per every 20 samples or per digestion batch, whichever is more frequent, and spiked with a solution prepared from a second source or lot number, other than the source used to verify the accuracy of the standard curve for the determinative analytical method. The LCS contains all target elements of interest, and is digested along with the samples as verification of the accuracy of the entire digestion procedure.

The acceptable recovery QC limits are 80 – 120%. The recovery limits are continuously monitored and documented in-house through control charts. The *Control Limit Generation* SOP (1734) provides details explaining how control charts are generated and used for quality control.

Corrective Action: Analysis according to the appropriate analytical SOP may be repeated once to see if an analytical error has occurred. If the LCS recovery is still out of control, re-prepare and re-analyze the LCS and all associated samples. Samples cannot be reported until an acceptable LCS is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with the out of control LCS are also associated with a matrix spike that is in control. This is an acceptable measure of accuracy of the digestion and analytical procedures. An explanation of the out of control LCS recovery must be included in the project narrative to the client and the sample data reported noting the acceptable MS results as batch QC.

9.3 Initial Calibration Verification (ICV)

An initial calibration verification standard from a source different from that used to calibrate the instrument (see 8.9.2) must be analyzed immediately following calibration as an independent check of instrument performance. Nominal acceptance limits are 90% - 110% of the true value.

Corrective Action: May reanalyze the standard once. If the concentration still exceeds the acceptance limit, stop and recalibrate the instrument. Samples cannot be analyzed until an

acceptable ICV analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with a high recovery ICV are non-detect. In such cases, the sample results are accepted without corrective action for the high ICV recovery and the client is notified in a project narrative associated with the sample results.

9.4 Continuing Calibration Verification (CCV)

A continuing calibration verification standard must be analyzed after analysis of no more than 10 samples as an on-going check of instrument performance. Nominal acceptance limits are 90% - 110% of the true value.

Corrective Action: May reanalyze the standard once. If the concentration still exceeds the acceptance limit, stop, recalibrate the instrument and reanalyze all associated samples. Results cannot be accepted until an acceptable CCV analysis is obtained. Exceptions may be made with approval of the Metals Department Manager, Laboratory Director or QAO, if the samples associated with a high recovery CCV are non-detect. In such cases, the sample results are accepted without corrective action for the high CCV recovery and the client is notified in a project narrative associated with the sample results.

9.5 Matrix Spike

A matrix spike (MS) sample must be performed once per 20 samples (5% frequency), or per digestion batch, whichever is more frequent. The MS contains all target elements of interest.

The acceptable % recovery QC limits are 80% – 120%. The % recovery QC limits are continuously monitored and documented in-house through control charts. The *Control Limit Generation* SOP (1734) provides details explaining how control charts are generated and used for quality control.

Corrective Action: Analysis according to the appropriate analytical SOP may be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits and the LCS is compliant, include a project narrative with the results to the client noting that there may be potential matrix effects on the accuracy of the reported results as evidenced by MS recovery outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses (matrix or sample duplicate) must be performed once per 20 samples 5% frequency), or per digestion batch, whichever is more frequent.

Acceptable relative percent differences (RPD) of duplicates is $\leq$ 20%. This acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate the RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:

R1 = sample Replicate #1
R2 = sample Replicate #2

The RPD limits are continuously monitored and documented in-house through control charts. The *Control Limit Generation* SOP (1734) provides details explaining how control charts are generated and used for quality control.

Corrective Action: Analysis may be repeated once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the reported metals results as evidenced by the matrix duplicate %RPD exceedance.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- ICV
- ICB
- Method Blank
- Laboratory Control Sample
- Matrix QC Sample
- Matrix Duplicate
- Matrix Spike
- Samples 2-5
- CCV
- CCB
- Samples 6-15
- CCV
- CCB

10. Procedure

10.1 Equipment Set-up

Samples are prioritized by the Metals Department Manager or Preparation Group Leader for digestion based on hold time and client due date. The analyst must be familiar with instrument software and hardware as described in the user manuals supplied with the instrument before attempting to perform an analysis.

Power to the instrument, autosampler , PC and the argon gas supply are turned on. Peristaltic pump windings are inspected for wear and replaced if necessary. The winding clips are relaxed in between usage. The instrument manufacturer suggests that the pump tubing be changed once per month or more frequently as needed.

The stannous chloride solution is attached to the grey/grey pump tubing, 10% HCl is attached to the green/green autosampler rinse and DI is attached to the geen/green rinse blank. The

autosampler probe line is attached to a grey/grey pump tubing. The pumps are turned on via the software and the flow is checked to insure that solutions are delivered to the mixing valve.

10.1.1 Sample Preparation

10.1.1.1 Tissues

Tissue samples are prepared by microwave oven assisted acid digestion according to SOP 2150. The LCS and MS are spiked as indicated in the table in 8.10.3.

A 10 mL aliquot of each digest is added to a 50 mL screw cap digestion tube along with 2 mL of BrCl solution. The vial contents should remain a yellow color. If a yellow color does not remain, add more BrCl solution. This solution is allowed to stand for 15 minutes. Bring the vial to 50 mL with DI water . The vials are then decolorized with 0.4 mL of 12% hydroxyl amine hydrochloride and shaken. The yellow color should disappear. If the solution remains yellow, add more hydroxyl amine until the color disappears. Add the same amount to all samples and standards.

10.1.1.2 Soils/Sediment

A 0.5 – 1.0 g wet weight well mixed aliquot of soil/sediment is added to a 50 mL screw cap digestion tube with 5 mL of DI water. The LCS and MS are spiked as indicated in the table in 8.10.3. A 3.75 mL volume of concentrated HCl and 1.25 mL of concentrated HNO_3 are added and the tube placed on the hot block set to 95 ± 3 ^oC for 2 minutes. Samples are allowed to cool and then 25 mL of DI water is added to each vial and placed back on the hot block for 30 minutes.

After cooling bring to a final volume of 50 mL with DI water. Then a 10 mL aliquot of each digest is added to a 50 mL screw cap digestion tube along 2 mL of BrCl solution. The vial contents should remain a yellow color. If a yellow color does not remain, add more BrCl solution. Allow samples to react for 15 minutes. The vials are then decolorized with 0.4 mL of 12% hydroxyl amine hydrochloride, brought to a final volume of 50 mL and shaken. The yellow color should disappear. If the solution remains yellow, add more hydroxyl amine until the color disappears. Add the same amount to all samples and standards.

10.2 Initial Calibration

The instrument is calibrated prior to sample analysis and at least once every 24 hours. A linear least squares regression calibration curve is selected for Method 7474.

In all cases, a correlation coefficient ≥ 0.995 is required before the analysis of samples can begin. The calibration blank is included as a point in the calibration curve.

An ICV (Section 9.3) and ICB (Section 9.1.2) are analyzed after calibration.

10.3 Equipment Operation and Sample Processing

After initializing the Millennium software, the instrument is turned on via the software and allowed to warm up for 30 minutes. The analysis method is selected and the autosampler template is selected. Method 7474 is set up in the software which specifies delay, read and

rinse times as well as pump speeds. An autosampler template is set up specifying calibration standard, QC standards and positions on the autosampler rack. Once sample ID's have been typed in, the file is saved as the Job number.

A Work Group is obtained from LIMS and used as the data file name.

The analysis is initiated by clicking on the start button on the analysis page.

Following analysis, the data file is exported to the LIMS on the O: Metals\Millennium drive for data processing. The instrument and autosampler are then rinsed with DI water and turned off

10.4 Continuing Calibration

Periodically (after no more than 10 samples) and at the end of the analysis, CCV standards (Section 9.4) and CCB standards (Section 9.1.3) are analyzed.

10.5 Preventive Maintenance

Inspect pump tubing for signs of wear and replace monthly or more frequently if necessary.

Clean glass liquid separator when glass appears coated with yellow film.

Replace dryer tube annually.

11. Data Evaluation, Calculations and Reporting

Procedures for data and record management must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP (1729). All records must be stored in such a manner as to be safe and accessible for at least 10 years.

The digestion batch bench sheets and other relevant laboratory notebooks must follow the specifications in all record keeping and document control practices.

Sample concentrations are calculated as follows:

Sample Concentration = ((Dc*Df)*Dv)/((Md*(S%/100)*1000))

Where:

Dc = digest Concentration in µg/L
Df = Dilution factor
Dv = Digest volume in mL
Md = Mass of sample digested in g
S% = percent solid of the sample (if reported on wet weight basis then percent solid = 100)

All results are reportable without qualification if digestion and analytical holding times are met, preservation (including cooler temperatures) are met, all QC criteria defined in the table below are met, and matrix interference is not suspected during digestion or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis

QC Parameter	Acceptance Criteria
Method Blank	< reporting limit
Laboratory Control Sample	80%-120% Recovery
Matrix Duplicate	≤ 20% RPD
Matrix Spike	80% - 120% Recovery
Matrix Spike Duplicate (if needed)	≤ 20% RPD, 80% - 120% Recovery

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9 outlines sample batch QC acceptance criteria. If non-compliant results are to be reported, the Metals Department Manager and/or the Laboratory Director, and the QA Manager must approve the reporting of the results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The instrument analyst or Department Manager performing the secondary analytical review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method

13.3 Instrument Detection Limits

The instrument detection limit (IDL) is the smallest signal above the background noise that an instrument can detect. The IDL can be calculated taking the average of the standard deviations for three measurements of a reagent blank solution analyzed on three analytical runs on three non-consecutive days. Seven consecutive measurements must be taken per day. The IDLs are

not required to go through sample digestion. Each measurement must be performed as though it were a separate sample.

14. Pollution Prevention and Waste Management

The *Hazardous Waste and Sample Disposal* SOP (1797) must be referenced for disposal of used standards, solvents, acids, reagents or other chemicals.

Once sample batches have completed digestion, the sample containers are stored in the metals lab and held for 30 days. If there is no sample remaining in the sample collection bottle, it may be rinsed and thrown away. It must be noted in the *Internal COC* that there is no sample remaining in the bottle, and the bottle was discarded.

Once satisfactory results have been generated, the digestates are held for 30 days, or longer if specified by a client contract, then discarded into a 55-gallon drum labeled “Corrosive Liquid”.

All reagent waste generated during digestion must be stored in satellite containers in the metals preparation laboratory.

Once the reagent waste satellite containers are full, they must be emptied into 55-gallon drums marked “Corrosive Liquid”.

Refer to Alpha’s Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1729 Document Control

SOP/1732 DL/LOD/LOQ Generation

SOP/1734 Control Limit Generation

SOP/1739 Demonstration of Capability Generation

SOP/1797 Hazardous Waste and Sample Disposal

SOP/2150 Microwave Assisted Acid Digestion of Sediments, Soils, Tissues and Waters

16. Attachments

None.

Mercury in Solid or Semisolid Waste
(Manual Cold-Vapor Technique)

Reference Method No.: **EPA 7471B**

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, February 2007.

1. Scope and Application

Matrices: Method 7471 is approved for measuring total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials. All samples must be subjected to an appropriate dissolution step prior to analysis. If this dissolution procedure is not sufficient to dissolve a specific matrix type or sample, then this method is not applicable for that matrix.

Definitions: Refer to Alpha Analytical Quality Manual.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Mercury Analyzer and in the interpretation of Mercury data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability..

2. Summary of Method

Prior to analysis, the solid or semi-solid samples must be prepared according to the procedures discussed in this method.

Method 7471, a cold-vapor atomic absorption method, is based on the absorption of radiation at the 253.7-nm wavelength by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.1 Method Modifications from Reference

Alpha analyzes only one 0.3g aliquot of sample. The original method does not address the automated instrument procedure. A reduced volume of sample is digested in disposable digestion tubes on a hot block digestor.

3. Reporting Limits

The reporting limit for this method is 0.08mg/Kg.

4. Interferences

Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/Kg of sulfide, as sodium sulfide, do not interfere with the recovery of added inorganic mercury in reagent water.

Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/Kg had no effect on recovery of mercury from spiked samples.

Samples high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL).

Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

Because mercury vapor is toxic, precaution must be taken to avoid its inhalation.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples may be collected in plastic or glass containers.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are stored under refrigeration at 4°C and analyzed as soon as possible after collection. The samples have a 28-day holding time from the time of collection.

7. Equipment and Supplies

7.1 Instrumentation:

Perkin Elmer FIMS 100 Atomic absorption spectrophotometer: Use instrument settings recommended by the manufacturer. The PE FIMS is designed specifically

for the measurement of mercury using the cold-vapor technique with BOC (background offset correction) performed by a survey scan prior to each sample introduction. PE S10 autosampler is coupled to the instrument.

Cetac M-6100 Atomic absorption spectrophotometer: Use instrument settings recommended by the manufacturer. This instrument employs a reference cell off-set correction and full automation through the CETAC software. A Cetac ASX-260 autosampler is coupled to the instrument.

Nippon Instrument model# RA-4300A analyzer with integrated 80 position autosampler:

The instrument adds a stannous chloride (II) solution to the sample post digestion, the divalent mercury ion (Hg2+) is reduced to zero-valent metallic mercury and turns into mercury gas by bubbling. $Hg2{+}{+}SnCl2 \rightarrow Hg0 \uparrow$

After removing the acid mist and water vapor generated by bubbling with an electronic cooling unit, the instrument measures the absorbance of mercury at 253.7 nm absorption wavelength. It measures the known mercury amount, creates a calibration curve, and then calculates the mercury amount from the absorbance.

7.2 Hot Block Digestor: Environmental Express, 54 position, capable of maintaining a temperature of 95°C +/-3°C.

7.3 Graduated cylinder. Rinse once with 50% Nitric Acid, then rinse with reagent water prior to use.

7.4 Volumetric Flasks, Class A, various volumes. Rinse once with 50% Nitric Acid, then rinse with reagent water prior to use.

7.5 Polypropylene Digestion Vessels: 50 mL volume, with plastic screw caps

7.6 Pump Tubing: Environmental Express, three stop and two stop tubing in various IDs.

7.7 PTFE membranes: Pall TF1000 disks

7.8 Dilution vials: 20mL capacity, used when making analytical dilutions.

7.9 Laboratory Wipes

7.10 Compressed Air

7.11 Whatman 41 or equivalent filter paper

7.12 Eppendorf pipets: Accurate means to make trace standards

8. Reagents and Standards

8.1 Reagent Water: Reagent water is DI water shown to be interference free. All references to water in this method refer to reagent water unless otherwise specified.

8.2 Aqua regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3.

8.3 Concentrated Nitric Acid, (HNO_3): Trace grade. Store at room temperature in the appropriately marked acid cabinet.

8.4 Concentrated Hydrochloric Acid, (HCl): Trace grade. Store at room temperature in the appropriately marked acid cabinet.

8.5 Reductant, Stannous Chloride in 3% HCl: This is the ***reductant*** for the Instrument. In a 1L volumetric flask, add 30mL concentrated trace grade HCl and 11g $SnCl_2 \cdot 2H_20$. Mix to dissolve the solid and bring to volume with reagent water. Store at room temperature, prepare as needed.

8.6 Carrier, Hydrochloric Acid, 3%: This is the ***carrier*** for the instrument. In a 1L volumetric flask, add 30mL concentrated trace grade HCl (Section 8.4). Bring to volume with reagent water. Store at room temperature, prepare as needed.

8.7 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 5 g of potassium permanganate in 100 mL of reagent water. Store at room temperature.

8.8 Sodium chloride-hydroxylamine hydrochloride solution: Dissolve 12 g of sodium chloride and 12 g of hydroxylamine hydrochloride in reagent water and dilute to 100 mL. Store at room temperature.

8.9 Mercury stock solution, 1000ppm: This solution is purchased commercially prepared with a certificate of analysis. Three solutions are purchased, each from a different source. Store at room temperature. Expires according to manufacturer's specifications.

8.9.1 10ppm Mercury Stock Standard: To a 100mL volumetric flask, add 5mL of concentrated HNO_3 and 1.0mL of 1000ppm Mercury Stock Solution (Section 8.9). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.

8.9.1.1 0.1ppm Mercury Working Stock / Matrix Spike Solution: To a 500mL volumetric flask, add 5mL of concentrated HNO_3 and 5 mL of 10ppm Mercury Stock Standard (Section 8.9.1). Bring to volume with reagent water. Store at room temperature. Expires one week from date of preparation.

8.9.2 10ppm Mercury ICV Stock Standard: To a 100mL volumetric flask, add 5mL of concentrated HNO_3 and 1.0mL of Mercury Stock Solution (Section 8.9, from an alternate source than that used in Section 8.9.1). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.

8.9.2.1 0.3ppm Mercury ICV Working Stock: To a 100mL volumetric flask, add 5mL of concentrated HNO_3 and 3.0mL of 10ppm Hg ICV Stock Standard (Section 8.9.2). Bring to volume with reagent water. Store at room temperature. Expires one week from date of preparation.

8.9.3 10ppm Mercury LCS Stock Standard: To a 100mL volumetric flask, add 5mL of concentrated HNO_3 and 1.0mL of 1000ppm Mercury Stock Solution (Section 8.9, from an alternate source than that used in Sections 8.9.1 and 8.9.2). Bring to volume with reagent water. Store at room temperature. Expires one month from date of preparation.

8.9.3.1 0.1ppm Mercury LCS Working Stock: To a 100mL volumetric flask, add 5mL of concentrated HNO_3 and 1.0mL of 10ppm Mercury LCS Stock Standard (Section

8.9.3). Bring to volume with reagent water. Store at room temperature. Expires one week from date of preparation.

8.10 SRM: Purchased from ERA in 300-500g lots.

9. Quality Control and Data Assessment

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

The Method Blank consists of the 0ppm standard as prepared in Section 10.1.2.1.1. Analyze one Method Blank per analytical batch of twenty samples or less. The Method Blank must be less than the Reporting Limit (RL). See Section 12.1 for corrective action.

9.1.1 PBS

A preparation blank is analyzed once per batch of twenty samples or less. It is prepared in the same manner as the 0ppm standard (Section 10.1.2.1.1). The PBS must be recovered within ± 0.2 µg/L.

9.1.2 ICB

The ICB is analyzed after the ICV, and is prepared in the same manner as the 0ppm standard (Section 10.1.2.1.1). The ICB must be recovered within ± 0.2 µg/L.

9.1.3 CCB

The CCB is analyzed after the CCV. It is prepared in the same manner as the 0ppm standard (Section 10.1.2.1.1). The CCB must be recovered within ± 0.2 µg/L.

9.2 Laboratory Control Sample (LCS)

The LCS is analyzed once per each analytical batch of twenty samples or less. It is prepared in the same manner as in Section 10.1.2.3. The LCS must be recovered within 80 – 120% of the true value. See Section 12.3 for corrective action.

9.3 Initial Calibration Verification (ICV)

The ICV is analyzed after the calibration curve. It is prepared in the manner specified in Section 10.1.2.2. The ICV must be recovered within ± 10% of the true value. See Section 12.2 for corrective action.

9.4 Continuing Calibration Verification (CCV)

The CCV is analyzed after every ten analytical samples. It is prepared in the same manner as the 5.0ppb calibration standard (Section 10.1.2.1.5). The CCV must be recovered within 20% of the true value. See Section 12.2 for corrective action.

9.5 Matrix Spike

Analyze one matrix spike per twenty or less analytical samples. The recovery of the matrix spike must be between 80 – 120%. Calculate percent recovery using Section 11.2.

If the recovery of the matrix spike is out of range, a post-analytical spike is analyzed. Prepare the post analytical spike by adding 1.25mL of 0.020ppm Calibration Standard

(Section 10.1.2.1.6) and 1.25mL of the sample digestate to a 15mL centrifuge tube for a final concentration of 0.0105mg/L. Analyze the post spike as outlined in Section 10.3.

Calculate the post spike concentration as follows:

Post Analytical Spike Sample Concentration (mg/L) =

[Sample Concentration (mg/L) x (0.5)] + 0.010mg/L

The percent recovery of the post-analytical spike must be between 75 – 125%.

See Section 12.4 for corrective action.

9.6 Laboratory Duplicate

Analyze one sample in duplicate per twenty or less analytical samples. The RPD between the sample and its duplicate must be ≤ 20% (as calculated in Section 11.3). See Section 12.5 for corrective action.

9.7 Method-specific Quality Control Samples

None

9.8 Method Sequence

- Sample preparation
- Sample digestion
- Standards preparation:
  - Calibration standards
  - ICV standard
  - LCS standard
- Standards digestion
- Analysis of calibration standards
- Generation of calibration curve
- Analysis of samples and standards:
  - ICV
  - ICB
  - analytical samples
  - CCV
  - CCB
  - analytical samples
  - CCV
  - CCB
  - etc.

10. Procedure

10.1 Equipment Set-Up

10.1.1 Sample preparation: Weigh a 0.3-g portion of untreated and homogenized sample and place in the bottom of a polypropylene digestion vessel. Record the weight in the laboratory notebook. NOTE: When preparing the batch, include one sample duplicate aliquot to be prepared in the same manner.

Add 2.5 mL of reagent water and 2.5 mL of aqua regia (Section 8.2). Heat 2 min on a hot block at 95°C +/-3°C. Cool; then add 15 mL reagent water, and 7.5 mL potassium permanganate solution (Section 8.7) to the digestion vessel. Wait 15 minutes to be sure the potassium permanganate is not exhausted (purple color disappears), if it does add additional potassium permanganate to all samples and QC until stable. Mix thoroughly and place in the hot block for 30 min at 95°C +/-3°C. Cool and add 3 mL of sodium chloride-hydroxylamine hydrochloride (Section 8.8) to reduce the excess permanganate.

CAUTION: Perform this addition under a hood, as Cl_2 could be evolved.

Bring up to a final volume of 50 mL with reagent water. Continue as described under Section 10.3.1.

10.1.2 Standard preparation: Standard preparation is performed each time samples are digested.

10.1.2.1 Calibration Standards

10.1.2.1.1 0 ppb: Add 10mL of reagent water to a polypropylene digestion vessel. This aliquot may be used for the CCB. Another separate aliquot is prepared for use as the ICB and the diluent for any samples with concentration greater than 90% the highest calibration standard used to determine the linear range.

10.1.2.1.2 0.5ppb: Add 10 mL of reagent water to a polypropylene digestion vessel. Using a volumetric pipet, add 0.25 mL of 0.1ppm Mercury Working Stock (Section 8.9.1.1) to the digestion vesel.

10.1.2.1.3 1.0ppb: Add 10 mL of reagent water to a polypropylene digestion vessel.. Using a volumetric pipet, add 0.5 mL of 0.1ppm Mercury Working Stock (Section 8.9.1.1) to the digestion vessel.

10.1.2.1.4 2.0ppb: Add 10 mL of reagent water to a polypropylene digestion vessel.. Using a volumetric pipet, add 1.0mL of 0.1ppm Mercury Working Stock (Section 8.9.1.1) to the digestion vessel.

10.1.2.1.5 5.0ppb/CCV: Add 10 mL of reagent water to a polypropylene digestion vessel.. Using a volumetric pipet, add 2.5 mL of 0.1ppm Mercury Working Stock (Section 8.9.1.1) to the digestion vessel.

10.1.2.1.6 10ppb: Add 10 mL of reagent water to a polypropylene digestion vessel.Using a volumetric pipet, add 5.0 mL of 0.1ppm Mercury Working Stock (Section 8.9.1.1) to the digestion vessel.

10.1.2.1.7 20ppb: Add 10 mL of reagent water to a polypropylene digestion vessel.Using a volumetric pipet, add 10.0 mL of 0.1ppm Mercury Working Stock (Section 8.9.1.1) to the digestion vessel.

10.1.2.2 ICV Standard, 3.0ppb: This standard is used for calibration verification.

Add 10.0 mL of reagent water to a digestion vessel. Using a volumetric pipet, add 0.5 mL of 0.3ppm Mercury Working Stock (Section 8.9.2.1) to the digestion vessel. Digest the ICV Standard as in Section 10.1.3.

10.1.2.3 LCS Standard, 1.0ppb: This standard is prepared and analyzed with each analytical batch.

Add 5.0mL of reagent water to a digestion vessel. Add 0.15g of SRM (Section 8.10). Digest the LCS Standard as in Section 10.1.3.

10.1.2.4 Matrix Spike, 0.001mg/L: Weigh two aliquots of the sample designated to be the batch matrix spike.

Add 10.0 mL of the reagent water to the digestion vessel containing the weighed sample aliquot. Add a 5 mL aliquot of 0.1ppm Mercury LCS Working Stock (Section 8.9.3.1). Digest the MS as in Section 10.1.3.

10.1.3 Standard Digestion:

To each standard (Sections 10.1.2.1 through 10.1.2.3), add 2.5 mL of reagent water and 2.5 mL of aqua regia (Section 8.2) and heat 2 min on the hot block at 95°C +/- 3°C. Allow the standard to cool; add 15 mL reagent water and 7.5 mL of $KMnO_4$ solution (Section 8.7) to each bottle and return to the hot block for 30 min. Cool and add 3 mL of sodium chloride-hydroxylamine hydrochloride solution (Section 8.8) to reduce the excess permanganate. Bring up to final volume of 50 mL with reagent water, continue as described in Section 10.3.3.

Note: Alternate volumes of standards may be made base on need as long as they are made with the same proportions as describe above.

10.2 Initial Calibration

Construct a calibration curve by plotting the absorbances of prepared standards (Section 10.1.2) versus micrograms of mercury. (See Section 11.1.) Determine the peak height of the unknown from the absorbance maxima on the spectrometer, and read the mercury value from the standard curve.

The curve correlation coefficient (cc) must be greater than or equal to 0.995 in order for the curve to be linear. If the correlation coefficient is less than 0.995, find and correct the problem. When the problem has been corrected, re-analyze either the previous standards or new standards. When the curve has generated an acceptable cc, then the analysis can continue with the ICV/ICB.

Analyze an Initial Calibration Verification Standard (ICV) (Section 9.3), an Initial Calibration Blank sample (ICB) (Section 9.1.2) at the start of the analytical run. The results for the ICV must be within 10% of the true value. If results are outside this range, refer to Section 12.2 for corrective action.

10.3 Equipment Operation and Sample Processing

10.3.1 Instrument Setup

10.3.1.1 Turn the instrument on. The autosampler will initialize itself.

10.3.1.2 Choose the instrument software from the desktop menu. The autosampler will initialize again.

FIMS 100 NOTE: The instrument must be turned on before the application is started. Otherwise, an error message will result.

10.3.1.3 Enter the appropriate fields for sample identification, and data storage.

10.3.1.4 Fill the carrier and reductant bottles.

10.3.1.4.1 The Carrier is 3% HCl (Section 8.6).

10.3.1.4.2 The Reductant is 1.1% $SnCl_2$ in 3% HCl (Section 8.5).

10.3.1.5 Allow the instrument to warm up while clearing samples. Samples that are cloudy or with particulate present after clearing must be filtered through Whatman 41 filter paper (Section 7.11) before analysis.

10.3.1.6 Place carrier uptake line and reductant uptake line.

10.3.1.7 Load carrier and reductant lines into pump magazines

10.3.1.8 Load the two waste lines into the pump magazines below the roller.

10.3.1.9 Lock the magazines into place.

FIMS100 only:

10.3.1.10 Remove the cap from the liquid/vapor separator and wipe dry with a Lab Wipe (Section 7.9). Compressed air (Section 7.10) through the vapor transfer line to dry it out.

10.3.1.11 Place a PTFE membrane (Section 7.7), rough side up, in the liquid/vapor separator; replace the cap and reattach the vapor transfer line to the sample absorption cell.

10.3.1.12 Adjust the gas flow by turning the black knob below the air flow meter to obtain a reading of just over 50.

10.3.2 Calibration and Sample Analysis

10.3.2.1 The instrument will now run the calibration standards; verify a CC of 0.995 or better before proceeding with the ICV and ICB. Ten analytical samples, a CCV and CCB, ten analytical samples, CCV, CCB, etc. The CCBs and CCVs must be recovered within the proper ranges (Sections 9.4 and 9.1.3) for analysis to continue.

10.3.2.2 If the sample result is beyond 90% of the concentration of the highest point on the calibration curve or LDR study used to establish the linear range, dilute the sample extract with a portion of one of the prepared blanks (ICB, CCB or PBS) to produce an analytical result that is within the range.

10.3.3 Instrument Shut Down

10.3.3.1 When analysis is complete place reagent uptake lines in a beaker of DI water.

10.3.3.1.1 Continue to run the pumps for several minutes to flush reagents out of the lines.

10.3.3.1.2 Continue to run the pumps for several more minutes to flush the lines thoroughly with DI water.

10.3.3.2 Pull the reagent uptake lines out of the DI water beaker to allow the pump to draw air completely through the lines.

10.3.3.3 Unlock the top and bottom pump magazines and remove tubing from the magazines.

10.3.3.4 Exit from the software application.

Note: This procedure must be followed every time the instrument is shut down to prevent water or reagents from being draw back into the instrument which can cause severe damage.

10.3.3.4.1 Dump the samples and instrument waste in the Metals/WetChem waste drum located in the transfer room only after all data has been processed through final review and package generation.

10.4 Continuing Calibration

After every 10 samples, analyze a Continuing Calibration Verification Standard (CCV), and a Continuing Calibration Blank sample (CCB). Determine the concentrations from the calibration curve. The results for the CCV must be within 20% of the true value.

10.5 Preventive Maintenance

Preventative maintenance is conducted per the manufacturer's instructions. All preventative maintenance is recorded in the Instrument Maintenance Logbook.

11. Data Evaluation, Calculations and Reporting

11.1 Calculate Mercury Concentration From the Daily Calibration Curve

The curve is generated utilizing a straight-line equation defined as:

$$A = k_1 + k_2C$$

Where:

A = Average peak height of the sample/standard integrations
C = Sample/Standard Concentration, µg/L
k_1 = y-intercept
k2 = slope

The instrument will plot peak height against concentration (µg/L). The result is generated in µg/L. This value is divided by 1000 to convert the units to mg/L. The mg/L units are converted to mg/Kg by multiplying by L/Kg. A dilution factor (DF) is applied if necessary. The Result is then divided by the % Total Solids prior to release to the client.

$$\text{Result, mg/Kg} = (\text{concentration, µg/L}) \times (1\text{mg}/1000\mu\text{g}) \times (\text{DF}) \times (\text{L/Kg})$$

Where:

$$\text{L/Kg} = \frac{\text{Final volume of digestate, in L}}{\text{Weight of original sample, in Kg}}$$

11.2 Calculate Percent Recovery for the Matrix Spike corrected for concentrations measured in the unfortified sample. Percent recovery is calculated using the following equation:

$$\%\ \text{Recovery} = \frac{(Cm - C)}{s} \times 100$$

Where:

Cm = measured Mercury in the fortified sample
C = measured native mercury sample concentration
S = concentration equivalent of spike added to sample

11.3 Calculate the Relative Percent Difference (RPD) for each Duplicate of the initial quantitated concentration (IC) and duplicate quantitated concentration (Dc) using the following formula:

$$RPD = \frac{|(IC - Dc)|}{\{(IC + Dc) / 2\}} \times 100$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 Method Blank Failure

When a prep blank mercury concentration is ≥10% of the mercury concentration determined for any associated sample, or is greater than 2.2x the MDL value (whichever is greater), the entire batch associated with the prep blank must be redigested.

12.2 ICV / CCV Failure

If the ICV %Recovery is outside of acceptance criteria, analysis is terminated until the problem is found and corrected. If the CCV %Recovery is outside of acceptance criteria, all samples analyzed since the last acceptable CCV must be reanalyzed following correction of the problem.

12.3 LCS Failure

If the LCS is not recovered within acceptance criteria, the associated batch and another LCS must be redigested (Section 10.1).

12.4 Matrix Spike/Post Digestion Spike Failure

If the recovery of the matrix spike is outside of the acceptance criteria of 80 – 120%, a post digestion spike is performed (Section 9.5). If the post digestion spike is beyond 75 – 125%, the sample and its spike are redigested (Section 10.1).

12.5 Duplicate Failure

If the RPD between the sample and its duplicate is greater than 20%, visually evaluate the sample matrix. If the sample matrix appears clean, the sample and its duplicate are removed from the batch and redigested (Section 10.1). If the matrix appears problematic, the sample digestate may be diluted and reanalyzed, or a narrative included with the data to explain the matrix problem.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

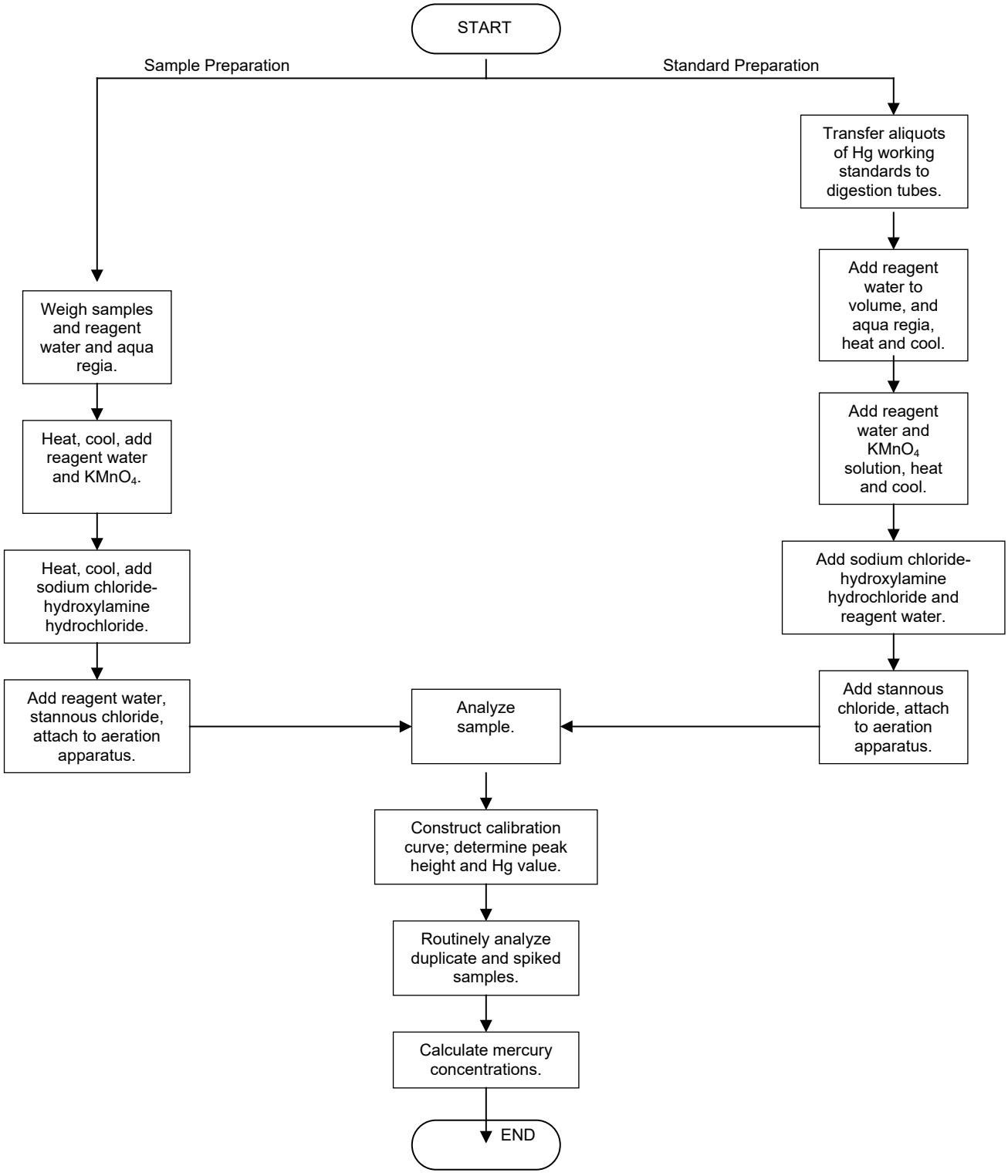
SOP/1797 Waste Management and Disposal SOP

16. Attachments

FIGURE 1: Flow Chart for Method 7471B

Figure 1

Method 7471B Flow Chart



Mercury Determination in Solids by Cold Vapor Atomic Absorption Technique (CVAA)

References: USEPA, "Method 7471B Mercury in Solid or Semisolid Waste (Manual Cold-Vapor Technique)," in Test Methods for Evaluating Solid Waste, SW846, Revision 2, February 2007

1. Scope and Application

Matrices: This cold-vapor atomic absorption method is applicable to the determination of total mercury (organic and inorganic) in solid samples (soils, sediments and sludges).

Definitions: Refer to Alpha Analytical Quality Manual.

This digestion and analytical procedure measures total mercury (organic & inorganic) in soil, sediment and sludge samples. Mercury can accurately be determined in the range of 0.005 to 0.25 mg/Kg for solid samples that do not require dilution.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the AA and in the interpretation of AA data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Parameter	CAS
Mercury	7439-97-6

2. Summary of Method

A 0.5 to 1.5 gram aliquot of sample is digested in an acidic, oxidizing solution (consisting of Hydrochloric acid, nitric acid, and potassium permanganate) and heated for a total of 32 minutes on a hotplate at 95°C to convert all forms of mercury to inorganic Hg (II). Once the samples have been digested, they are ready for analysis by the cold-vapor atomic absorption technique (CVAA).

The CVAA technique is based on the absorption of radiation at 253.7-nm by mercury vapor. The addition of the hydroxylamine-hydrochloride solution to the digestate, reduces the excess potassium permanganate without reducing the mercury and transforms the oxidized mercury to the insoluble and non-volatile $HgCL_2$. Mercury is then reduced with stannous chloride from $HgCL_2$ to elemental mercury, [Hg (0)]. The elemental mercury is aerated as mercury vapor from the digestate in a closed system where the vapor passes through a cell positioned in the light path of an atomic absorption spectrometer. Absorbance is measured as a function of mercury concentration based on the peak height measured.

2.1 Method Modifications from Reference

Method 7471B specifies use of 300 mL BOD bottles for sample and standard preparation and a final volume of approximately 100 mL. This method has been modified for use of 50 mL plastic digestion tubes. Reagent volumes have been reduced proportionately.

3. Reporting Limits

The mercury solid RL is 0.005 mg/Kg.

4. Interferences

4.1 Potassium permanganate is added to eliminate potential interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of inorganic mercury from distilled water.

4.2 High concentrations of copper may cause interference; however copper concentrations as high as 10 mg/L have no effect on the recovery of spiked mercury samples.

4.3 Additional portions of potassium permanganate may need to be added until the purple color persists due to interference from chloride or organic matter. During the oxidation step, chlorides are converted to free chlorine which absorbs radiation at 253.7-nm. Care must be taken to ensure that free chlorine is absent before mercury is reduced to its elemental state and mercury vapor is swept into the cell. This may be accomplished by using additional portions of sodium chloride-hydroxylamine hydrochloride solution.

Note: Chloride interference is not as common in solid matrices as aqueous matrices.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. All digestion and analysis procedures must be conducted in a laboratory exhaust hood.

5.2 Care must be taken when handling all samples, digestates, and standards since they are preserved to a pH <2. In addition, the digestate solutions contain strong oxidizing reagents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

A minimum of 10.0 grams of sample must be collected in a glass jar.

6.2 Sample Preservation

The samples must be refrigerated and maintained at 4°±2°C until digestion and analysis.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

All solid samples must be analyzed within 28 days from date of collection.

7. Equipment and Supplies

7.1 PSA 10.035 MILLENNIUM MERLIN MERCURY ANALYZER

7.1.1 Lamp: Mercury, low pressure

7.1.2 HP Laser Jet P2015dn or equivalent, compatible with AA and AF software

7.1.3 Millennium AAS Detector:

7.1.4 Pumps: Two variable speed and independently controlled peristaltic pumps to deliver reagent and sample solutions as well as remove waste.

7.1.5 Drying Tube: Gas liquid separator and dryer tube to remove mercury vapor from solution and prevent water vapor from entering the absorption cell.

7.2 Disposable Digestion tubes, 50mL

7.3 Electric Hot Plate- Adjustable and capable of maintaining a temperature of 90-95°C equipped with graphite carbon blocks that each have 36 positions to hold sample tubes. Glassware - Assorted Class A: volumetric flasks, beakers, graduated cylinders and pipettes of appropriate sizes for preparing reagents, standards, and measuring sample volumes.

7.4 Air Displacement pipettes: Digital pipettes capable of delivering volumes ranging from 0.1 to 5000 µL with an assortment of high quality disposable pipette tips.

7.5 Analytical balance: Capable of accurate measurement to the nearest 0.0001 g

7.6 Top-loading balance: Capable of accurate measurement to the nearest 0.01 g

8. Reagents and Standards

ACS Trace Metal grade chemicals shall be used in all tests. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.

Solutions below expire six months from preparation unless noted.

Stock standard solutions are stored in a cabinet, out of direct light

8.1 Deionized (DI) water: The Barnstead NANO-pure system provides Type I water used in the preparation of samples and standards.

8.2 Concentrated hydrochloric acid (HCl). Fisher trace metals grade or equivalent. *(Digestion reagent).* Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.3 Concentrated nitric acid (HNO_3). Fisher trace metals grade or equivalent. *(Digestion reagent).* Lots should be checked for purity prior to use and the results stored in a reagent check log book.

8.4 Aqua Regia. Mix concentrated HCl and HNO_3 at a ratio of 3:1. *(Digestion reagent)*

8.5 Potassium Permanganate ($KMnO_4$), Fisher P279-500 or equivalent

8.6 5% Solution (w/v) of Potassium Permanganate Solution. Dissolve 50g of $KMnO_4$ in deionized water and dilute to 1L. *(Digestion reagent)*

8.7 Hydroxylamine Hydrochloride ($NH_2OH \bullet HCl$). Fisher H330-500, or equivalent. *(Analytical reagent)*

8.8 Stannous Chloride ($SnCl_2 \bullet 2H_2O$). Fisher T142-500, or equivalent. *(Analytical reagent)*

8.9 Stannous Chloride Reduction Solution (2%). Add 20 g of $SnCl_2 \bullet 2H_2O$ to 100mL of concentrated HCl and dilute to 1L with deionized water. Prepare fresh daily. *(Analytical reagent)*

8.10 HCl Rinse Solution (10%). Dilute 900mL of concentrated HCl to 9L with deionized water. *(Analytical reagent)*

8.11 Sodium Chloride (NaCl). Fisher S271-3, or equivalent. *(Analytical reagent, added to the Hydroxylamine Hydrochloride Solution below)*

8.12 Sodium Chloride-Hydroxylamine Hydrochloride Solution. Dissolve 120g of NaCl and of $NH_2OH \bullet HCl$ in deionized water and dilute to 1L. *(Analytical reagent)*

8.13 1000mg/L Mercury Stock Standard from two different sources. One source is used to prepare the calibration curve (Ultra Scientific ICP-080) and the other is for verification of the calibration curve (Inorganic ventures CGHG1-1). Use the vendor's expiration date. *(Standards preparation)*

8.14 Mercury Working Standards. Three mercury working standards are prepared from successive dilutions of the mercury stock standard and are used to prepare the calibration curve. Acidity of the working standards must be maintained at 1% HCl acid. Add 1mL of concentrated HCl to a 100mL volumetric flask. A 1.0mg/L working standard is prepared by adding 0.1mL of the 1000mg/L Ultra Scientific stock standard to a final volume of 100mL. This standard is diluted 1:10 to make a 0.1mg/L working standard and 1:100 to make a 0.01 mg/L working standard. The 0.1 and 0.01mg/L working standard is diluted prior to preparation of the calibration standards. The 1.0mg/L working standard is prepared monthly. See Section 10.0, for specific details regarding working standard preparations. *(Standards preparation)*

8.15 1.0mg/L LCS and Matrix Spike Solution. Prepare from the 1000 mg/L stock standard (8.13) by adding a 0.1mL aliquot to a final volume of 100mL. This solution is stable for 1 month. *(Spiking Solution)*

8.16 Solid Matrix LCS Standard: Purchased from Environmental Resource Associates (ERA), Catalog #540 Metals in Soil QC Standard, or equivalent.

8.17 Solid Matrix Blank Sample: Purchased from Environmental Resource Associates (ERA), Catalog #058 Blank Soil QC Sample, or equivalent.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

9.1.1 A method blank must be analyzed once per every 20 samples or per mercury digestion batch, whichever is more frequent.

9.1.2 Mercury concentrations must not be detectable in the method blank at values greater than the reporting limit.

9.1.3 Corrective Action: Digestion of the method blank and all associated samples must be performed until the method blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Section Head if the samples associated with the method blank are non-detect for mercury or if the concentration of mercury is greater than 10x the blank level in the samples. In such cases, the sample results are accepted without corrective action for the high method blank result. The client must be notified in the project narrative associated with the sample results.

9.2 Laboratory Control Sample (LCS)

9.2.1 The LCS is digested along with the samples. A LCS must be digested and analyzed once per every 20 samples or per mercury digestion batch, whichever is more frequent.

9.2.2 The acceptable recovery QC limits are 80%-120%. The solid LCS recovery limits are continuously monitored and documented in-house through control charts. The SOP *Control Limit Generation* (1734) provides details explaining how control charts are generated and used for quality control.

9.2.3 Corrective Action: May repeat analysis once to see if an analytical error has occurred. If the LCS recovery is still out of control, re-digest and re-analyze the LCS and all associated samples. Samples cannot be analyzed until an acceptable LCS is obtained. Exceptions may be made with approval of the Section Head if the samples associated with the out of control LCS are also associated with a matrix spike that is in control. This is an acceptable measure of accuracy of the digestion and analytical procedures. An explanation of this out of control LCS recovery must be included in the project narrative to the client and the sample data reported noting the acceptable MS results as batch QC.

9.3 Initial Calibration Verification (ICV)

9.3.1 Initial Calibration Curve

9.3.1.1 The correlation coefficient (r) of the initial calibration curve must be ≥0.995. The initial calibration curve consists of five standards and a blank.

9.3.1.2 Corrective Action: If the correlation coefficient does not exceed 0.995, individual standards or the entire curve may be re-analyzed until the correlation coefficient is in control. If the correlation coefficient is still not in control, the initial calibration curve must be re-prepared and re-analyzed until the correlation coefficient is acceptable.

9.3.2 Initial Calibration Verification (ICV) Check Standard

9.3.2.1 The initial calibration verification check standard must be from a second source or lot number to verify the accuracy of the standard curve. The concentration of the ICV is at approximately the mid-level of the calibration curve.

9.3.2.2 The acceptable recovery QC limits for the ICV is 90%-110%.

9.3.2.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the ICV still exceeds the control limits, re-calibrate the instrument.

9.3.3 Initial Calibration Blank (ICB)

9.3.3.1 An ICB must be analyzed immediately following the ICV.

9.3.3.2 The ICB concentration must not be greater than the reporting limit.

9.3.3.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the ICB still exceeds the control limits, re-calibrate the instrument and re-analyze a fresh blank.

9.4 Continuing Calibration Verification (CCV)

9.4.1 Continuing Calibration Verification (CCV) Check Standard

9.4.1.1 A CCV must be analyzed at a minimum of every 10 samples and at the close of an analytical sequence. The concentration of the CCV is at approximately the mid-level of the calibration curve. This standard monitors instrument performance throughout the duration of the analytical run.

9.4.1.2 The acceptable recovery QC limits for the CCV is 90%-110%.

9.4.1.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the CCV still exceeds the control limits, re-calibrate and re-analyze all samples since the last acceptable CCV.

9.4.2 Continuing Calibration Blank (CCB)

9.4.2.1 A CCB must be analyzed immediately after every CCV.

9.4.2.2 The CCB concentration must not be greater than the reporting limit.

9.4.2.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the CCB still exceeds the control limits, re-calibrate and/or re-analyze a fresh blank. All samples associated with the out of control CCB

must be re-analyzed (since the last acceptable CCB). Exceptions may be made with approval of the Section Head if the samples associated with the out of control CCB are non-detect for mercury or if sample concentrations for the affected metals are greater than 10x the blank levels. In such cases, the sample results are accepted without corrective action for the high CCB and the client is notified in a project narrative associated with the sample results.

9.5 Matrix Spike

9.5.1 A matrix spike must be performed once per 20 samples (5% frequency). When project specifications dictate, a Matrix Spike Duplicate (MSD) may also need to be performed at the same frequency as the MS.

9.5.2 The acceptable recovery QC limits is 80%-120% for the solid MS/MSD. For MCP projects the recovery QC limits are 75%-125% for the solid MS/MSD. Calculate MS Recovery as follows:

$$\% \text{ Recovery} = \frac{MS - R1}{MS_{\text{True Value}}} \times 100$$

9.5.3 Calculate the %RPD as in 12.3.2 above when analyzing a MS/MSD pair. The acceptable %RPD is $\leq$ 20%. Both the solid recovery limits and %RPD are continuously monitored and documented in-house through control charts.

9.5.4 Corrective Action: Repeat analysis once to see if an analytical error has occurred. If the % recovery, or %RPD, still exceed the control limits and the LCS is compliant; include a project narrative with the results to the client noting that there may be potential matrix effects on the accuracy or precision of the affected mercury results as evidenced by matrix spike %recovery or %RPD outside of the QC limits.

9.6 Laboratory Duplicate

9.6.1 Duplicate analyses (matrix duplicate) must be performed once per 20 samples (5% frequency).

9.6.2 Acceptable relative percent difference (RPD) for duplicate analysis is $\leq$ 20 % for solid matrices. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

The RPD limits are continuously monitored and documented in-house through control charts.

9.6.3 Corrective Action: Repeat analysis once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the affected mercury results as evidenced by the matrix duplicate RPD exceedance.

9.7 Method-specific Quality Control Samples

9.7.1 Serial Dilution (SD)

9.7.1.1 Serial dilution of one sample per batch of 20, at a 1:5 dilution, is useful in the assessment of matrix effects, and must be analyzed as needed to assess a new or unusual matrix.

9.7.1.2 The acceptable agreement between the undiluted and diluted results is ± 10%.

9.7.1.3 Corrective Action: May repeat analysis once to see if an analytical error has occurred. If the precision between the results still exceeds the control limits, include a project narrative with the results to the client noting that there may be potential matrix effects on the precision of the affected mercury results as evidenced by the SD outside of the QC limits.

9.7.2 CRA Standard Check Sample

9.7.2.1 The CRA Check sample is typically analyzed as part of the initial calibration curve, however, project specifications may require this to be analyzed at the beginning of the analytical sequence after the ICV and ICB. This solution verifies the accuracy at the low end of the calibration curve.

9.7.2.2 Results for mercury in the CRA solution must be within 70-130% recovery criteria or within project specified acceptance limits.

9.7.2.3 Corrective Action: May repeat analysis once to see if an analytical error occurred. If the CRA solutions still exceed the control limits, re-calibrate and/or re-analyze a fresh CRA solution. All samples associated with the out of control mercury CRA must be re-analyzed.

***NOTE**: The CRA analysis is not preformed if a calibration standard is prepared at the reporting limit and included in the initial calibration curve. The calibration curve must meet the criteria for linearity.*

9.8 Method Sequence

Initial calibration curve (STD0, STD1, etc., to STD5)
ICV
ICB
CRA (if required by the project)
Method Blank (The assigned LIMS batch name)
LCS (The assigned LIMS batch name)
Sample analysis (samples 1-5 including SD, MS and MD samples)
CCV
CCB
Sample analysis (6-15)
CCV
CCB

The CRA analysis is not preformed if a calibration standard is prepared at the reporting limit and included in the initial calibration curve. The calibration curve must meet the criteria for linearity.

10. Procedure

10.1 Equipment Set-up

10.1.1 Digestion Procedure Using Block Digestion

10.1.1.1 Weigh a 0.5 to 1.5g aliquot of well-homogenized sample into the digestion tube. A nominal weight of 0.5-0.6g of each sample is generally sufficient; however an increased amount of wet sample may be used, and depends upon the required reporting limits for the project. See the Section Head or the Project Manager if there are any questions regarding the reporting limits required or the amount of sample to be used.

10.1.1.2 A method blank, LCS, matrix spike sample, and matrix duplicate sample must be digested with each analytical batch at the frequency listed in Section 9, Quality Control. In some instances, the client may request matrix spike duplicate analysis as an alternative to matrix duplicate analysis.

If required, and a blank solid material of a similar matrix type of high enough purity can be obtained to meet required reporting limits, a solid material may be used in the Method Blank preparation to matrix match QC samples and field samples.

If required, a solid LCS may be prepared to matrix match QC samples and field samples.

10.1.1.3 The calibration standards are digested along with the samples.

10.1.1.4 Add 5mL of ASTM Type II laboratory reagent ASTM Type II laboratory reagent and 5 mL Aqua Regia (3:1 HCL to HNO3) to sample and calibration tubes.

10.1.1.5 Spike Laboratory Control Sample and Matrix Spike sample with 0.125mL of the Hg 1 mg/L spiking solution (see 8.14).

10.1.1.6 Heat samples and calibration curve to 95°C +/- 3°C on heated Blocks for 2 minutes, remove from heat, let cool to room temperature, then add 15mL ASTM Type II laboratory reagent grade water.

10.1.1.7 Add 7.50mL of 5% Potassium Permanganate solution, wait 15 minutes.

10.1.1.8 If the purple color dissipates, add additional portions of $KMnO_4$ solution until the purple color persists. Note the additional amount of $KMnO_4$ on the preparation bench sheet. Heat for an additional 30 minutes on heated blocks.

10.1.1.9 Allow the samples and standards to cool. Add 3.0mL of NaCl-NH_2OH•HCl solution to each digestion tube to reduce the excess permanganate. Add deionized water to bring the final volume to 50mL.

10.1.1.10 The digestates are now ready for analysis. The batch must be transferred to the metals instrument room with the project folder, the preparation paperwork and the metals preparation checklist.

10.2 Initial Calibration

A series of five calibration standards are prepared by pipetting suitable volumes of standard solution into 50mL centrifuge tubes. The preparation date of these standards, the initials of the analyst, the lot number of the source material, stock concentrations, volumes used, final volumes, final concentrations, and manufacturer, and expiration date must be recorded in the *Mercury CVAF, Mercury CVAA and Amalgam Working Standards Preparation Logbook*. The

PSA MILLENIUM MERLIN is calibrated using a multi-point calibration curve consisting of a blank and six standards.

10.2.1 The working standards are prepared as follows:

10.2.1.1 Mercury Stock Solution (1000mg/L). See Section 8.13. Check expiration date to ensure the standard has not expired.

10.2.1.2 Mercury Working Standard (1.0mg/L). See Section 8.14. Check expiration date to ensure the standard has not expired.

10.2.1.3 Diluted Mercury Working Standard (0.1mg/L): Dilute 0.1mL of working standard (10.2.1.2) to 1.0mL. This solution must be prepared fresh daily.

10.2.1.4 Diluted Mercury Working Standard (0.01mg/L): Dilute 0.1 mL of working standard (10.2.1.3) to 1.0mL. This solution must be prepared fresh daily.

10.2.1.5 The calibration standards may be prepared in 50mL screw cap plastic digestion tubes. Each tube must contain approximately 25mL of deionized water, which is then spiked with the volume listed in the table below. Prepare a six-point mercury calibration curve daily as follows

Volume of the Working Standard (mL)	Concentration of Working Standard (mg/L)	Concentration of Calibration Standard (µg/L)
0	None	Blank
0.025	0.1	0.05
0.05	0.1	0.1
0.25	0.1	0.5
0.1	1	2
0.25	1	5

10.2.1.6 Digest all standards along with the samples as described in Section 10.1, Equipment Set-up.

10.2.2 The PSA MILLENIUM MERLIN computer will calculate a linear regression, correlation coefficient ("r"), and slope of standard curve. The correlation coefficient must be greater than $\geq$ 0.995 for linearity. The linear regression is computed on a multipoint calibration.

10.3 Equipment Operation and Sample Processing

10.3.1 Before turning the instrument on, unplug and power off on rear panel. Connect the millennium AAS detector, plug back in and power on the back panel. The pump windings must be inspected for wear, rotated and/or changed. Secure the pump windings to the pump. Turn the gas on to 52 P.S.I. Turn on the mercury analyzer and the computer. Allow the instrument to warm up for 30 to 60 minutes.

10.3.2 Place the rinse line in the 10% HCl rinse solution. The $SnCl_2$ line from the instrument must be immersed into the 2% stannous chloride reduction solution.

10.3.3 All standard and sample information is entered into a sample information, or instrument sequence file. The following information is entered: sample ID, dilution and units. A hard copy of the sequence file is printed, given a page number, and becomes part of the permanent instrument run log. After one month, or more if suitable, of mercury analyses, the sequence printouts are bound and given an internal log identification number.

10.3.4 Place the calibration standards and samples onto the autosampler rack in the appropriate positions according to the sample information sequence file.

10.3.5 Begin the run sequence with the initial calibration curve and evaluate it for QC acceptance. Evaluate the ICV, ICB, CRA, Method Blank and LCS for QC acceptance prior to the analysis of the samples. The typical analytical sequence is listed in Section 9.8.

10.3.6 The instrument detection limits (IDLs) may be performed if required by the regulatory program or the client.

10.3.7 Dilute and reanalyze any samples that exceed the linear calibration range for mercury. Report the mercury result from the dilution analysis.

10.3.8 The *Mercury Data Review Checklist* must accompany all acceptable mercury results for primary and secondary review.

10.4 Continuing Calibration

10.4.1 The same time must be elapsed between CCVs and CCBs as is allowed between samples. Analyze the continuing calibration verification (CCV) and the continuing calibration blank (CCB) after each 10 samples and at the end of the analytical sequence.

10.5 Preventive Maintenance

Pump windings should be inspected for wear on a daily basis and replaced when they appear flattened (approximately once per month depending on use). The gas-liquid separator should be cleaned periodically when it appears coated with a yellow film. Add 30% KOH solution and let set for approximately 30 minutes. Rinse with DI water. The dryer tube should be replaced approximately once per year. (Part number H003S001 PS Analytical)

11. Data Evaluation, Calculations and Reporting

11.1 The mercury results are calculated by the following equation:

Solid:

$$\text{Mercury result in mg/Kg} = \frac{C \times B}{A} \times DF \times 1000 \times \frac{100}{\%\ \text{solid}}$$

Where:

A = Initial sample weight in grams, typically 1 gram
B = Digestion final volume in mL, typically 50 mL
C = Concentration of sample from instrument read-out in µg/L
DF = Dilution Factor

11.2 All mercury results must be reported to three significant figures.

11.3 The primary analyst does the batching and data upload into the LIMS system.

11.4 All solids including soils, sediments, and sludges must be reported on a dry-weight basis.

11.5 A secondary review is performed on all.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9, Quality Control, defines the corrective actions that must be taken in instances where QC outliers exist.

13. Method Performance

13.1 Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1797 Waste Management and Disposal SOP

16. Attachments

None.

Inductively Coupled Plasma - Atomic Emission Spectrometry

Reference Method No.: **Method 6010D** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update V, July, 2014.

SM 2340B, Hardness by Calculation, Standard Methods for the Examination of Water and Wastewater, APHA-AWWA-WPCF, 21st Edition, 1997.

1. Scope and Application

Matrices: Digestates from all matrices.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Inductively coupled plasma-atomic emission spectrometry (ICP-AES) determines trace elements, including metals, in solution. The method is applicable to all of the elements listed in Table 1. All matrices, excluding filtered groundwater samples but including ground water, aqueous samples, TCLP and EP extracts, industrial and organic wastes, soils, sludge, sediments, and other solid wastes, require digestion prior to analysis. Groundwater samples that have been prefiltered and acidified will not need acid digestion unless chemical interferences are suspected. Samples which are not digested are matrix matched with the standards. Refer to Metals Preparation SOPs for the appropriate digestion procedures.

Table 1 lists the elements for which this method is applicable. Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Table 1 lists the recommended analytical wavelengths for the elements in clean aqueous matrices. Table 3 lists the Reported Detection Limits. The reported detection limit data may be used to estimate instrument and method performance for other sample matrices. Elements other than those listed in Table 1 may be analyzed by this method if performance at the concentration levels of interest (see Section 9) is demonstrated.

Users of the method should state the data quality objectives prior to analysis and must document and have on file the required initial demonstration performance data described in the following sections prior to using the method for analysis.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is made by one of the following laboratory personnel before performing the modification: Area Supervisor, Metals Manager, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

Use of this method is restricted to spectroscopists who are knowledgeable in the correction of spectral, chemical, and physical interferences described in this method. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

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2. Summary of Method

Prior to analysis, samples must be solubilized or digested using appropriate Sample Preparation Methods. When analyzing groundwater samples for dissolved constituents, acid digestion is not necessary if the samples are filtered and acid preserved prior to analysis.

This method describes multielemental determinations by ICP-AES using sequential or simultaneous optical systems and axial or radial viewing of the plasma. The instrument measures characteristic emission spectra by optical spectrometry. Samples are nebulized and the resulting aerosol is transported to the plasma torch. Element-specific emission spectra are produced by a radio-frequency inductively coupled plasma. The spectra are dispersed by a grating spectrometer, and the intensities of the emission lines are monitored by photosensitive devices. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. In one mode of analysis the position used must be as free as possible from spectral interference and must reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in Section 4.0 must also be recognized and appropriate corrections made; tests for their presence are described in Section 9.7. Alternatively, users may choose multivariate calibration methods. In this case, point selections for background correction are superfluous since whole spectral regions are processed.

This SOP includes the manual calculations for Total Hardness and Calcium Hardness, according to SM 2340B.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Refer to Table 3 for method Reporting Limits.

4. Interferences

4.1 Spectral

Spectral interferences are caused by background emission from continuous or recombination phenomena, stray light from the line emission of high concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

4.1.1 Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for

routine measurement must be free of off-line spectral interference (interelement or molecular) or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans must be included in the correction algorithm. Off-line spectral interferences are handled by including spectra on interfering species in the algorithm.

4.1.2 To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line interelement spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a concentration near the upper analytical range limit.

4.1.3 Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for interelement contributions. Instruments that use equations for interelement correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply interelement correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.

4.1.4 When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that As is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Al. 100 mg/L of Al would yield a false signal for As equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Al would result in a false signal for As equivalent to approximately 0.13 mg/L. The user is cautioned that each instrument may exhibit somewhat different levels of interference. The interference effects must be evaluated for each individual instrument since the intensities will vary.

Major known interferences are Fe, Al, Ca, Mg, V, Ni, Cu, and Cr. To minimize any of these interferences, every analyte is analyzed on each instrument at or near its linear range and corrected for these interferences. This is done on an annual basis, and data is kept on file.

4.1.5 Inter-element corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear must be avoided when practical. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users must not forget that some samples may contain uncommon elements that could contribute spectral interferences.

4.1.6 The interference effects must be evaluated for each individual instrument whether configured as a sequential or simultaneous instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The analyst is encouraged to utilize a computer routine for automatic correction on all analyses.

4.1.7 The primary wavelength for each analyte is based upon the instrument manufacturer's recommendations. An alternate wavelength is chosen if there is an indication of elevated background or overlap of another spectral wavelength. The wavelength for each analyte must be as free from interferences as possible.

4.1.8 If the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution must fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change must be determined and corrected and the correction factor updated. The interference check solutions must be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.

4.1.9 When inter-element corrections are applied, their accuracy must be verified, daily, by analyzing the spectral interference check solution. The correction factor or multivariate correction matrices tested on a daily basis. All inter-element spectral correction factors or multivariate correction matrices are verified and updated when an instrumentation change, such as in the torch, nebulizer, injector, or plasma conditions occurs. The standard solution must be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

4.1.10 When inter-element corrections are <u>not</u> used, verification of absence of interferences is required.

4.1.10.1 One method is to use a computer software routine for comparing the determinative data to limits, files for notifying the analyst when an interfering element is detected in the sample at a concentration that will produce either an apparent false positive concentration, (i.e., greater than) the analyte

instrument detection limit, or false negative analyte concentration, (i.e., less than the lower control limit of the calibration blank defined for a 99% confidence interval).

4.1.10.2 Another method is to analyze an Interference Check Solution(s) which contains similar concentrations of the major components of the samples (>10 mg/L) on a continuing basis to verify the absence of effects at the wavelengths selected. These data must be kept on file with the sample analysis data. If the check solution confirms an operative interference that is >20% of the analyte concentration, the analyte must be determined using (1) analytical and background correction wavelengths (or spectral regions) free of the interference, (2) by an alternative wavelength, or (3) by another documented test procedure.

4.2 Physical

Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, they must be reduced by diluting the sample, using a peristaltic pump, use of an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also, it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers. The test described in Section 10.3.4.1 will help determine if a physical interference is present.

4.3 Chemical

Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique, but if observed, can be minimized by careful selection of operating conditions (incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. Additionally, if filtered samples are found to have an organic or sulfur like odor they are processed by heating after the addition of the acids to matrix match. Chemical interferences are highly dependent on matrix type and the specific analyte element.

4.4 Memory

Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the element and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences must be recognized within an analytical run and suitable rinse times must be used to reduce them. The rinse times necessary for a particular element must be estimated prior to analysis. This may be achieved by aspirating a standard containing elements at a concentration ten times the usual amount or at the top of the linear dynamic range. The aspiration time for this sample must be the same as a normal sample analysis period, followed by analysis of the rinse blank at designated intervals. The length of time required to reduce analyte signals to within a factor of two of the method detection limit must be noted. Until the required rinse time is established, this method suggests a rinse period of at least 60 seconds between samples and standards. If memory interference is suspected, the sample must be reanalyzed after a rinse period of sufficient length. Alternate rinse times may be established by the analyst based upon their DQOs.

4.5 Other Interferences

4.5.1 Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. If the instrument does not display negative values, fortify the interference check solution with the elements of interest at 0.5 to 1 mg/L and measure the added standard concentration accordingly. Concentrations must be within 20% of the true spiked concentration or dilution of the samples will be necessary. In the absence of measurable analyte, overcorrection could go undetected if a negative value is reported as zero.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in plastic bottles.

6.2 Sample Preservation

If samples are for soluble metals analysis, filtration must take place prior to preservation with 1:1 HNO3 to a pH < 2. Soluble samples must be held at pH < 2 for at least 24 hours prior to digestion if not preserved at the time of filtration. Samples for total metals analysis are preserved with 1:1 HNO3 to a pH < 2. Samples must be pH <2 for at least 24 hours prior to digestion if not preserved at the time of collection.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples to be analyzed for soluble metals, that have not been filtered, must be filtered and preserved within 24 hours of sample collection.

Preserved samples have a hold time of 6 months, and are stored at ambient temperature.

7. Equipment and Supplies

7.1 Inductively coupled argon plasma emission spectrometer:

- **Thermo Scientific ICAP Duo 6500 (Trace7)**

7.1.1 Computer-controlled emission spectrometer with background correction.

7.1.2 Radio-frequency generator compliant with FCC regulations.

7.1.3 Optional mass flow controller for argon nebulizer gas supply.

7.1.4 Optional peristaltic pump.

7.1.5 Optional Autosampler.

7.1.6 Argon gas supply - high purity.

7.2 Volumetric flasks of suitable precision and accuracy.

7.3 Volumetric pipets of suitable precision and accuracy.

8. Standards and Reagents

Reagent semiconductor and/or trace grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination. If the purity of a reagent is in question, analyze for contamination. If the concentration of the contamination is less than the MDL then the reagent is acceptable.

8.1 Hydrochloric acid (conc), HCl. Stored at room temperature in acid resistant cabinet. Expiration date if defined by vendor.

8.2 Hydrochloric acid (1:1), HCl. Add 500 mL concentrated HCl to 400 mL DI water and dilute to 1 liter in an appropriately sized beaker. Stored at room temperature in polypropylene bottle, expiration date if defined by vendor..

8.3 Nitric acid (conc), HNO_3. Stored at room temperature in acid resistant cabinet. Expiration date if defined by vendor.

8.4 Nitric acid (1:1), HNO_3. Add 500 mL concentrated HNO_3 to 400 mL DI water and dilute to 1 liter in an appropriately sized beaker. Stored at room temperature in polypropylene bottle, expiration date if defined by vendor..

8.5 Reagent Water. All references to water in the method refer to reagent water unless otherwise specified. Reagent water will be interference free. Refer to Chapter One for a definition of reagent water.

8.6 Standard stock solutions may be purchased or prepared from ultra- high purity grade chemicals or metals (99.99% pure or greater). All stock standards are ordered through ISO and American Association for Lab Accreditation vendors. All standards are in aqueous solutions and are generally at concentrations of 1000ppm and 10,000ppm.

8.7 Mixed calibration standard solutions

Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. Add the appropriate types and volumes of acids so that the standards are matrix matched with the sample digestates. Care must be taken when preparing the mixed standards to ensure that the elements are compatible and stable together. Transfer the mixed standard solutions to HDPE or polypropylene bottles for storage. Fresh

mixed standards must be prepared, as needed, with the realization that concentration can change on aging as evidenced by failures in the ICV.

NOTE: If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of water and warm the flask until the solution clears. Cool and dilute to 100 mL with water. For this acid combination, the silver concentration must be limited to 2 mg/L. Silver under these conditions is stable in a tap-water matrix for 30 days. Higher concentrations of silver require additional HCl.
Additionally, sulfur standards are stand-alone single element standards and therefore are not to be combined in a mixed calibration standard solution.

8.8 Blanks

Three types of blanks are required for the analysis for samples. The calibration blank is used in establishing the analytical curve, and the method blank is used to identify possible contamination resulting from varying amounts of the acids used in the sample processing. The rinse blank is used to flush the system between all samples and standards.

8.8.1 The calibration blank is prepared by acidifying reagent water to the same concentrations of the acids found in the standards. Prepare a sufficient quantity to flush the system between standards and samples. The calibration blank will also be used for all initial (ICB) and continuing calibration blank (CCB) determinations (see Sections 10.2 and 10.4). Refer to Section 10.4.1.2 for acceptance criteria and/or corrective actions.

8.8.2 The method blank must contain all of the reagents in the same volumes as used in the processing of the samples. The method blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis. Refer to Section 9.1 for acceptance criteria and/or corrective actions.

8.8.3 The rinse blank consists of HNO_3 (1% or 2%) (v/v) in reagent water. Prepare a sufficient quantity to flush the system between standards and samples.

8.9 The Initial Calibration Verification Standard (ICV) and the Continuing Calibration Verification Standard (CCV)

These ICV is prepared by the analyst by combining compatible elements from a standard source different than that of the calibration standard at a concentration at or near the mid-point of the calibration curve. The CCV is prepared from the same source as the calibration standards and must be at a concentration near the mid-point of the calibration curve.

8.10 Low Level of Quantification, (LLOQ)

The LLOQ is initially verified by the analysis of at least 7 replicate samples, spiked at the LLOQ and processed through all preparation and analysis steps of the method at or below the lowest calibration point. The mean recovery and relative standard deviation of these samples provide an initial statement of precision and accuracy at the LLOQ. In most cases the mean recovery should be +/- 35% of the true value and RSD should be < 20%. In-house limits may be calculated when sufficient data points exist. Monitoring recovery of LLOQ over time is useful for assessing precision and bias.

Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

8.11 Spectral Interference Check Solution

These solutions are prepared to contain known concentrations of interfering elements that will provide an adequate test of the correction factors. Analysts are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests.

Single element interference checks - At a minimum, single element SIC checks must be performed for the following elements: Aluminum 500mg/L; Boron 50mg/L, Barium, 50mg/L, Calcium 500mg/L; Copper 50mg/L; Iron 200mg/L; Magnesium 500mg/L; Manganese 50mg/L; Molybdenum 20mg/L; Sodium 1000mg/L; Nickel 20mg/L; Selenium 20mg/L; Silicon 200mg/L; Tin 20mg/L; Vanadium 20mg/L; Zinc 20mg/L The absolute value of the concentration observed for any unspiked analyte in the single element SIC checks must be less than two times the analytes' LLOQ.

The concentration of the SIC checks are suggested, but become the highest concentration allowed in a sample analysis, and cannot be higher than the highest established linear range. Samples with concentrations of elements higher than the SIC check must be diluted until the concentration is less than the SIC check solution. Note that reanalysis of a diluted sample is required even if the high concentration element is not required to be reported for the specific sample, since the function of the SIC check is to evaluate spectral interferences on other elements. The single element SIC checks are performed when the instrument is setup and periodically (at least once every 6 months) thereafter.

Mixed element interference check - The mixed element SIC solution is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than +/- the LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, or alternatively the LLOQ may be raised to twice the concentration observed in the SIC solution. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions These may be present up to the concentration documented plus the LLOQ.

Mixed element SIC solution: Aluminum, 500mg/L; Calcium, 500mg/L; Iron, 200mg/L; Magnesium, 500mg/L

8.12 Ongoing Low Level of Quantification, (LLOQ)

Ongoing LLOQ verification, at a minimum, is on a quarterly basis to validate quantitation capability at low analyte concentration levels. This verification may be accomplished either with clean control material (e.g., reagent water, method blanks. Optimally, the LLOQ should be less than the desired regulatory action levels based on the stated project-specific requirements.

8.13 Internal Standard

The internal standard consists of a multi-element solution; each internal standard covers a range of the spectrum (low, middle, or high wavelengths) and the elements within that range.

5.0ug/mL Scandium

3.0ug/mL Yttrium

Note: The standard is used to monitor instrument fluctuations including but not limited to nebulization efficiency, plasma variations, environmental temperature changes, peristaltic pump pulsations, etc. Therefore, the solution used to start an analysis calibration cannot be added to or changed out during analysis without requiring subsequent full recalibration.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank is a volume of reagent water carried through the same preparation process as a sample.

The method blank results must be less than ½ of the LLOQ for all analytes of concern. If the results of the method blank exceed the RDL for any analyte, perform re-analysis of a new aliquot of the method blank.

If the results continue to exceed the RDL, proceed as follows:

If all of the samples for the analyte are non-detected, and the method blank is at or above the RDL, no action is required.

If one or more associated samples for that analyte have positive results at or above the RDL, those samples must be considered to be out of control, and are re-digested and reanalyzed.

9.2 Laboratory Control Sample (LCS)

Analyze one LCSW/SRM per sample batch. A LCS/SRM sample is a spiked volume of reagent water that is brought through the entire preparation and analytical process. The LCSW must have a % Recovery of ± 20% within the actual value or within vendor control limits (95% confidence limits) for the solid SRM.

If the LCSW or SRM % Recovery is outside the acceptable limits as stated in Table 2, or outside any vendor control limits, the LCS is rerun once. If upon reanalysis the LCS is still out of control, the failed analytes are re-prepped and re-analyzed. Otherwise, a nonconformance report form is raised to document the exact problem and this form is then authorized by the QA/QC Director and/or the Laboratory Manager(s).

9.3 Initial Calibration Verification (ICV)

For all analytes and determinations, the laboratory must analyze an ICV (Section 8.9), and a calibration blank (ICB, Section 8.8.1), immediately following daily calibration. The results of the ICV are to agree within 10% of the expected value; if not, re-analyze once, if still failing terminate the analysis, correct the problem, and recalibrate the instrument.

9.4 Continuing Calibration Verification (CCV)

A calibration blank (CCB, Section 8.8.1) and a calibration verification standard (CCV, Section 8.9) must be analyzed after every tenth sample and at the end of the sample run. Analysis of the calibration verification (CCV) must verify that the instrument is within 10% of the calibration with the relative standard deviation < 5% from replicate (minimum of two) integrations.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The batch sheet is noted. If the standard fails again, instrument maintenance must be performed and the CCV/CCB standard is reanalyzed. If the standard passes, then all samples run after the last passing CCV/CCB pair must be re-analyzed.

If the standard fails after instrument maintenance, the instrument is recalibrated. A new ICV/ICB is performed, and all previous data after the last passing CCV/CCB is reanalyzed.

9.5 Matrix Spike

Analyze matrix spike samples at a frequency of one per matrix batch. The matrix spike is the same solution as used for the LCS (Table 4). A matrix spike sample is a sample brought through the entire sample preparation and analytical process.

9.5.1 The percent recovery is to be calculated as follows:

$$\% \text{ Recovery} = \frac{MS - S}{C} \times 100$$

where:

MS = Matrix Spike value
S = Sample value.
C = Concentration of the Spiking solution.

9.5.2 If the Matrix Spike falls outside of the limits as stated in Table 2, or outside any historical documentation for analytes of interest a post analytical spike is performed for the failed analytes. The same sample from which the MS/MSD aliquots were prepared should be spiked with a post digestion spike at a minimum level of 10 times and a maximum of 100 times the lower limit of quantitation. The acceptable % Recovery of the post analytical spike is 75-125%%. A nonconformance is noted in the LIMS and approved in secondary peer review and/or by the Metals Manager.

9.5.3 If the Post Spike fails the dilution test should be performed**.** If the analyte concentration is sufficiently high (minimally, a factor of 25 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution should agree within ± 20% of the original determination. If not, then a chemical or physical interference effect should be suspected.

9.6 Laboratory Duplicate

A duplicate sample is analyzed once per matrix batch. This sample is brought through the entire sample preparation and analytical process.

9.6.1 The relative percent difference between duplicate determinations is to be calculated as follows:

$$RPD = \frac{|D_1 - D_2|}{(|D_1 + D_2|)/2} \times 100$$

where:

RPD = relative percent difference.
D_1 = first sample value.
D_2 = second sample value (replicate).

9.6.2 If the Duplicate falls outside of the limits as stated in Table 2, or outside any historical documentation and the concentrations of the failing analytes are less than 5x the RL or a matrix interference is found a nonconformance is noted in the LIMS and approved in secondary peer review and/or by the Metals Manager.

9.7 Method-specific Quality Control Samples

9.7.1 Spectra Interference Check Standard

A mixed check solution is analyzed once daily (section 8.11). One solution (SIC) has only elevated concentrations of Fe, Al, Ca, Mg to ensure no interferences occur. The concentrations of the analytes of interest must have an absolute value of the LLOQ. This solution is analyzed at the beginning of the first analytical run of the day.

The high level interferences are not evaluated for recovery. If the SIC fails take corrective action which may include re-evaluation of the inter-element correction values (IECs) after running single element SIC. The instrument calibration routine must then be performed and confirmed by the ICV/ICB pair and the SIC re-analyzed before proceeding with analysis. Otherwise, the nonconformance issue is raised to the Department Supervisor and/or the QA Department.

9.7.2 Internal Standard

The internal standards are added prior to the nebulizer and corrects for intensity differences in the instrument response between the standard's and sample's matrix. They are monitored for any variation in response during the sample analyses and used to ratio the sample response to the internal standard response of the calibration blank. The ratio is applied to compensate for instrument conditions in the plasma or nebulization caused by the matrix. The internal standard is monitored for 50-150% recovery or laboratory generated control ranges difference from the calibration blank IS response to ensure the proper functioning of the internal standard introduction system and matrix interferences. If an injection falls outside of this acceptance range the sample or QC check is rerun once to check for an introduction error.

If a sample continues to fail it's to be run on successive increasing dilutions until the internal standards associated with the elements of interest are within range. If a QC check fails on the single rerun the analysis is stopped, the root cause investigated, corrected and the instrument re-calibrated/verified. The analysis begins again with all samples that were run after the last acceptable CCV/CCB pair.

9.7.3 LDR Check Solution: A multiple element or single element solution run at a point above the highest calibration standard under the same calibration used to quantify the associated sample data. The LDR check must be within +/-10% of the true value of each element of interest to be considered valid.

9.8 Method Sequence

- Calibration of instrument
- Initial Calibration Verification Standard
- Initial Calibration Blank
- Mixed SIC Solution
- Continuing Calibration Verification Standard
- Continuing Calibration Blank

- 10 samples
- Continuing Calibration Verification Standard
- Continuing Calibration Blank
- 10 Samples
- Continuing Calibration Verification Standard
- Continuing Calibration Blank

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation

Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Groundwater samples which have been prefiltered and acidified will not need acid digestion. Samples which are not digested must be matrix matched with the standards.

NOTE: Sample digestates intended for Silver analysis must be analyzed as soon as possible.

10.1.2 Instrument Set-Up

Set up the instrument with proper operating parameters established as detailed below. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).

Startup Procedures

For iCAP Duo 6500

- Turn on power to the chiller
- Click on ThermoSpec Icon; enter analyst initials in login screen
- Click on Plasma icon to start instrument
- Allow to warm up for 30 minutes
- Enter analytical workgroup number (obtained from LIMS) globally under the Instrument menu by selecting Tools, then Options, then Analyst.
- Click on the Sequence tab and enter the sequence by selecting New Autosampler Table, Add Sequence, Add # of spaces.
- Enter the sample locations and IDs
- Press Run Auto-Session button (►) in menu bar.

10.1.2.1 Specific wavelengths are listed in Table 1. Other wavelengths may be substituted if they can provide the needed sensitivity and are corrected for spectral interference. The instrument and operating conditions utilized for determination must be capable of providing data of acceptable quality to the program and data user.

Operating conditions for axial plasma will vary from 1100 – 1500 watts forward power, 15-19 Liters/min argon coolant flow, 0.5 – 0.7 L/min argon nebulizer flow, 140 – 200 rpm pump rate and a default 1 minute preflush time and 10 second measurement time is recommended for all simultaneous instruments.

10.1.2.2 The plasma operating conditions need to be optimized prior to use of the instrument. This routine is not required on a daily basis, but only when first setting up a new instrument or following a change in operating conditions. The following procedure is recommended or follow manufacturer's recommendations. The purpose of plasma optimization is to provide a maximum signal to background ratio for some of the least sensitive elements in the analytical array. The use of a mass flow controller to regulate the nebulizer gas flow or source optimization software greatly facilitates the procedure.

10.1.2.2.1 The Thermo ICP's typically use a Meinhard Nebulizer. The nebulizer flow for each instrument is 1.0 +/- 0.2 mL/min.

10.1.2.2.2 The 6500 Duo instruments automatically perform a wavelength check at start up without user interaction.

10.1.2.2.3 The instrument operating condition finally selected as being optimum must provide the lowest reliable instrument detection limits and method detection limits.

10.1.2.2.4 If either the instrument operating conditions, such as incident power or nebulizer gas flow rate are changed, or a new torch injector tube with a different orifice internal diameter is installed, the plasma and argon pressures must be reoptimized.

10.1.2.2.5 After completing the initial optimization of operating conditions, but before analyzing samples, the laboratory must establish and initially verify an interelement spectral interference correction routine to be used during sample analysis. A general description concerning spectral interference and the analytical requirements for background correction in particular are discussed in the section on interferences. Criteria for determining an interelement spectral interference is an apparent positive or negative concentration for the analyte that falls within ± ½ LLOQ. The upper control limit is the analyte instrument detection limit. Once established, the entire routine is periodically verified every six months. In between that time, IEC's are done on a need be basis per analyte. Only a portion of the correction routine must be verified more frequently or on a daily basis. Initial and periodic verification of the routine must be kept on file. Special cases where continual verification is required are described elsewhere.

10.1.2.3 Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on each particular instrument. All measurements must be within the instrument linear range where the correction equations are valid.

10.1.2.3.1 Method detection limits must be established for all wavelengths utilized for each type of matrix commonly analyzed. The matrix used for the MDL calculation must contain analytes of known concentrations within 3-5 times the anticipated detection limit.

10.1.2.3.2 Determination of limits using reagent water MDLs represent a best case situation and do not represent possible matrix effects of real world samples.

10.1.2.3.3 If additional confirmation is desired, reanalyze the seven replicate aliquots on two more non-consecutive days and again calculate the method detection limit values for each day. An average of the three values for each analyte may provide for a more appropriate estimate.

10.1.2.3.4 The upper limit of the linear dynamic range must be established for each wavelength utilized by determining the signal responses with 10% of the true value of each element from a concentration standard at the upper limit of the range run on the same calibration as required by the sample responses above the calibration range. The range which may be used for the analysis of samples must be no more than 90% of the resulting data. Determined analyte concentrations that are above the upper range limit must be diluted and reanalyzed. The analyst must also be aware that if an inter-element correction from an analyte above the linear range exists, a second analyte where the inter-element correction has been applied may be inaccurately reported.

NOTE: Many of the alkali and alkaline earth metals have non-linear response curves due to ionization and self-absorption effects. These curves may be used if the instrument allows; however the effective range must be checked and the second order curve fit must have a correlation coefficient of 0.995 or better. Third order fits are not acceptable. These curves are much more sensitive to changes in operating conditions than the linear lines and must be checked whenever there have been moderate equipment changes.

10.1.2.4 The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.

10.2 Initial Calibration

Calibrate the instrument according to the instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Section 8.7. Flush the system with the calibration blank (Section 8.8.1) between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a calibration blank, RL standard and a high level standard. Calibration curve verification is accomplished through the analysis of the ICV, ICB and SIC standards.

10.3 Equipment Operation and Sample Processing

10.3.1 For all analytes and determinations, the laboratory must analyze an ICV (Section 8.9), and a calibration blank (ICB, Section 8.8.1), immediately following daily calibration.

A calibration blank (CCB, Section 8.8.1) and a calibration verification standard (CCV, Section 8.9) must be analyzed after every tenth sample and at the end of the sample run. Analysis of the calibration verification (CCV) must verify that the instrument is within 10% of the calibration with the relative standard deviation < 5% from replicate (minimum of three) integrations.

If the calibration cannot be verified within the specified limits, the sample analysis must be discontinued, the cause determined and the instrument recalibrated. All samples following the last acceptable ICV/ICB, or CCV/CCB must be reanalyzed. The analysis

data for the calibration blank, check standard, and ICV or CCV must be kept on file with the sample analysis data.

10.3.2 Rinse the system with the rinse blank solution (Section 8.8.3) before the analysis of each sample. The suggested default rinse time is one minute. Each ICP instrument may establish a reduction in this rinse time through a suitable demonstration.

10.3.3 Dilute and reanalyze samples that exceed the linear range or use a calibrated alternate, less sensitive line for which quality control data is already established.

10.3.4 If less than acceptable accuracy and precision data are generated a series of tests are performed prior to reporting concentration data for analyte elements. At a minimum, these tests should be performed with each batch of samples prepared/analyzed with corresponding unacceptable data quality results. These tests, as outlined in Sections 10.3.5 and 10.3.6, will ensure that neither positive nor negative interferences are operating on any of the analyte elements to distort the accuracy of the reported values.

10.3.5 **Post Digestion Spike Addition:** If the sample concentrations are insufficient to perform a dilution test a post digestion spike added to a portion of a prepared sample, or its dilution for the elements failing the matrix spike recoveries must be run, recovery limits equal to 75% to 125% of the known spike value. If the spike is not recovered within the specified limits If the post-digestion recovery fails to meet the acceptance criteria, the sample results must be reported as estimated values

10.3.6 **Dilution Test:** If the analyte concentration is sufficiently high (minimally, a factor of 25 above the lower limit of quantitation after dilution), an analysis of a 1:5 dilution must agree within ± 20% of the original determination. Elements that fail the dilution test are reported as estimated values.

10.3.7 **CAUTION:** If spectral overlap is suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended.

10.4 Continuing Calibration

10.4.1 Check calibration with an ICV following the initial calibration (Section 8.9). Verify calibration with the Continuing Calibration Verification (CCV) Standard (Section 8.9) at the end of the initial calibration sequence (ICV, ICB), after every ten samples, and at the end of an analytical run. Use a calibration blank (Section 8.8.1) immediately following daily calibration, after every 10 samples and at the end of the analytical run.

10.4.1.1 The results of the ICV are to agree within 10% of the expected value, and CCVs are to agree within 10% of the expected value; if not, terminate the analysis, correct the problem, and recalibrate the instrument. Each may be rerun once to confirm or cure the initial failure.

10.4.1.2 The results of the calibration blank should be below ½ of LLOQ or RL (whichever is lower). If not, repeat the analysis and if the failure is repeated terminate the analysis, correct the problem, recalibrate, and reanalyze the previous 10 samples. If the blank is less than 1/10 the concentration of the action level of interest, and no sample is within ten percent of the action limit, analyses need not be rerun and recalibration need not be performed before continuation of the run.

10.4.2 Verify the inter-element and background correction factors at the beginning of each analytical run. Do this by analyzing the SIC (Section 8.10). Results must be less than +/- LLOQ for all non-spiked elements.

10.4.3 When low-level sensitivity is required, a check standard at the requested limit of quantitation is analyzed to confirm the reported detection limit (RDL). This is performed on a project-by-project basis.

10.5 Preventive Maintenance

Whenever instrument maintenance is performed, it is noted in the instrument's Maintenance Logbook.

10.5.1 Daily

Inspect the nebulizer pump tubing from the Autosampler to the Nebulizer. Replace if necessary.

10.5.2 Monthly or as needed

Remove the torch, "shot glass", nebulizer and spray chamber. Clean each with 10% Nitric Acid and rinse with tap water. Coat the inside of the spray chamber and shot glass with concentrated Sulfuric Acid and soak for one hour, then rinse well with DI water. Soak the torch and nebulizer in aqua regia overnight, then rinse with DI water.

10.5.3 Every 6 months

Preventive Maintenance is performed by the Vendor or in-house personnel as follows:

- check the cooling system
- flush/refill the chiller with distilled water and antibacterial conditioner
- clean the instrument to regain intensity
- clean/replace air filters.

11. Data Evaluation, Calculations and Reporting

11.1 If dilutions were performed, the appropriate factors must be applied to sample values. All results must be reported with up to three significant figures.

11.2 Soil samples

Soil samples are calculated as follows:

$$\mathbf{A} = \frac{\text{Sample weight (grams)}}{\text{Final Volume (mL)}}$$

$$\mathbf{B}\ (\text{concentration in mg/Kg}) = \frac{\text{Concentration of analyte (mg/L)}}{\mathbf{A}}$$

11.2.1 Dry weight correction

The LIMS calculates the dry weight correction, however it is calculated as follows:

$$\text{Final concentration in mg/Kg dry weight} = \frac{B}{\%\ \text{Solids}}$$

11.3 Liquid samples

Liquid samples are calculated as follows:

$$\text{Dilution Factor} = \frac{\text{Final Volume (mL)}}{\text{Sample Volume (mL)}}$$

Final concentration in mg/L = Concentration of analyte (mg/L) x Dilution Factor

11.4 Calculations for Hardness

The method for determining hardness is to compute it from the results of separate determinations of Calcium and Magnesium on aqueous samples.

11.4.1 Total Hardness

Total Hardness, mg equivalent $CaCO_3$/L = [2.497 (Ca, mg/L)] + [4.118 (Mg, mg/L)]

11.4.2 Calcium Hardness

Calcium Hardness, mg equivalent $CaCO_3$/L = [2.497 (Ca, mg/L)]

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Also refer to Section 9 for Quality Control and acceptance criteria.

If the SIC is outside of the recovery window, then the standard is reanalyzed. If the standard failure continues, the IECs for the element/elements in question are reviewed and recalculated if necessary.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The raw data is noted. If the standard fails again, the problem must be found and corrected and the instrument is recalibrated. The ICV/ICB standard is reanalyzed and all previous data that had failed back to the previous passing CCV/CCB is reanalyzed.

The reanalysis procedure outline above is also conducted for a failing LCS or Method Blank; they may be rerun alone on the new or any subsequent passing bracket. The LCS or Method Blank do not qualify a bracket of samples but the batch run itself.

If the Matrix Spike does not meet acceptance criteria, a dilution test is performed. If the levels of the native sample is inadequate (see section 10.3.6) The RPD must be within 20% of the true value of the native sample. If the dilution test fails or the concentrations in the native sample are inadequate, the post spike is analyzed and evaluated (section 10.3.5). If these criteria are met, then the Matrix Spike data is reported, with the post spike narrated on the final report. If the post spike fails the data is reported as estimated.

If sample Duplicates are outside of the acceptance criteria, the analyst examines the sample for homogeneity. If the sample is not homogenous, this is narrated on the final report. Clean, homogenous samples are reanalyzed and if still outside of the acceptance limits, redigested and reanalyzed.

Sample nonconformance regarding a Matrix Spike recovery or a duplicate %RSD is narrated on the final report along with the corrective action(s) taken.

The mixed element SIC solution is analyzed at least once per day, immediately after the initial calibration. The concentration measured for any target analytes must be less than +/- the LLOQ. If this criterion is not met then sample analysis may not proceed until the problem is corrected, instrument is recalibrated, verified with the ICV/ICB and the SIC is then re-analyzed. Alternatively, the LLOQ may be raised to twice the concentration observed in the SIC solution if approved by the Department Manger or QA Department and the level is below the regulatory action limit or project specific requirements. The only exceptions are those elements that have been demonstrated to be contaminants in the SIC solutions These may be present up to the concentration documented plus the LLOQ. If failure continues notify the Department Supervisor or Manager.

Silver is only slightly soluble in the presence of chloride unless there is a sufficient chloride concentration to form the soluble chloride complex. Therefore, low recoveries of silver may occur in samples, matrix spikes and even laboratory control samples.

Silver is flagged in our data software during analysis if the concentration of silver is >100ug/L. Liquid samples that exceed 100ug/L will be redigested at dilution. Soil and solid samples that exceed 500ug/L (approximately 20mg/kg) will be re-digested at a reduced weight of 0.625g to allow more silver to become soluble during digestion.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05 unless supersede within this SOP. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP #1732 MDL/LOD/LOQ Generation

SOP# 1739 IDC/DOC Generation

SOP# 1728 Waste Management and Disposal

16. Attachments

TABLE 1: Element Wavelengths

TABLE 2: Precision and Accuracy Acceptance Criteria

TABLE 3: Reporting Limits

TABLE 4: LCS and Matrix Spike Concentrations

TABLE 1
ELEMENT WAVELENGTHS

Element	6500 Duo wavelength (nm)
Pb	220.3
Se	196.0
Sb	206.8
As	189.0
Ba	455.4
Be	313.0
Cd	214.4
Co	228.6
Cu	324.7
Cr	267.7
Fe	259.9
Mn	257.6
Mo	202.0
Ni	231.6
Ag	328.0
Tl	190.8
V	292.4
Zn	206.2
Al	396.1
Ca	315.8
Mg	279.0
B	208.9
Si	212.9
Sn	189.9
Sr	421.5
Ti	334.9
Bi	223.0
Na	589.5
K	766.4
S	180.7
Li	

TABLE 2
PRECISION AND ACCURACY ACCEPTANCE CRITERIA

	% Recovery LCS		Aqueous % Recovery MS		Soil % Recovery SRM *		Duplicate	
Element	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**	**Aqueous %RPD**	**Soil %RPD**
Aluminum	80	120	75	125	29	171	20	20
Antimony	80	120	75	125	4	196	20	20
Arsenic	80	120	75	125	81	119	20	20
Barium	80	120	75	125	83	118	20	20
Beryllium	80	120	75	125	83	117	20	20
Boron	80	120	75	125	70	129	20	20
Cadmium	80	120	75	125	82	117	20	20
Calcium	80	120	75	125	83	117	20	20
Chromium	80	120	75	125	80	119	20	20
Cobalt	80	120	75	125	83	117	20	20
Copper	80	120	75	125	83	117	20	20
Iron	80	120	75	125	51	150	20	20
Lead	80	120	75	125	80	120	20	20
Lithium	80	120	75	125	80	120	20	20
Magnesium	80	120	75	125	74	126	20	20
Manganese	80	120	75	125	83	117	20	20
Molybdenum	80	120	75	125	81	119	20	20
Nickel	80	120	75	125	82	117	20	20
Potassium	80	120	75	125	74	126	20	20
Sulfur	80	120	75	125	NA	NA	20	20
Selenium	80	120	75	125	80	120	20	20
Silica (SiO_2)	80	120	75	125	NA	NA	20	20
Silver	80	120	75	125	66	134	20	20
Sodium	80	120	75	125	74	127	20	20
Strontium	80	120	75	125	80	120	20	20
Thallium	80	120	75	125	79	120	20	20
Tin	80	120	75	125	69	131	20	20
Titanium	80	120	75	125	82	118	20	20
Vanadium	80	120	75	125	79	121	20	20
Zinc	80	120	75	125	82	119	20	20

** Ranges of the SRM are presented as an example of a typical SRM; actual limits may vary by lot provided by the vendor.

TABLE 3
REPORTING LIMITS

Element	Aqueous (mg/L)	Soil (mg/Kg)
ALUMINUM	0.10	4.0
ANTIMONY	0.05	2.0
ARSENIC	0.005	0.40
BARIUM	0.01	0.40
BERYLLIUM	0.005	0.20
BORON	0.03	1.2
CADMIUM	0.005	0.40
CALCIUM	0.10	4.0
CHROMIUM	0.01	0.40
COBALT	0.02	0.80
COPPER	0.01	0.40
IRON	0.05	2.0
LEAD	0.01	2.0
LITHIUM	0.009	0.36
MAGNESIUM	0.10	4.0
MANGANESE	0.01	0.40
MOLYBDENUM	0.05	2.0
NICKEL	0.025	1.0
POTASSIUM	2.5	100
SULFUR	0.25	10
SELENIUM	0.01	0.80
SILICA	0.50	20
SILVER	0.007	0.40
SODIUM	2.0	80
STRONTIUM	0.01	2.0
THALLIUM	0.02	0.80
TIN	0.05	4.0
TITANIUM	0.01	0.40
VANADIUM	0.01	0.40
ZINC	0.05	2.0

TABLE 4
LCS and Matrix Spike

Analyte	Liquid Concentration (mg/L)	Soil Concentration * (MS spike only) (mg/Kg)
Antimony	0.5	160
Arsenic	0.12	160
Barium	2.00	160
Beryllium	0.05	80
Cadmium	0.051	80
Chromium	0.20	160
Copper	0.25	160
Lead	0.51	160
Nickel	0.50	160
Selenium	0.12	160
Silver	0.05	40
Thallium	0.12	160
Zinc	0.50	160
Iron	1.00	800
Manganese	0.50	160
Calcium	10.0	800
Magnesium	10.0	800
Potassium	10.0	800
Sodium	10.0	800
Silica	1.0	800
Aluminum	2.00	800
Cobalt	0.50	160
Vanadium	0.50	160
Boron	1.0	NA
Molybdenum	1.0	NA
Lithium	1.0	800
Titanium	1.0	NA

*MS spike of a solid based on 1.25g and a final volume of 50 mL.

Note: Solids LCS is an SRM with certified value provided by the vendor on a lot basis.

Method 1633 Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, Oil and Tissue Samples by LC-MS/MS

References: Method 1633 - Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, Oil and Tissue Samples by LC-MS/MS (2nd Draft -June 2022)

DOD QSM (US Department of Defense Quality Systems Manual for Environmental Laboratories, version 5.4, 20221)

1. Scope and Application

Matrices: Drinking water, Non-potable Water, Tissues, Oils, Biosolids and Solid Matrices

Definitions: Refer to Alpha Analytical Quality Manual.

1.1 Method 1633 is for use in the Clean Water Act (CWA) for the determination of the per- and polyfluoroalkyl substances (PFAS) in Table 1 in aqueous, solid (soil, biosolids, sediment) and tissue samples by liquid chromatography/mass spectrometry (LC-MS/MS).

1.2 The method calibrates and quantifies PFAS analytes using isotopically labeled standards. Where linear and branched isomers are present in the sample and either qualitative or quantitative standards containing branched and linear isomers are commercially available, the PFAS analyte is reported as a single analyte consisting of the sum of the linear and branched isomer concentrations

1.3 This is a liquid chromatography/tandem mass spectrometry (LC/MS/MS) method for the determination of selected perfluorinated alkyl substances (PFAS) in Non-Drinking Water, tissue soil and biosolid Matrices. Accuracy and precision data have been generated for the compounds listed in Table 1.

1.4 The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of the LC/MS/MS and in the interpretation of LC/MS/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

2.1 Environmental samples are prepared and extracted using method-specific procedures. Sample extracts are subjected to cleanup procedures designed to remove interferences. Analyses of the sample extracts are conducted by LC-MS/MS in the multiple reaction monitoring (MRM) mode. Sample concentrations are determined by isotope dilution or extracted internal standard quantification using isotopically labeled compounds added to the samples before extraction.

2.2 Aqueous samples are spiked with isotopically labeled standards, extracted using solid-phase extraction (SPE) cartridges and undergo cleanup using carbon before analysis.

2.3 Solid and Oil samples are spiked with isotopically labeled standards, extracted into basic methanol, and cleaned up by carbon and SPE cartridges before analysis

2.4 Tissue samples are spiked with isotopically labeled standards, extracted in potassium hydroxide and acetonitrile followed by basic methanol, and cleaned up by carbon and SPE cartridges before analysis.

2.5 A sample extract is injected into an LC equipped with a C18 column that is interfaced to an MS/MS). The analytes are separated and identified by comparing the acquired mass spectra and retention times to reference spectra and retention times for calibration standards acquired under identical LC/MS/MS conditions. The concentration of each analyte is determined by using the isotope dilution technique. Extracted Internal Standards (EIS) analytes are used to monitor the extraction efficiency of the method analytes.

2.6 Method Modifications from Reference

N/A

3. Reporting Limits

The reporting limit for PFAS's are listed in Table 8.

4. Interferences

4.1 PFAS standards, extracts and samples should not come in contact with any glass containers or pipettes as these analytes can potentially adsorb to glass surfaces. PFAS analyte and EIS standards commercially purchased in glass ampoules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene containers.

4.2 Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The method analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, SPE sample transfer lines, etc. All items such as these must be routinely demonstrated to be free from interferences (less than 1/2 the RL for each method analyte) under the conditions of the analysis by analyzing laboratory reagent blanks as described in Section 9.1. Subtracting blank values from sample results is not permitted.

4.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the water. Humic and/or fulvic material can be co-extracted during SPE and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent. Total organic carbon (TOC) is a good indicator of humic content of the sample.

4.4 SPE cartridges can be a source of interferences. The analysis of field and laboratory reagent blanks can provide important information regarding the presence or absence of such interferences. Brands and lots of SPE devices should be tested to ensure that contamination does not preclude analyte identification and quantitation.

5. Health and Safety

5.1 The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

5.2 All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.3 PFOA has been described as “likely to be carcinogenic to humans.” Pure standard materials and stock standard solutions of these method analytes should be handled with suitable protection to skin and eyes, and care should be taken not to breathe the vapors or ingest the materials.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection for Aqueous Samples

6.1.1 Samples must be collected in two (2) 500-mL or 250-mL high density polyethylene (HDPE) container with an unlined plastic screw cap. All sample containers must have linerless HDPE or polypropylene caps.

6.1.2 The sample handler must wash their hands before sampling and wear nitrile gloves while filling and sealing the sample bottles. PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

6.1.3 Open the tap and allow the system to flush until the water temperature has stabilized (approximately 3 to 5 min). Collect samples from the flowing system.

6.1.4 Fill sample bottles. Samples do not need to be collected headspace free.

6.1.5 After collecting the sample and cap the bottle. Keep the sample sealed from time of collection until extraction.

6.1.6 Maintain all aqueous samples protected from light at 0 - 6 ºC from the time of collection until shipped to the laboratory. Samples must be shipped as soon as practical with sufficient ice to maintain the sample temperature below 6 ºC during transport and be received by the laboratory within 48 hours of collection. The laboratory must confirm that the sample temperature is 0 - 6 ºC upon receipt. Once received by the laboratory, the samples must be stored at ≤ -20 ºC until sample preparation.

6.2 Sample Collection for Solid and Oil samples.

6.2.1 Grab samples are collected in polypropylene containers. Sample containers and contact surfaces containing PTFE shall be avoided. Samples should fill no more than ¾ full.

6.2.2 Maintain solid samples protected from light (in HDPE containers) at 0 - 6 ºC from the time of collection until receipt at the laboratory. The laboratory must confirm that the sample temperature is 0 - 6 ºC upon receipt. Once received by the laboratory, the samples must be stored at ≤ -20 ºC until sample preparation.

6.3 Sample Collection for fish and other tissue samples

6.3.1 If the time of collection to the time of receipt at the laboratory is expected to exceed 24 hours, the tissue samples must be frozen upon collection and shipped to the laboratory on dry ice.

6.3.2 Once received by the laboratory, the samples must be maintained protected from light at ≤ -20 ºC until prepared. Store unused samples in HDPE containers or wrapped in aluminum foil at ≤ -20 ºC.

6.3.3 The nature of the tissues of interest may vary by project. Field sampling plans and protocols should explicitly state the samples to be collected and if any processing will be conducted in the field (e.g., filleting of whole fish or removal of organs). All field procedures must involve materials and equipment that have been shown to be free of PFAS.

6.4 Sample Preservation

Not applicable.

6.5 Sample Shipping

Samples must be chilled during shipment and must not exceed 0 – 6 °C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 0 – 6 °C when the samples are received at the laboratory. Samples stored in the lab must be held at or below 6 °C until extraction but should not be frozen.

NOTE: Samples that are significantly above 0 – 6 ° C, at the time of collection, may need to be iced or refrigerated for a period of time, in order to chill them prior to shipping. This will allow them to be shipped with sufficient ice to meet the above requirements.

6.6 Sample Handling

6.6.1 Aqueous samples (including leachates) should be analyzed as soon as possible; however, samples may be held in the laboratory for up to 90 days from collection, when stored at ≤ -20 ºC and protected from the light. When stored at 0 - 6 ºC and protected from the light, aqueous samples may be held for up to 28 days, with the caveat that issues were observed with certain perfluorooctane sulfonamide ethanols and perfluorooctane sulfonamidoacetic acids after 7 days. These issues are more likely to elevate the observed concentrations of other PFAS compounds via the transformation of these precursors if they are present in the sample.

6.6.2 Solid samples (soils and sediments), Oil and tissue samples may be held for up to 90 days, if stored by the laboratory in the dark at either 0 - 6 ºC or ≤ -20 ºC, with the caveat that samples may need to be extracted as soon as possible if NFDHA is an important analyte.

6.6.3 Biosolids samples may be held for up to 90 days, if stored by the laboratory in the dark at 0 - 6 ºC or at -20 ºC. Because microbiological activity in biosolids samples at 0 - 6 ºC may lead to production of gases which may cause the sample to be expelled from the container when it is opened, as well as producing noxious odors, EPA recommends that samples be frozen if they need to be stored for more than a few days before extraction. Store sample extracts in the dark at less than 0 - 4 ºC until analyzed. If stored in the dark at less than 0 - 4 ºC, sample extracts may be stored for up to 90 days, with the caveat that issues were observed for some ether sulfonates after 28 days. These issues may elevate the observed concentrations of the ether sulfonates in the extract over time. Samples may need to be extracted as soon as possible if NFDHA is an important analyte.

7. Equipment and Supplies

7.1 SAMPLE CONTAINERS – 500-mL or 250-mL high density polyethylene (HDPE) bottles fitted with unlined screw caps. Sample bottles must be discarded after use.

7.2 SAMPLE JARS – 8-ounce wide mouth high density polyethylene (HDPE) bottles fitted with unlined screw caps. Sample bottles must be discarded after use.

7.3 POLYPROPYLENE BOTTLES – 4-mL narrow-mouth polypropylene bottles.

7.4 CENTRIFUGE TUBES – 50-mL conical polypropylene tubes with polypropylene screw caps for storing standard solutions and for collection of the extracts.

7.5 AUTOSAMPLER VIALS – Polypropylene 0.7-mL autosampler vials with polypropylene caps.

7.5.1 NOTE: Polypropylene vials and caps are necessary to prevent contamination of the sample from PTFE coated septa. However, polypropylene caps do not reseal, so evaporation occurs after injection. Thus, multiple injections from the same vial are not possible.

7.6 POLYPROPYLENE GRADUATED CYLINDERS – Suggested sizes include 25, 50, 100 and 1000-mL cylinders.

7.7 Auto Pipets – Suggested sizes include 5, 10, 25, 50, 100, 250, 500, 1000, 5000 and 10,000-µls.

7.8 PLASTIC PIPETS – Polypropylene or polyethylene disposable pipets.

7.9 Silanized glass wool (Sigma-Aldrich, Cat # 20411 or equivalent) – store in a clean glass jar and rinsed with methanol (2 times) prior to use.

7.10 Disposable syringe filter, 25-mm, 0.2-µm Nylon membrane, PALL/Acrodisc or equivalent

7.11 Variable volume pipettes with disposable HDPE or polypropylene tips (10 µL to 5 mL) used for preparation of calibration standards and spiked samples.

7.12 ANALYTICAL BALANCE – Capable of weighing to the nearest 0.0001 g.

7.13 ANALYTICAL BALANCE – Capable of weighing to the nearest 0.1 g.

7.14 SOLID PHASE EXTRACTION (SPE) APPARATUS FOR USING CARTRIDGES

7.14.1 SPE CARTRIDGES – (Phenomenex WAX 150 or 250mg or equivalent). The SPE sorbent must have a pKa above 8 so that it remains positively charged during the extraction.

7.14.1.1 Note: SPE cartridges with different bed volume (e.g., 500 mg) may be used; however, the laboratory must demonstrate that the bed volume does not negatively affect analyte absorption and elution, by performing the initial demonstration of capability analyses described in Section 13.

7.14.2 VACUUM EXTRACTION MANIFOLD – A manual vacuum manifold with large volume sampler for cartridge extractions, or an automatic/robotic sample preparation system designed for use with SPE cartridges, may be used if all QC requirements discussed in Section 9 are met. Extraction and/or elution steps may not be changed or omitted to accommodate the use of an automated system. Care must be taken with automated SPE systems to ensure the PTFE commonly used in these systems does not contribute to unacceptable analyte concentrations in the MB.

7.14.3 SAMPLE DELIVERY SYSTEM – Use of a polypropylene transfer tube system, which transfers the sample directly from the sample container to the SPE cartridge, is recommended, but not mandatory. Standard extraction manifolds come equipped with PTFE transfer tube systems. These can be replaced with 1/8" O.D. x 1/16" I.D. polypropylene or polyethylene tubing cut to an appropriate length to ensure no sample contamination from the sample transfer lines. Other types of non-PTFE tubing may be used provided it meets the MB and LCS QC requirements.

7.15 EXTRACT CONCENTRATION SYSTEM – Extracts are concentrated by evaporation with nitrogen using a water bath set no higher than 55 °C.

7.16 LABORATORY OR ASPIRATOR VACUUM SYSTEM – Sufficient capacity to maintain a vacuum of approximately 10 to 15 inches of mercury for extraction cartridges.

7.17 LIQUID CHROMATOGRAPHY (LC)/TANDEM MASS SPECTROMETER (MS/MS) WITH DATA SYSTEM

7.17.1 LC SYSTEM – Instrument capable of reproducibly injecting up to 10-µL aliquots and performing binary linear gradients at a constant flow rate near the flow rate used for development of this method (0.4 mL/min). The LC must be capable of pumping the water/methanol mobile phase without the use of a degasser which pulls vacuum on the mobile phase bottle (other types of degassers are acceptable). Degassers which pull vacuum on the mobile phase bottle will volatilize the ammonium acetate mobile phase causing the analyte peaks to shift to earlier retention times over the course of the analysis batch. The usage of a column heater is optional.

7.17.2 LC/TANDEM MASS SPECTROMETER – The LC/MS/MS must be capable of negative ion electrospray ionization (ESI) near the suggested LC flow rate of 0.4 mL/min. The system must be capable of performing MS/MS to produce unique product ions for the method analytes within specified retention time segments. A minimum of 10 scans across the chromatographic peak is required to ensure adequate precision.

7.17.3 DATA SYSTEM – An interfaced data system is required to acquire, store, reduce, and output mass spectral data. The computer software should have the

capability of processing stored LC/MS/MS data by recognizing an LC peak within any given retention time window. The software must allow integration of the ion abundance of any specific ion within specified time or scan number limits. The software must be able to calculate relative response factors, construct linear regressions or quadratic calibration curves, and calculate analyte concentrations.

7.17.4 INSTRUMENT COLUMNS

7.17.4.1 ANALYTICAL: C18 column, 1.7 µm, 50 x 2.1 mm (Waters Acquity UPLC® BEH or equivalent)

7.17.4.2 OPTIONAL GUARD COLUMN: (Phenomenex Kinetex® Evo C18 or equivalent)

8. Reagents and Standards

8.1 GASES, REAGENTS, AND SOLVENTS – Reagent grade or better chemicals must be used.

8.1.1 REAGENT WATER – Purified water which does not contain any measurable quantities of any method analytes or interfering compounds greater than 1/2 the RL for each method analyte of interest. Prior to daily use, at least 3 L of reagent water should be flushed from the purification system to rinse out any build-up of analytes in the system's tubing.

8.1.2 METHANOL (CH_3OH, CAS#: 67-56-1) – High purity, demonstrated to be free of analytes and interferences.

8.1.3 AMMONIUM ACETATE ($NH_4C_2H_3O_2$, CAS#: 631-61-8) – High purity, demonstrated to be free of analytes and interferences. Store at 2-8° and replace 2 years after opening date.

8.1.4 ACETIC ACID (H_3CCOOH, CAS#: 64-19-7) - High purity, demonstrated to be free of analytes and interferences and stored at room temperature.

8.1.4.1 Acetic Acid (0.1%) – Dissolve acetic acid (1 mL) in reagent water (1 L), store at room temperature, replace after 3 months.

8.1.5 1M AMMONIUM ACETATE/REAGENT WATER – High purity, demonstrated to be free of analytes and interferences.

8.1.6 2mM AMMONIUM ACETATE/METHANOL:WATER (5:95) – To prepare, mix 2 ml of 1M AMMONIUM ACETATE,1 ml ACETIC ACID and 50 ml METHANOL into I Liter of REAGENT WATER.

8.1.7 ACETONITRILE – UPLC grade or equivalent, store at room temperature

8.1.8 TOLUENE – HPLC grade or equivalent.

8.1.9 ACETONE – pesticide grade or equivalent

8.1.10 AMMONIUM HYDROXIDE (NH_3, CAS#: 1336-21-6) – High purity, demonstrated to be free of analytes and interferences, and stored at room temperature.

8.1.11 AQUEOUS AMMONIUM HYDROXIDE (3%) – Add ammonium hydroxide (10 mL, 30%) to reagent water (90 mL), store at room temperature, replace after 3 months.

8.1.12 METHANOLIC AMMONIUM HYDROXIDE (0.3%) - add ammonium hydroxide (1 mL, 30%) to methanol (99 mL), store at room temperature, replace after 1 month

8.1.13 METHANOLIC AMMONIUM HYDROXIDE (1%) - add ammonium hydroxide (3.3 mL, 30%) to methanol (97 mL), store at room temperature, replace after 1 month

8.1.14 METHANOLIC AMMONIUM HYDROXIDE (2%) - add ammonium hydroxide (6.6 mL, 30%) to methanol (93.4 mL), store at room temperature, replace after 1 month

8.1.15 METHANOLIC POTASSIUM HYDROXIDE (0.05 M) – add 3.3 g of potassium hydroxide to 1 L of methanol, store at room temperature, replace after 3 months

8.1.16 METHANOL WITH 4% WATER, 1% AMMONIUM HYDROXIDE AND 0.625% ACETIC ACID - add ammonium hydroxide (3.3 mL, 30%), reagent water (1.7 mL) and acetic acid (0.625 mL) to methanol (92 mL), store at room temperature, replace after 1 month. This solution is used to prepare the instrument blank and calibration standards (Section 8.3.2).

8.1.17 FORMIC ACID – (greater than 96% purity or equivalent). Store at room temperature and replace after 2 years.

8.1.18 FORMIC ACID (aqueous, 0.1 M) - dissolve formic acid (4.6 g) in reagent water (1 L), store at room temperature, replace after 2 years.

8.1.19 FORMIC ACID (aqueous, 0.3 M) - dissolve formic acid (13.8 g) in reagent water (1 L), store at room temperature, replace after 2 years.

8.1.20 FORMIC ACID (aqueous, 5% v/v) - mix 5 mL formic acid with 95 mL reagent water, store at room temperature, replace after 2 years.

8.1.21 FORMIC ACID (methanolic 1:1, 0.1 M formic acid/methanol) - mix equal volumes of methanol and 0.1 M formic acid, store at room temperature, replace after 2 years.

8.1.22 FORMIC ACID (aqueous, 50% v/v) - mix 50 mL formic acid with 50 mL reagent water, store at room temperature, replace after 2 years.

8.1.23 POTASSIUM HYDROXIDE – certified ACS or equivalent, store at room temperature, replace after 2 years.

8.1.24 CARBON - – EnviCarb® 1-M-USP or equivalent, verified by lot number before use, stored at room temperature. Loose carbon allows for better adsorption of interferent organics. Note: The single-laboratory validation laboratory achieved better performance with loose carbon than carbon cartridges. Loose carbon will be used for the multi-laboratory validation to set statistically based method criteria.

8.1.25 NITROGEN – Used for the following purposes: Nitrogen aids in aerosol generation of the ESI liquid spray and is used as collision gas in some MS/MS instruments. The nitrogen used should meet or exceed instrument manufacturer's specifications. In addition, Nitrogen is used to concentrate sample extracts (Ultra High Purity or equivalent).

8.1.26 ARGON – Used as collision gas in some MS/MS instruments. Argon should meet or exceed instrument manufacturer's specifications. Nitrogen gas may be used as the collision gas provided sufficient sensitivity (product ion formation) is achieved.

8.2 REFERENCE MATRICES - Matrices in which PFAS and interfering compounds are not detected by this method. These matrices are to be used to prepare the batch QC samples, LOQ/MDL, and IDOC samples.

8.2.1 Reagent water - purified water, Type I

8.2.2 Solid reference matrix Ottawa Sand or equivalent

8.2.3 Tissue Reference matrix – Cod loin or other animal tissue demonstrated to be PFAS free.

8.3 STANDARD SOLUTIONS – When a compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. PFAS analyte and IS standards commercially purchased in glass ampoules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene containers and are stored at ≤4 °C. Standards for sample fortification generally should be prepared in the smallest volume that can be accurately measured to minimize the addition of excess organic solvent to aqueous samples.

8.3.1 Stock standards and diluted stock standards are stored at ≤4 °C. Prepare a spiking solution, containing the method analytes listed in Table 1, in methanol from prime stocks. The solution is used to prepare the calibration standards and to spike the known reference QC samples that are analyzed with every batch. Quantitative standards containing a mixture of branched and linear isomers must be used for method analytes if they are commercially available. Currently, these include PFOS, PFHxS, NEtFOSAA, and NMeFOSAA.

8.3.2 Calibration standard solutions – A series of calibration solutions containing the target analytes and the Labeled extracted internal standards (EIS) and non-extracted internal standards (NIS) is used to establish the initial calibration of the analytical instrument. Table 4 represents the concentrations of the native, EIS and NIS analytes of the calibration curve. Calibration standard solutions are made using the solution described in section 8.1.16.

8.3.3 ISOTOPE DILUTION EXTRACTED INTERNAL STANDARD (EIS) – Isotopically labelled analogs of the target analytes to be used for the quantification of target analytes. EIS stock standard solutions are purchased in glass ampoules and are stored in accordance with the manufacturer's recommendations. The EIS stock solution to be used for the fortification of samples and QC in accordance with the isotope dilution procedure. Table 2 represents the EIS concentrations and nominal sample amounts added to each field sample and QC element.

8.3.4 ISOTOPE DILUTION NON-EXTRACTED INTERNAL STANDARDS (NIS) – Isotopically labelled analogs to be added post extraction for the measurement of EIS extraction efficiency and is added to the final volume of all extractions. Table 3 represents the EIS concentrations and nominal sample amounts added to each field sample and QC element.

9. Quality Control

9.1 Method Blank

9.1.1 A Method Blank (MB) is required with each extraction batch to confirm that potential background contaminants are not interfering with the identification or quantitation of method analytes. An aliquot of reagent water that is treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents and standards. Prep and analyze a MB for every 20 samples. If the MB produces a peak within the retention time window of any analyte that would prevent the determination of that analyte, determine the source of contamination, and eliminate the interference before processing samples. Background contamination must be reduced to an acceptable level before proceeding. Background from method analytes or other contaminants that interfere with the measurement of method analytes must be below the RL. If the method analytes are detected in the MB at concentrations equal to or greater than this level, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch.

9.2 Laboratory Control Sample (LCS)

9.2.1 Low Level LCS or OPR (Ongoing Precision Recovery) sample is required with each extraction batch. A LLCS or OPR samples is a method blank spiked with known quantities of analytes. The fortified concentration of the LCS is spiked at 2X the LOQ. Default limits of 70-130% of the true value may be used for analytes until sufficient replicates have been analyzed to generate proper control limits. Calculate the percent recovery (%*R*) for each analyte using the equation:

9.2.2 An LCS or OPR (Ongoing Precision Recovery) sample is required with each extraction batch. A LCS or OPR samples is a method blank spiked with known quantities of analytes. The fortified concentration of the LCS is spiked at the midpoint of the calibration curve. Default limits of 70-130% of the true value may be used for analytes until sufficient replicates have been analyzed to generate proper control limits. Calculate the percent recovery (%*R*) for each analyte using the equation:

$$\%R = \frac{A \times 100}{B}$$

Where:

A = measured concentration in the fortified sample
B =fortification concentration.

9.1.1 Where applicable, in the absence of additional sample volume required to perform matrix specific QC, LCSD's are to be extracted and analyzed. The concentration and analyte

recovery criteria for the LCSD must be the same as the batch LCS The RSD's must fall within ≤30% of the true value for medium and high-level replicates, and ≤50% for low level replicates. Calculate the relative percent difference (RPD) for duplicate MSs (MS and MSD) using the equation:

$$RPD = \frac{|LCS - LCSD|}{(LCS + LCSD) / 2} \times 100$$

9.1.2 If the LCS and or LCSD results do not meet these criteria for method analytes, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch.

9.3 Non-extracted Internal Standard Area (NIS)

Each time an initial calibration is performed, use the data from all the initial calibration standards used to meet the linearity test in Section 10.3.3.3 to calculate the mean area response for each of the NIS compounds, using the equation below.

$$\text{Mean Area}_{NISi} = \sum \text{AREA}_{NISi} / n$$

where:

AreaNISi = Area counts for the ith NIS, where i ranges from 1 to 7, for the seven NIS compounds listed in Table 1
n = The number of ICAL standards (the default value is n = 6). If a different number of standards is used for the ICAL, for example, to increase the calibration range or by dropping a point at either end of the range to meet the linearity criterion, change 6 to match the actual number of standards used)

Record the mean areas for each NIS for use in evaluating results for sample analyses. There is no acceptance criterion associated with the mean NIS area data.

9.4 Extracted Internal Standards (EIS)

9.4.1 The EIS standard is fortified into all samples, CCVs, MBs, LCSs, MSs, MSDs, FD, and FRB prior to extraction. It is also added to the CAL standards. The EIS is a means of assessing method performance from extraction to final chromatographic measurement. Calculate the recovery (%R) for the EIS using the following equation:

$$\%R = (A / B) \times 100$$

Where:

A = calculated EIS concentration for the QC or Field Sample
B = fortified concentration of the EIS.

9.4.2 Default limits of 50-150% may be used for analytes until sufficient replicates have been analyzed to generate proper control limits. A low or high percent recovery for a sample, blank, or CCV does not require discarding the analytical data but it may indicate a potential problem with future analytical data. When EIS recovery from a sample, blank, or CCV are outside control limits, check 1) calculations to locate

possible errors, 2) standard solutions for degradation, 3) contamination, and 4) instrument performance. For CCVs and QC elements spiked with all target analytes, if the recovery of the corresponding target analytes meet the acceptance criteria for the EIS in question, the data can be used but all potential biases in the recovery of the EIS must be documented in the sample report. If the associated target analytes do not meet the acceptance criteria, the data must be reanalyzed.

9.5 Matrix Spike (MS/MSD)

9.5.1 Analysis of an MS is prepared one per preparation batch (if required).

9.5.2 Aliquots of field samples that have been fortified with a known concentration of target compounds, prior to sample preparation and extraction, and analyzed to measure the effect of matrix interferences. The use of MS/MSD samples is generally not required in isotope dilution methods because the labeled compounds added to every sample provide more performance data than spiking a single sample in each preparation batch. Aliquots of field samples

9.5.3 Analyte recoveries may exhibit matrix bias. For samples fortified at or above their native concentration, recoveries should range between 50-150%. If the accuracy of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the LCS, the recovery is judged to be matrix biased. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.6 Laboratory Duplicate

9.6.1 FIELD DUPLICATE OR LABORATORY FORTIFIED SAMPLE MATRIX DUPLICATE (FD or MSD) – Within each extraction batch (not to exceed 20 Field Samples), a minimum of one FD or MSD must be analyzed. Duplicates check the precision associated with sample collection, preservation, storage, and laboratory procedures. If method analytes are not routinely observed in Field Samples, an MSD should be analyzed rather than an FD.

9.6.2 Calculate the relative percent difference (*RPD*) for duplicate measurements (*FD1* and *FD2*) using the equation:

$$RPD = \frac{|FD1 - FD2|}{(FD1 + FD2) / 2} \times 100$$

9.6.3 RPDs for FDs should be ≤30%. Greater variability may be observed when FDs have analyte concentrations that are within a factor of 2 of the RL. At these concentrations, FDs should have RPDs that are ≤50%. If the RPD of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the CCV, the recovery is judged to be matrix biased. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.6.4 If an MSD is analyzed instead of a FD, calculate the relative percent difference (RPD) for duplicate MSs (MS and MSD) using the equation:

$$RPD = \frac{|MS - MSD|}{(MS + MSD) / 2} \times 100$$

9.6.5 RPDs for duplicate MSs should be ≤30% for samples fortified at or above their native concentration. Greater variability may be observed when MSs are fortified at analyte concentrations that are within a factor of 2 of the RL. MSs fortified at these concentrations should have RPDs that are ≤50% for samples fortified at or above their native concentration. If the RPD of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the LCSD where applicable, the result is judged to be matrix biased. If no LCSD is present, the associated MS and MSD are to be re-analyzed to determine if any analytical has occurred. If the resulting RPDs are still outside control limits, the result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.7 Bile Salt Interference Check

9.7.1 The laboratory must analyze a TDCA standard after the initial calibration, prior to the analysis of tissue samples, to check for interferences caused by bile salts. If an interference is present, the chromatographic conditions must be modified to eliminate the interference from TDCA (e.g., changing the retention time of TDCA such that it falls outside the

9.8 Initial Calibration Verification (ICV)

9.8.1 After each ICAL, analyze a QCS sample from a source different from the source of the CAL standards. If a second vendor is not available, then a different lot of the standard should be used. The QCS should be prepared and analyzed just like a CCV. Acceptance criteria for the QCS are identical to the CCVs; the calculated amount for each analyte must be ± 30% of the expected value. If measured analyte concentrations are not of acceptable accuracy, check the entire analytical procedure to locate and correct the problem.

9.9 Instrument Sensitivity Check (ISC)

9.9.1 At the start of each 12-hour shift, analyze a standard at the LOQ. The signal-to-noise ratio of the ISC standard must be greater than or equal to 3:1. If the requirements cannot be met, the problem must be corrected before analyses can proceed.

9.10 Continuing Calibration Verification (CCV)

9.10.1 CCV Standards must be analyzed at the beginning of each analysis batch, after every 10 Field Samples, and at the end of the analysis batch.

9.10.2 The recovery of native and isotopically labeled compounds for the CVs must be within 70 - 130%

9.11 Method-specific Quality Control Samples

9.11.1 Instrument Blank – During the analysis of a batch of samples, a solvent blank is analyzed after samples containing high level of target compounds (e.g., calibration, CV) to monitor carryover from the previous injection. The injection blank consists of the solution in Section 8.1.16 fortified with the EIS and NIS for quantitation purposes.

9.12 Example Method Sequence

- INSTRUMENT BLANK
- INSTRUMENT SENSITIVITY CHECK
- CALIBRATION VERIFICATION STANDARD
- QUALITATIVE IDENTIFICATION STANDARDS
- TDCA STANDARD (only if analyzing tissues)
- INSTRUMENT BLANK
- METHOD BLANK
- LOW-LEVEL LCS/OPR
- OPR/LCS
- SAMPLE (10 or fewer)
- CALIBRATION VERIFICATION STANDARD
- INSTRUMENT BLANK
- SAMPLE (10 or fewer)
- CALIBRATION VERIFICATION STANDARD
- INSTRUMENT BLANK

10. Procedure

10.1 Equipment Set-up

10.1.1 This procedure may be performed manually or in an automated mode using a robotic or automatic sample preparation device. If an automated system is used to prepare samples, follow the manufacturer's operating instructions, but all extraction and elution steps must be the same as in the manual procedure. Extraction and/or elution steps may not be changed or omitted to accommodate the use of an automated system. If an automated system is used, the MBs should be rotated among the ports to ensure that all the valves and tubing meet the MB requirements.

10.1.2 Some of the PFAS's adsorb to surfaces, including polypropylene. Therefore, the aqueous sample bottles must be rinsed with the elution solvent whether extractions are performed manually or by automation. The bottle rinse is passed through the cartridge to elute the method analytes and is then collected.

10.1.3 The SPE cartridges and sample bottles described in this section are designed as single use items and should be discarded after use. They may not be refurbished for reuse in subsequent analyses.

10.1.4 All SPE apparatus, including manifolds, tubing and sample ports must be thoroughly rinsed following each use with 1% methanolic ammonium hydroxide, followed by Methanol and then DI water. Additionally, sample manifold ports and transfer tubing should be inspected regularly for signs of wear and/or

discoloration. When such observations are made, the associated components should be replaced.

10.1.5 Prior to the start of any extraction, sample site information must be evaluated for any potentially high level PFAS concentrations or sample matrix irregularities that may impact the extraction process. If such samples are identified, aqueous samples may be pre-screened via direct aqueous injection prior to analysis to estimate the potential PFAS concentrations present.

10.1.6 To perform a direct aqueous injection (DAI) screen, the sample should be inverted several times to try and evenly disperse any organic matter present. A 1 ml aliquot (or less depending on the matrix) is to be taken from the parent sample, volume adjusted to 1 ml with reagent water if less than 1ml, fortified with EIS and NIS spiking solutions to match the concentrations of an extracted sample (typically 5 µl per 1 ml DAI), and then analyzed under the same analytical conditions as field samples.

10.2 Sample Preparation of Aqueous Samples

10.2.1 Samples are preserved, collected, and stored as presented in Section 6.

10.2.2 Determine sample volume. Weigh all samples to the nearest 1g. If visible sediment is present, centrifuge and decant into a new HDPE bottle and record the weight of the new container.

NOTE: Some of the PFAS's adsorb to surfaces, thus the sample volume may not be transferred to a graduated cylinder for volume measurement.

10.2.3 The MB, LCS and FRB may be prepared by measuring reagent water with a polypropylene graduated cylinder or filling an HDPE sample bottle near the top.

10.2.4 Check that the pH is 6.5 ± 0.5. If necessary, adjust pH with 50% formic acid or ammonium hydroxide and 3% aqueous ammonium hydroxide. The extract is now ready for solid-phase extraction (SPE) and cleanup.

10.2.5 Add 20 µL of the EIS to each sample and QC, cap and invert to mix.

10.2.6 If the sample is an LCS, LCSD, MS, or MSD, add the necessary amount of analyte PDS. Cap and invert each sample to mix.

10.3 Sample Prep and Extraction Protocol for Solids.

10.3.1 Homogenize and weigh 5 grams of sample (measured to the nearest hundredth of a gram) into a 50 ml polypropylene centrifuge tube. For laboratory control blanks and spikes, 5 grams of clean sand is used.

10.3.1.1 For Biosolids and other complex matrices, a small aliquot may be required due to co-extracted matrix interferences.

10.3.1.2 For batch QC samples using 5 g of reference solid, add 2.5 g of reagent water. The addition of reagent water to the sand provides a matrix closer in composition to real-world samples.

10.3.2 Add 20 µL of the EIS to each sample and QC.

10.3.3 If the sample is an LCS, LCSD, MS, or MSD, add the necessary amount of analyte PDS. Cap and invert each sample to mix.

10.3.4 Vortex the samples to evenly disperse the spiking solutions and allow to equilibrate for 30 minutes.

10.3.5 To all samples, add 10 ml of 0.3% methanolic ammonium hydroxide, cap, vortex for 25 seconds.

10.3.6 Following mixing, shake each sample for 30 minutes on a shaker table.

10.3.7 Centrifuge each sample at 2800RPM for 10 minutes.

10.3.8 Remove the supernatant and transfer to a clean 50 ml polypropylene centrifuge tube.

10.3.9 Repeat steps 10.3.4 to 10.3.7, with 15 ml of 0.3% methanolic ammonium hydroxide, combining the supernatants.

10.3.10 Add 5ml of 0.3% methanolic ammonium hydroxide to the sample, vortex for 25 seconds and centrifuge each sample at 2800RPM for 10 minutes.

10.3.11 Remove the supernatant and transfer to the same 50 ml polypropylene centrifuge tube containing eluates from the previous cycles.

10.3.12 Add 10 mg of carbon to the combined extract, mix by occasional hand shaking for no more than five minutes and then centrifuge at 2800 rpm for 10 minutes. Immediately decant the extract into a 50 ml polypropylene centrifuge tube.

10.3.13 Dilute to approximately 35 mL with reagent water. Samples containing more than 50% water may yield extracts that are greater than 35 mL in volume; therefore, do not add water to these. Determine the water content in the sample as follows (percent moisture is determined from the % solids):

Water Content in Sample = (Sample Weight * Percent moisture) / 100

10.3.14 Concentrate each extract at approximately 55 ºC with a gentle N2 flow to a final volume that is based on the water content of the sample (see table below). Allow extracts to concentrate for 10 minutes, then mix (by vortex if the volume is < 20. Continue concentrating and mixing every 5 minutes until the extract has been reduced to the required volume as specified in the table below. If the extract volume appears to stop dropping, the concentration must be stopped and the volume at which it was stopped recorded.

Water Content in Sample	Concentrated Final Volume
< 5 grams	15 ml
5-8 grams	15-20 ml
8-9 grams	20-22.5 ml
9-10 grams	22.5-25 ml

10.3.15 Add 40 - 50 mL of reagent water to the extract and vortex. Check that the pH is 6.5 ±0.5 and adjust as necessary with 50% formic acid or 30% ammonium hydroxide, or with 5% formic acid and 3% aqueous ammonium hydroxide. The extracts are ready for SPE and cleanup.

10.4 Sample Prep and Extraction Protocol for Oils.

10.4.1 Weigh 1-2 grams of sample (measured to the nearest hundredth of a gram) into a 50 ml polypropylene centrifuge tube. For laboratory control blanks and spikes, 1 grams of mineral oil is used.

10.4.1.1 For batch QC samples use 1 g of reference oil.

10.4.2 Add 20 µL of the EIS to each sample and QC.

10.4.3 If the sample is an LCS, LCSD, MS, or MSD, add the necessary amount of analyte PDS. Cap and invert each sample to mix.

10.4.4 Vortex the samples to evenly disperse the spiking solutions and allow to equilibrate for 30 minutes.

10.4.5 To all samples, add 10 ml of 0.3% methanolic ammonium hydroxide, cap, vortex for 25 seconds.

10.4.6 Following mixing, shake each sample for 30 minutes on a shaker table.

10.4.7 Centrifuge each sample at 2800RPM for 10 minutes.

10.4.8 Remove the supernatant and transfer to a clean 50 ml polypropylene centrifuge tube.

10.4.9 Repeat steps 10.3.4 to 10.3.7, with 15 ml of 0.3% methanolic ammonium hydroxide, combining the supernatants.

10.4.10 Add 5ml of 0.3% methanolic ammonium hydroxide to the sample, vortex for 25 seconds and centrifuge each sample at 2800RPM for 10 minutes.

10.4.11 Remove the supernatant and transfer to the same 50 ml polypropylene centrifuge tube containing eluates from the previous cycles.

10.4.12 Add 10 mg of carbon to the combined extract, mix by occasional hand shaking for no more than five minutes and then centrifuge at 2800 rpm for 10 minutes. Immediately decant the extract into a 50 ml polypropylene centrifuge tube.

10.5 Sample Prep and Extraction Protocol for Tissues.

10.5.1 Homogenize and weigh 2 grams of sample (measured to the nearest hundredth of a gram) into a 50 ml polypropylene centrifuge tube. For laboratory control blanks and spikes, 2 grams of clean tissue is used.

10.5.2 Add 20 µL of the EIS PDS to each sample and QC.

10.5.3 If the sample is an LCS, LCSD, MS, or MSD, add the necessary amount of analyte PDS. Cap and invert each sample to mix.

10.5.4 Add 10 mL of 0.05M KOH in methanol to each sample. Vortex to disperse the tissue then place tubes on a mixing table to extract for at 16 hours. Centrifuge at 2800 rpm for 10 minutes and collect the supernatant in a 50-mL polypropylene centrifuge tube.

10.5.5 Add 10 mL of acetonitrile to remaining tissue in the 50-mL centrifuge tube, vortex to mix and disperse the tissue. Sonicate for 30 minutes. Centrifuge at 2800 rpm for 10 minutes and collect the supernatant, adding it to the 50-mL centrifuge tube containing the initial extract.

10.5.6 Add 5 mL of 0.05M KOH in methanol to the remaining sample in each centrifuge tube. Vortex to disperse the tissue and hand mix briefly. Centrifuge at 2800 rpm

for 10 minutes and collect the supernatant, adding it to the 50-mL centrifuge tube containing the first two extracts.

10.5.7 Add 10 mg of carbon to the combined extract, mix by occasional hand shaking over a period of no more than five minutes and then centrifuge at 2800 rpm for 10 minutes. Immediately decant the extract into a 50-mL centrifuge tube.

10.5.8 Add 1 mL of reagent water to each tube and concentrate each extract at approximately 55 ºC with a gentle N2 flow to a final volume of 2.5 ml.

10.5.9 Add reagent water to each evaporation/concentrator tube to dilute the extracts to 50 mL. Check that the pH = 6.5 ± 0.5 and adjust as needed with 50% formic acid, or ammonium hydroxide or with 5% formic acid and 3% aqueous ammonium hydroxide. The extracts are ready for SPE and cleanup.

10.6 SPE Extract: All matrices

10.6.1 Pack clean silanized glass wool to half the height of the WAX SPE cartridge barrel.

10.6.2 Pre-condition the cartridges by washing them with 3 X 5 mL of 1% methanolic ammonium hydroxide, discarding the wash volumes.

10.6.3 Rinse the cartridge with 5 mL of 0.3M formic acid, allowing the cartridge to drain using gravity only, discarding the rinse volume. Do not allow the cartridge to go dry

10.6.4 Adjust the vacuum so that the approximate flow rate is ~5 mL/min and load the sample across the cartridge. Do not allow the cartridge to go dry before all the sample has passed through.

10.6.5 Once all the sample has passed across the cartridge, rinse the walls of the reservoir with 2 X 5 mL reagent water, loading the rinse across the cartridge.

10.6.6 Rinse the walls of the reservoir with 5 mL of 1:1 0.1M formic acid/methanol and pass the rinse through the cartridge using vacuum. Dry the cartridge by pulling air through for 15 seconds.

10.6.7 Rinse the inside of the sample bottle with 5 mL of 1% methanolic ammonium hydroxide. Use vacuum to pull the elution solvent through the cartridge and into the collection tubes. When the cartridge bed and glass wool are submerged, stop the cartridge flow by closing the valve, keeping the sorbent bed and wool submerged.

10.6.8 Let the wetted sorbent bed and wool soak for 1 minute.

10.6.9 Open the cartridge valve and collect the eluate into a 15 ml polypropylene collection tube.

10.6.10 Add 25 µL of concentrated acetic acid to each sample eluted in the collection tubes and vortex to mix.

10.6.11 Add 10 mg of carbon to each sample and batch QC extract, using a 10-mg scoop. Handshake occasionally for no more than 5 minutes. It is important to minimize the time the sample extract is in contact with the carbon. Immediately vortex (30 seconds) and centrifuge at 2800 rpm for 10 minutes.

10.6.12 Add NIS solution to a clean collection tube. Place a syringe filter (25-mm filter, 0.2-µm nylon membrane) on a 5-mL polypropylene syringe. Take the plunger out and carefully decant the sample supernatant into the syringe barrel. Replace the

plunger and filter the entire extract into the new collection tube containing the NIS.

10.6.13 Vortex to mix and transfer a portion of the extract into a .7-mL polypropylene LC vial for LC-MS/MS analysis. Cap the collection tube containing the remaining extract and store at 4 °C

10.7 Sample Volume Determination

10.7.1 If using weight to determine volume, weigh the empty bottle to the nearest 1 g and determine the sample weight by subtraction of the empty bottle weight from the original sample weight. Assume a sample density of 1.0 g/mL. In either case, the sample volume will be used in the final calculations of the analyte concentration.

10.8 Initial Calibration - Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed. After the initial calibration is successful, a CCV is required at the beginning and end of each period in which analyses are performed, and after every tenth Field Sample.

10.8.1 ESI-MS/MS TUNE

10.8.1.1 Calibrate the mass scale of the MS with the calibration compounds and procedures prescribed by the manufacturer.

10.8.1.2 Optimize the [M-H]- or [M-CO_2]- for each method analyte by infusing approximately 0.5-1.0 µg/mL of each analyte (prepared in the initial mobile phase conditions) directly into the MS at the chosen LC mobile phase flow rate (0.4 mL/min). This tune can be done on a mix of the method analytes. The MS parameters (voltages, temperatures, gas flows, etc.) are varied until optimal analyte responses are determined. The method analytes may have different optima requiring some compromise between the optima.

The Mass spec conditions found in Table 7 show the Sciex Triple Quad 5500+ operation conditions used in this method.

10.8.1.3 Optimize the product ion for each analyte by infusing approximately 0.5-1.0 µg/mL of each analyte (prepared in the initial mobile phase conditions) directly into the MS at the chosen LC mobile phase flow rate (approximately 0.4 mL/min). This tune can be done on a mix of the method analytes. The MS/MS parameters (collision gas pressure, collision energy, etc.) are varied until optimal analyte responses are determined. Typically, the carboxylic acids have very similar MS/MS conditions, and the sulfonic acids have similar MS/MS conditions.

The conditions found on table 5 are representative of expected tune optimizations for each analyte. If conditions other the ones close to the values provided in table 5 are achieved, the process should be re-performed and/or instrument maintenance performed to resolve the problem.

10.8.2 Establish LC operating parameters that optimize resolution and peak shape. Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted.

Table 6 represents the operation conditions of a Sciex Exion LC system when running this method.

10.8.3 Inject 2µl of a mid-level CAL standard under LC/MS conditions to obtain the retention times of each method analyte. Divide the chromatogram into retention time windows each of which contains one or more chromatographic peaks. During MS/MS analysis, fragment a small number of selected precursor ions ([M-H]-) for the analytes in each window and choose the most abundant product ion. For maximum sensitivity, small mass windows of ±0.5 daltons around the product ion mass were used for quantitation.

10.8.4 Inject a mid-level CAL standard under optimized LC/MS/MS conditions to ensure that each method analyte is observed in its MS/MS window and that there are at least 10 scans across the peak for optimum precision.

NOTE: PFHxS, PFOS, NMeFOSAA, and NEtFOSAA have multiple chromatographic peaks using the LC conditions in Table 7 due to chromatographic resolution of the linear and branched isomers of these compounds. Most PFAS's are produced by two different processes. One process gives rise to linear PFAS's only while the other process produces both linear and branched isomers. Thus, both branched and linear PFAS's can potentially be found in the environment. For the aforementioned compounds that give rise to more than one peak, all the chromatographic peaks observed in the standard must be integrated and the areas totaled. Chromatographic peaks in a sample must be integrated in the same way as the CAL standard.

10.8.5 Prepare a set of CAL standards as outlined in table 5. The lowest concentration CAL standard must be at or below the LOQ.

10.8.6 The LC/MS/MS system is calibrated using the isotope dilution technique. Target analytes are quantitated against their isotopically labeled analog (Extracted Internal Standard) where commercially available. If a labeled analog is not commercially available, the extracted internal standard with the closest retention time and /or closest chemical similarity is to be used. Use the LC/MS/MS data system software to generate a linear regression or quadratic calibration curve for each of the analytes. This curve must always be forced through zero and may be concentration weighted, if necessary. Forcing zero allows for a better estimate of the background levels of method analytes. A minimum of 6 calibration points are required for a linear or quadratic calibration model.

10.8.7 **CALIBRATION ACCEPTANCE CRITERIA** – A linear fit is acceptable if the calculated RSD or RSE for each target analyte is ≤20%. If linear or Quadratic regressions are used, coefficient of determination (r^2) values must be greater than 0.99. When quantitated using the initial calibration curve, each calibration point at or above the LOQ for each analyte must calculate to be within 70-130% of its true value. The calculate value of each EIS analyte must be within 50-150% of its true value. If these criteria cannot be met, corrective action is taken to reanalyze the CAL standards, restrict the range of calibration.

10.8.8 **Bile salts interference check -** The laboratory must analyze a TDCA standard after the initial calibration, prior to the analysis of tissue samples, to check for interferences caused by bile salts. If an interference is present, the chromatographic conditions must be modified to eliminate the interference from TDCA (e.g., changing the retention time of TDCA such that it falls outside the

retention window for PFOS by at least one minute), and the initial calibration repeated.

10.9 CONTINUING CALIBRATION CHECK (CCV) – Minimum daily calibration verification is as follows. Verify the initial calibration at the beginning and end of each group of analyses, and after every tenth sample during analyses. In this context, a "sample" is considered to be a Field Sample. MBs, CCVs, LCSs, MSs, FDs FRBs and MSDs are not counted as samples. The beginning CCV of each analysis batch must be at or below the RL in order to verify instrument sensitivity prior to any analyses. If standards have been prepared such that all low CAL points are not in the same CAL solution, it may be necessary to analyze two CAL standards to meet this requirement. Alternatively, the analyte concentrations in the analyte PDS may be customized to meet these criteria. Subsequent CCVs should alternate between a medium and Low concentration CAL standard.

10.9.1 Inject an aliquot of the appropriate concentration CAL standard and analyze with the same conditions used during the initial calibration.

10.9.2 Calculate the concentration of each analyte and EIS in the CCV. The calculated amount for each native and EIS analyte for medium level CCVs must be within ± 30% of the true. If these conditions do not exist, then all data for the problem analyte must be considered invalid, and remedial action should be taken which may require recalibration. Any Field or QC Samples that have been analyzed since the last acceptable calibration verification should be reanalyzed after adequate calibration has been restored, with the following exception. If the CCV fails because the calculated concentration is greater than 130% for a particular method analyte, and Field Sample extracts show no detection for that method analyte, non-detects may be reported without re-analysis.

10.9.3 REMEDIAL ACTION – Failure to meet CCV QC performance criteria may require remedial action. Major maintenance, such as cleaning the electrospray probe, atmospheric pressure ionization source, cleaning the mass analyzer, replacing the LC column, etc., requires recalibration and verification of sensitivity by analyzing a CCV at or below the LOQ.

10.10 EXTRACT ANALYSIS

10.10.1 The same operating conditions used for the initial calibration and summarized in Tables 6 and 7 are to be used.

10.10.2 Prior to analysis of sample extracts, the Instrument mass calibration verification must be performed using standards whose mass range brackets the masses of interest and performed in the negative ion mode. The mass calibration is verified if the calculated mass is within ± .2 daltons of the specified mass.

10.10.3 Establish an appropriate retention time window for each analyte. This should be based on measurements of actual retention time variation for each method analyte in CAL standard solutions analyzed on the LC over the course of time. A value of plus or minus three times the standard deviation of the retention time obtained for each method analyte while establishing the initial calibration can be used to calculate a suggested window size. However, the experience of the analyst should weigh heavily on the determination of the appropriate retention window size.

10.10.4 Calibrate the system by either the analysis of a calibration curve or by confirming the initial calibration is still valid by analyzing a CCV.

10.10.5 Begin analyzing Field Samples, including QC samples, at their appropriate frequency by injecting the same size aliquots under the same conditions used to analyze the CAL standards.

10.10.6 For concentrations at or above the method LOQ, the total (branched and linear isomer) quantification ion response to the total (branched and linear isomer) confirmation ion response ratio must fall within ± 50% of the ratio observed in the midpoint initial calibration standard.

10.10.7 At the conclusion of data acquisition, use the same software that was used in the calibration procedure to identify peaks of interest in predetermined retention time windows. Use the data system software to examine the ion abundances of the peaks in the chromatogram. Identify an analyte by comparison of its retention time with that of the corresponding method analyte peak in a reference standard.

10.10.7.1 Method analyte, EIS analyte, and NIS analyte RTs must fall within 0.4 minutes of the predicted retention times from the midpoint standard of the ICAL or initial daily CV, whichever was used to establish the RT window position for the analytical batch. All branched isomer peaks identified in either the calibration standard or the qualitative (technical grade) standard must fall within in the retention time window for that analyte.

10.10.7.2 For all method analytes with exact corresponding isotopically labeled analogs, method analytes must elute within 0.1 minutes of the associated EIS.

10.10.8 The analyst must not extrapolate beyond the established calibration range. If an analyte peak area exceeds the range of the initial calibration curve, the sample should be re-extracted with a reduced sample volume in order to bring the out of range target analytes into the calibration range. If a smaller sample size would not be representative of the entire sample, the following options are recommended. Re-extract an additional aliquot of sufficient size to ensure that it is representative of the entire sample. Spike it with a higher concentration of internal standard. Prior to LC/MS analysis, dilute the sample so that it has a concentration of internal standard equivalent to that present in the calibration standard. Then, analyze the diluted extract.3

10.10.9 In instances where re-extraction is not an option, dilute a subsample of the sample extract with 0.1% acetic acid by a factor no greater than 10x adjust the amount of the NIS in the diluted extract, and analyze the diluted extract. If the responses for each EIS in the diluted extract meet the S/N and retention time, and the EIS recoveries from the analysis of the diluted extract are greater than 5%, then the compounds associated with those EISs may be quantified using isotope dilution. Use the EIS recoveries from the original analysis to select the dilution factor, with the objective of keeping the EIS recoveries in the dilution above that 5% lower limit. If the adjusted EIS recoveries are below 5%, the dilution is assumed invalid. If the adjusted EIS recoveries are greater than 5%, adjust the compound concentrations, detection limits, and minimum levels to account for the dilution.

11. Data Evaluation, Calculations and Reporting

11.1 Complete chromatographic resolution is not necessary for accurate and precise measurements of analyte concentrations using MS/MS. In validating this method, concentrations were calculated by measuring the product ions listed in Table 9.

11.2 Calculate analyte concentrations using the multipoint calibration established in Section 10.9. Do not use daily calibration verification data to quantitate analytes in samples. Adjust final analyte concentrations to reflect the actual sample volume determined in Section 10.8

C_{ex} = (Area of target analyte * Concentration of Labeled analog) / (area of labeled analog * CF)

C_s = (C_{ex} / sample volume in ml) * 1000

C_{ex} = The concentration of the analyte in the extract
CF = calibration factor from calibration.

11.3 Prior to reporting the data, the chromatogram should be reviewed for any incorrect peak identification or poor integration.

11.4 PFHxS, PFOS, PFOA, NMeFOSAA, and NEtFOSAA have multiple chromatographic peaks using the LC conditions in Table 7 due to the linear and branch isomers of these compounds (Sect. 10.10.4.). The areas of all the linear and branched isomer peaks observed in the CAL standards for each of these analytes must be summed and the concentrations reported as a total for each of these analytes.

11.5 Calculations must utilize all available digits of precision, but final reported concentrations should be rounded to an appropriate number of significant figures (one digit of uncertainty), typically two, and not more than three significant figures.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 Section 9.0 outlines sample batch QC acceptance criteria. If non-compliant organic compound results are to be reported, the Organic Section Head and/or the Laboratory Director, and the Operations Manager must approve the reporting of these results. The laboratory Project Manager shall be notified and may choose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst, Data Reviewer, or Department Supervisor performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

12.2 All results for the organic compounds of interest are reportable without qualification if extraction and analytical holding times are met, preservation requirements (including cooler temperatures) are met, all QC criteria are met, and matrix interference is not suspected during extraction or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

13.1.1 The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

13.2.1 Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.2 The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

14.1 Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

14.2 This method utilizes SPE to extract analytes from water. It requires the use of very small volumes of organic solvent and very small quantities of pure analytes, thereby minimizing the potential hazards to both the analyst and the environment as compared to the use of large volumes of organic solvents in conventional liquid-liquid extractions.

14.3 The analytical procedures described in this method generate relatively small amounts of waste since only small amounts of reagents and solvents are used. The matrices of concern are finished drinking water or source water. However, laboratory waste management practices must be conducted consistent with all applicable rules and regulations, and that laboratories protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Also, compliance is required with any sewage discharge permits and regulations, particularly the hazardous waste identification rules and land disposal restrictions.

15. Referenced Documents

Chemical Hygiene Plan – ID 2124

SOP ID 1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

SOP ID 1739 Demonstration of Capability (DOC) Generation SOP

SOP ID 1728 Hazardous Waste Management and Disposal SOP

16. Attachments

Table 1: Names, Abbreviations, and CAS Registry Numbers for Target PFAS, Extracted Internal Standards and Non-extracted Internal Standards

Parameter	Acronym	CAS
PER- and POLYFLUOROALKYLETHER CARBOXYLIC ACIDS (PFECAs)		
Tetrafluoro-2-(heptafluoropropoxy)propanoic acid	HFPO-DA	13252-13-6
4,8-dioxa-3H-perfluorononanoic acid	ADONA	919005-14-4
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6
PERFLUOROALKYLCARBOXILIC ACIDS (PFCAs)		
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnA	2058-94-8
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluorotetradecanoic acid	PFTeDA	376-06-7
PERFLUOROALKYL SULFONIC ACIDS (PFASs)		
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoS	79780-39-5
CHLORO-PERFLUOROALKYLSULFONATE		
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid	9Cl-PF3ONS	756426-58-1
FLUOROTELOMER CARBOXYLIC ACIDS		
3-Perfluoropropyl propanoic acid	3:3FTCA	356-02-5
2H,2H,3H,3H-Perfluorooctanoic acid	5:3FTCA	914637-49-3
Perfluoroheptyl propanoic acid	7:3FTCA	812-70-4
PERFLUOROOCTANESULFONAMIDES		

Perfluorooctanesulfonamide	PFOSA	754-91-6
N-methylperfluoro-1-octanesulfonamide	NMeFOSA	31506-32-8
N-ethylperfluoro-1-octanesulfonamide	NEtFOSA	4151-50-2
PERFLUOROCTANE SULFONAMIDE ETHANOLS		
N-Methyl perfluorooctanesulfonamidoethanol	NMeFOSE	24448-09-7
N-ethyl perfluorooctanesulfonamidoethanol	NEtFOSE	1691-99-2
TELOMER SULFONIC ACIDS		
1H,1H,2H,2H-perfluorohexanesulfonic acid (4:2)	4:2FTS	757124-72-4
1H,1H,2H,2H-perfluorooctanesulfonic acid (6:2)	6:2FTS	27619-97-2
1H,1H,2H,2H-perfluorodecanesulfonic acid (8:2)	8:2FTS	39108-34-4
PERFLUOROOCTANESULFONAMIDOACETIC ACIDS		
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
PERFLUOROETHER AND POLYETHER CARBOXYLIC ACIDS		
Perfluoro-3-methoxypropanoic acid	PFMPA	377-73-1
Perfluoro-4-methoxybutanoic acid	PFMBA	863090-89-5
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	113507-82-7
Nonafluoro-3,6-dioxaheptanoic acid	NFDHA	151772-58-6

Table 2: Stock and Nominal Extracted Internal Standard Concentrations

Isotope Labeled Standard	Conc. of EIS Stock (ng/mL)	Nominal amount of EIS added to extracts (ng)
M4PFBA	2000	40
M5PFPeA	1000	20
M5PFHxA	500	10
M4PFHpA	500	10
M8PFOA	500	10
M9PFNA	250	5
M6PFDA	250	5
M7PFUdA	250	5
MPFDoA	250	5
M2PFTeDA	250	5
M3PFBS	466	9.32
M3PFHxS	474	9.48
M8PFOS	479	9.58
M2-4:2FTS	938	18.8
M2-6:2FTS	951	19
M2-8:2FTS	960	19.2
M8FOSA	500	10
d3-N-MeFOSA	500	10
d5-N-EtFOSA	500	10
d3-N-MeFOSAA	1000	20
d5-N-EtFOSAA	1000	20
d7-N-MeFOSE	5000	100
d9-N-EtFOSE	5000	100
M3HFPO-DA	2000	40

Table 3: Stock and Nominal Non-Extracted Internal Standard Concentrations

Isotope Labeled Standard	Conc. of EIS Stock (ng/mL)	Nominal amount of EIS added to extracts (ng)
M3PFBA	1000	40
M2PFHxA	500	10
M4PFOA	500	10
M5PFNA	250	5
M2PFDA	250	5
18O2PFHxS	474	9.48
M4PFOS	479	9.58

Table 4: Initial Calibration levels and Concentrations

Analyte	Cal A	Cal B (LOQ)	CAL C	Cal D	Cal E (CCV)	Cal F	Cal G	Cal H	Cal I
PFBA	.4	.8	2	5	10	20	50	250	500
PFPeA	.2	.4	1	2.5	5	10	25	125	250
PFHxA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
PFHpA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
PFOA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
PFNA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
PFDA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
PFUnA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
PFDoA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
PFTrDA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
PFTA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
PFBS	0.089	0.177	0.444	1.11	2.22	4.44	11.1	55.4	111
PFPeS	0.094	0.188	0.471	1.18	2.35	4.71	11.8	58.8	118
PFHxS	0.091	0.183	0.457	1.14	2.29	4.57	11.4	57.1	114
PFHpS	0.095	0.191	0.477	1.19	2.38	4.77	11.9	59.6	119
PFOS	0.093	0.186	0.464	1.16	2.32	4.64	11.6	58	116
PFNS	0.096	0.192	0.481	1.20	2.41	4.81	12	60.1	120
PFDS	0.097	0.193	0.483	1.21	2.41	4.83	12.1	60.3	121
PFDOS	0.097	0.194	0.485	1.21	2.43	4.85	12.1	60.6	121.
4:2FTS	0.375	0.75	1.88	4.69	9.38	18.8	46.9	234	469
6:2FTS	0.38	0.76	1.9	4.75	9.5	19	47.5	238	475
8:2FTS	0.384	0.768	1.92	4.8	9.6	19.2	48	240	480
PFOSA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
NMeFOSA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
NEtFOSA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
NMeFOSAA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
NEtFOSAA	.1	.2	.5	1.25	2.5	5	12.5	62.5	125
NMeFOSE	1	2	5	12.5	25	50	125	625	1250
NEtFOSE	1	2	5	12.5	25	50	125	625	1250
HFPO-DA	.4	.8	2	5	10	20	50	250	500
ADONA	0.378	0.756	1.89	4.73	9.45	18.9	47.3	236	473
9Cl-PFONS	0.374	0.748	1.87	4.68	9.35	18.7	46.8	234	468
11Cl-PFOUdS	0.378	0.756	1.89	4.73	9.45	18.9	47.3	236	473
PFMPA	.2	.4	1	2.5	5	10	25	125	250
PFMBA	.2	.4	1	2.5	5	10	25	125	250
PFEESA	0.178	0.356	0.89	2.23	4.45	8.9	22.3	111	223
NFDHA	.2	.4	1	2.5	5	10	25	125	250

3:3FTCA	.5	1	2.5	6.25	12.5	25	62.5	312	624
5:3FTCA	2.5	5	12.5	31.3	62.5	125	312	1560	3120
7:3FTCA	2.5	5	12.5	31.3	62.5	125	312	1560	3125
M4PFBA	10	10	10	10	10	10	10	10	10
M5PFPeA	5	5	5	5	5	5	5	5	5
M5PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
M4PFHpA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
M8PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
M9PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
M6PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
M7PFUdA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
MPFDoA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
M2PFTeDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
M3PFBS	2.33	2.33	2.33	2.33	2.33	2.33	2.33	2.33	2.33
M3PFHxS	2.37	2.37	2.37	2.37	2.37	2.37	2.37	2.37	2.37
M8PFOS	2.4	2.4	2.4	2.4	2.4	2.4	2.4	2.4	2.4
M2-4:2FTS	4.69	4.69	4.69	4.69	4.69	4.69	4.69	4.69	4.69
M2-6:2FTS	4.76	4.76	4.76	4.76	4.76	4.76	4.76	4.76	4.76
M2-8:2FTS	4.8	4.8	4.8	4.8	4.8	4.8	4.8	4.8	4.8
M8FOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d3-N-MeFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d5-N-EtFOSA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d3-N-MeFOSAA	5	5	5	5	5	5	5	5	5
d5-N-EtFOSAA	5	5	5	5	5	5	5	5	5
d7-N-MeFOSE	25	25	25	25	25	25	25	25	25
d9-N-EtFOSE	25	25	25	25	25	25	25	25	25
M3HFPO-DA	10	10	10	10	10	10	10	10	10
M3PFBA	5	5	5	5	5	5	5	5	5
M2PFHxA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
M4PFOA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
M5PFNA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
M2PFDA	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25	1.25
18O2PFHxS	2.37	2.37	2.37	2.37	2.37	2.37	2.37	2.37	2.37
M4PFOS	2.4	2.4	2.4	2.4	2.4	2.4	2.4	2.4	2.4

Table 5: Expected Mass Transitions and instrument conditions.

Q1	Q2	Analyte	DP Volts	CE Volts
213.032	169.022	PFBA	-50	-14
263.039	219.03	PFPeA	-55	-12
263.039	68.9	PFPeA_2	-55	-55
313.047	269.037	PFHxA	-45	-12
313.047	119	PFHxA_2	-45	-28
363.055	319.045	PFHpA	-60	-12
363.055	169.022	PFHpA_2	-60	-24
413.063	369.053	PFOA	-65	-14
413.063	169.022	PFOA_2	-65	-23
463.071	419.061	PFNA	-70	-14
463.071	219.03	PFNA_2	-70	-24
513.078	469.069	PFDA	-80	-16
513.078	219.03	PFDA_2	-80	-30
563.086	519.076	PFUnA	-85	-18
563.086	269.037	PFUnA_2	-85	-25
613.094	569.084	PFDoA	-85	-18
613.094	319.045	PFDoA_2	-85	-28
663.102	619.092	PFTrDA	-85	-20
663.102	169.022	PFTrDA_2	-85	-36
713.11	669.1	PFTA	-70	-22
713.11	169.022	PFTA_2	-70	-38
299.092	80.062	PFBS	-100	-65
299.092	99.061	PFBS_2	-100	-40
349.1	80.062	PFPeS	-100	-75
349.1	99.061	PFPeS_2	-100	-60
399.107	80.062	PFHxS	-120	-75
399.107	99.061	PFHxS_2	-120	-80
449.115	80.062	PFHpS	-140	-95
449.115	99.061	PFHpS_2	-140	-80
499.113	80.062	PFOS	-145	-108
499.113	99.061	PFOS_2	-145	-85
549.131	80.062	PFNS	-180	-100
549.131	99.061	PFNS_2	-180	-100
599.139	80.062	PFDS	-170	-110
599.138	99.061	PFDS_2	-170	-100
699.154	80.062	PFDoS	-160	-150
699.154	99.061	PFDoS_2	-160	-130

327.146	307.139	4:2FTS	-100	-28
327.146	81.07	4:2FTS_2	-100	-50
427.161	407.155	6:2FTS	-120	-33
427.161	81.07	6:2FTS_2	-120	-65
527.177	507.17	8:2FTS	-140	-39
527.177	81.07	8:2FTS_2	-140	-85
498.146	78.07	FOSA	-150	-90
498.146	478	FOSA_2	-150	-35
512.163	219.03	NMeFOSA	-130	-35
512.163	169.022	NMeFOSA_2	-130	-40
526.192	219.03	NEtFOSA	-140	-35
526.192	169.022	NEtFOSA_2	-140	-35
570.202	419.061	NMeFOSAA	-100	-28
570.202	483	NMeFOSAA_2	-100	-22
584.229	419.061	NEtFOSAA	-100	-28
584.229	526.192	NEtFOSAA_2	-100	-38
616.1	58.9	NMeFOSE	-90	-70
630	58.9	NEtFOSE	-80	-75
285.035	169.022	HFPO-DA	-60	-12
285.035	184.9	HFPO-DA_2	-60	-18
377.06	251.028	ADONA	-65	-18
377.06	84.8	ADONA_2	-65	-48
530.8	351.05	9Cl-PFONS	-130	-38
532.8	353	9Cl-PFONS_2	-130	-38
630.9	451.031	11Cl-PFOUdS	-145	-41
632.9	452.9	11Cl-PFOUdS_2	-145	-41
241.085	177.069	3:3FTCA	-60	-12
241.085	117	3:3FTCA_2	-60	-50
341.101	237.072	5:3FTCA	-70	-20
341.101	217	5:3FTCA_2	-70	-35
441.117	316.9	7:3FTCA	-85	-30
441.117	337.088	7:3FTCA_2	-85	-20
315.093	135.013	PFEESA	-100	-35
315.093	82.9	PFEESA_2	-100	-25
229.032	85.006	PFMPA	-40	-25
279.042	85.006	PFMBA	-45	-25
295.032	201	NFDHA	-30	-15
295.032	84.9	NFDHA_2	-30	-40
217.001	171.999	MPFBA	-50	-14

268.001	222.999	M5PFPeA	-55	-12
318.009	273.007	M5PFHxA	-45	-12
367.024	322.022	M4PFHpA	-60	-12
421.002	376	M8PFOA	-65	-14
472.002	427	M9PFNA	-70	-14
519.033	474.03	M6PFDA	-80	-16
570.033	525.031	M7-PFUdA	-85	-18
615.079	570.033	MPFDoA	-85	-18
715.094	670.092	M2PFTeDA	-70	-22
302.069	80.062	M3PFBS	-100	-65
402.084	80.062	M3PFHxS	-120	-74
507.062	80.062	M8PFOS	-145	-85
329.13	81.07	M2-4:2FTS	-100	-50
429.162	81.07	M2-6:2FTS	-120	-65
529.162	81.07	M2-8:2FTS	-140	-85
506.077	78.07	M8FOSA	-150	-90
515.183	219.03	d3-NMeFOSA	-130	-35
531.222	219.03	d5-NEtFOSA	-140	-35
573.22	419.061	d3-NMeFOSAA	-75	-28
589.259	419.061	d5-NEtFOSAA	-90	-28
623.2	58.9	d7-NMeFOSE	-100	-28
639.2	58.9	d9-NEtFOSE	-100	-28
287.02	169.022	M3HFPO-DA	-60	-12
216.009	171.999	M3PFBA	-50	-14
315.032	270.03	M2PFHxA	-45	-12
417.032	372.03	M4PFOA	-65	-14
468.032	423.03	M5PFNA	-70	-14
515.063	470.061	M2PFDA	-80	-16
403.107	84.062	18O2-PFHxS	-120	-74
503.093	80.062	M4PFOS	-145	-85

Table 6: LC Method Conditions

Time (min)	2 mM Ammonium Acetate (5:95 CH/H_2O)	100% Acetonitrile	Gradient Curve
Initial	100.0	0.0	0
.2	100.0	0.0	2
4	70	30	7
7	45	55	8
9	25	80	8
10	5	95	6
10.4	98	2	10
11.8	100	0	7
12	100	0	1
Waters Aquity UPLC ® BEHC_{18} 2.1 x 50 mm packed with 1.7 µm BEH C_{18} stationary phase Flow rate of 0.4 mL/min 2 µL injection			

Table 7: ESI-MS Method Conditions

ESI Conditions	
Polarity	Negative ion
Curtain Gas	30
Collision gas	9
Ion Spray Voltage	-4500
Desolvation gas temp.	500 ºC
Ion Source Gas 1	30
Ion Source Gas 2	50
Entrance Poitential	-10
Exic Cell Potential	-11

Table 8. Reporting Limits by Matrix

Compound	Aqueous (ng/L)	Solid (ng/g)	Tissue (ng/g)
PFBA	6.4	0.8	2
PFPeA	3.2	0.4	1
PFHxA	1.6	0.2	0.5
PFHpA	1.6	0.2	0.5
PFOA	1.6	0.2	0.5
PFNA	1.6	0.2	0.5
PFDA	1.6	0.2	0.5
PFUnA	1.6	0.2	0.5
PFDoA	1.6	0.2	0.5
PFTrDA	1.6	0.2	0.5
PFTA	1.6	0.2	0.5
PFBS	1.6	0.2	0.5
PFPeS	1.6	0.2	0.5
PFHxS	1.6	0.2	0.5
PFHpS	1.6	0.2	0.5
PFOS	1.6	0.2	0.5
PFNS	1.6	0.2	0.5
PFDS	1.6	0.2	0.5
PFDoS	1.6	0.2	0.5
4:2FTS	6.4	0.8	2
6:2FTS	6.4	0.8	2
8:2FTS	6.4	0.8	2
FOSA	1.6	0.2	2
NMeFOSA	1.6	0.2	0.5
NEtFOSA	1.6	0.2	0.5
NMeFOSAA	1.6	0.2	0.5
NEtFOSAA	1.6	0.2	0.5
NMeFOSE	16	2	5
NEtFOSE	16	2	5
HFPO-DA	6.4	0.8	2
ADONA	6.4	0.8	2
9Cl-PFONS	6.4	0.8	2
11Cl-PFOUdS	6.4	0.8	2
3:3FTCA	8	1	2.5
5:3FTCA	40	5	12.5
7:3FTCA	40	5	12.5
PFEESA	3.2	0.4	1
PFMPA	3.2	0.4	1
PFMBA	3.2	0.4	1
NFDHA	3.2	0.4	1

17. TOP Assay SOP Addendum

The following modifications are applied to this SOP;

Section 7: Water Bath – Capable of monitoring recording to .1 C maintaining 85 C.

Section 8.1

1. Sodium Hydroxide (NaOH, CAS#: 1310-73-2) – High purity, demonstrated to be free of analytes and interferences.

2. Potassium Persulfate ($K_2S_2O_8$, CAS#: 7727-21-1) - High purity, demonstrated to be free of analytes and interferences.

3. Hydrochloric Acid (HCl, CAS#: 7647-01-0) - High purity, demonstrated to be free of analytes and interferences.

Table 1

Isotope Labeled Standard	Conc. Top Surr Stock (ng/mL)	Vol. of Top Surr Stock (µl)	Final Vol. of Top Surr PDS (mL)	Final Conc. of Top Surr PDS (ng/mL)
$^{13}C_2$-PFOA	50,000	40	4.0	500
$^{13}C_2D_4$-4:2FTS	50,000	80	4.0	1000

Section 10

1. Pretreatment of Samples - For Aqueous samples, prior to Section 10.1, add to each 125 ml sample with Potassium Persulfate until 60 mM (about 2 grams). Add Sodium Hydroxide until the sample is 125 mM (about .625 grams). Sample should have a pH >12.

2. Fortify each sample with 20 µl of TOP pre-assay surrogate containing 1 negative control surrogates and 1 positive control surrogates from table 1.

3. Place sample in a water bath at 85 C for 6 hours.

4. Remove sample from water bath and adjust pH 6-8 with Hydrochloric Acid.

5. Following pre-treatment, follow the aqueous extraction protocol from section 10 of SOP 45852 unaltered.

Section 11 - An additional calculation is added for the final reporting of the TOP assay results.

TOP Assay = Result of Post TOP assay extraction – Result of analysis from Pre-Top Assay extraction.

Nitrate, Nitrite and Nitrate_Nitrite Nitrogen

Reference: **Methods 353.2:** Methods for the Determination of Inorganic Substances in Environmental Samples, EPA 600/ R-93/ 100. August, 1993.

Methods 4500NO_3-F, 4500NO_2-B: Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 10-107-04-1, Lachat Instruments, 6645 West Mill Road, Milwaukee, WI 53218, 1992

1. Scope and Application

Matrices: This method is limited to optically clear water samples with a total concentration of nitrite and nitrate below 8mg N/L.

Definitions: Refer to Alpha Analytical Quality Manual.

In waters and wastewaters, the forms of nitrogen of greatest interest are, in order of decreasing oxidation state, nitrate, nitrite, ammonia, and organic nitrogen. All these forms of nitrogen, as well as nitrogen gas (N_2), are biochemically interconvertible and are components of the nitrogen cycle. They are of interest for many reasons.

Organic nitrogen is defined functionally as organically bound nitrogen in the trinegative oxidation state. It does not include all organic nitrogen compounds. Analytically, organic nitrogen and ammonia can be determined together and have been referred to as “kjeldahl nitrogen,” a term that reflects the technique used in their determination. Organic nitrogen includes such natural materials as proteins and peptides, nucleic acids and urea. Numerous concentrations vary from a few hundred micrograms per liter in some lakes to more than 20mg/L in raw sewage.

Total oxidized nitrogen is the sum of nitrate and nitrite nitrogen. Nitrate generally occurs in trace quantities in surface water but many attain high levels in some groundwater. In excessive amounts, it contributes to the illness known as methemoglobinemia in infants. A limit of 10mg nitrate as nitrogen/L has been imposed on drinking water to prevent this disorder. Nitrate is found only in small amounts in fresh domestic wastewater but in the effluent of nitrifying biological treatment plants, nitrate may be found in concentrations of up to 30mg nitrate as nitrogen/L. It is an essential nutrient for many photosynthetic autotrophs and has been identified as a growth-limiting nutrient.

Nitrite is an intermediate oxidation state of nitrogen, both in the oxidation of ammonia to nitrate and in the reduction of nitrate. Such oxidation and reduction may occur in wastewater treatment plants, water distribution systems, and natural waters. Nitrite can enter a water supply system through its use as a corrosion inhibitor in industrial process water. Nitrite is the actual etiologic agent of methemoglobinemia. Nitrous acid, which is formed from nitrite in acidic solution, can react with secondary amines (RR’NH) to form nitrosamines (RR’N-NO), many of which are known to be carcinogens. The toxicologic significance of nitrosation reactions in vivo and in the natural environment is the subject of much current concern and research.

Within this SOP, organic nitrogen is referred to as organic N, nitrate nitrogen as NO_3^--N, and nitrite nitrogen as NO_2^--N.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Lachat Analyzer and in the interpretation of Lachat data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Nitrate is quantitatively reduced to nitrite by passage of the sample through a copperized cadmium column. The nitrite (reduced nitrate plus original nitrite) is then determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl)ethylenediamine dihydrochloride. The resulting water-soluble dye has a magenta color, which is read at 520nm. Nitrite alone can be determined by removing the cadmium column. The nitrate is calculated as the difference between the reduced and non-reduced sample

2.1 Method Modifications from Reference

Soils can be analyzed using 1:10 ratio soil to water extraction, following filtration.

3. Reporting Limits

This method has an analytical range of 0.1 to 8.0mg N/L in the form of nitrate, and 0.05 to 8.0mg N/L in the form of nitrite.

The Reporting Limit is 0.1mg/L for Nitrate and 0.05 mg/L for Nitrite. Reporting limit is 1.0 mg/kg for soils

4. Interferences

4.1 Suspended matter in the column will restrict sample flow.

4.2 For turbid samples, filter through 0.45µm membrane filter prior to analysis.

4.3 Low results would be obtained for samples that contain high concentrations of iron, copper or other metals. In this method, EDTA is added to the buffer to reduce this interference.

4.4 Samples that contain large concentrations of oil and grease will coat the surface of the cadmium. In this case, only the water phase of the sample is used for analysis and a narrative is submitted with the data. Dilutions are performed as necessary.

4.5 Residual chlorine can interfere by oxidizing the Cd column, reducing its efficiency. Prior to analysis, check wastewater and drinking water samples for residual chlorine and record results in the Laboratory Notebook. If residual chlorine is present, and the samples are preserved with H_2SO_4, the sample may be analyzed for NO_3/NO_2 determination. However, NO_2 must be performed by a manual method. If it is not possible to analyze NO_2 by a manual method, the result is reported as NA and a narrative is submitted.

4.6 Sample color interferes if it is absorbed at about 540nm.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass or plastic bottles; 250mL minimum volume. Soils can be collected in plastic or glass containers.

6.2 Sample Preservation

Refrigerate samples at 4 ± 2 °C.

For Nitrate/Nitrite analysis, the samples are preserved with 1:1 H_2SO_4.

6.3 Sample Shipping

No specific requirement.

6.4 Sample Handling

Begin NO_3^- and/or NO_2^- determinations promptly after sampling. If storage is necessary, store for up to 48 hours at 4 ± 2 °C.

NOTE: If the 48-hour hold time cannot be met, the sample is to be handled as follows, only in an emergency situation. These instructions are not to be used on a regular basis.

Prior to the expiration of the 48-hour hold time, the following three steps are executed:

1. A manually colored Nitrite test is performed by Method 354.2. Results are recorded in the Laboratory Notebook.
2. A 50mL aliquot of the sample is preserved to a pH of <2 with concentrated H_2SO_4. Preservation is recorded in the Laboratory Notebook.

Prior to analysis, within 14 days of preservation, the preserved sample is neutralized using 6N NaOH. The sample is analyzed using only the Lachat Instrument.

CAUTION! Samples must NOT be preserved with mercuric chloride or thiosulfate because this will degrade the cadmium column

7. Equipment and Supplies

7.1 Lachat 8000 Automated Ion Analyzer or Lachat QuickChem 8500 Automated Ion Analyzer

7.2 Nitrate+Nitrite Lachat Board

7.3 Nitrite Lachat Board

7.4 Pre-packed Cadmium Columns: Available from Lachat.

7.5 Ottawa sand.

7.6 Disposable Culture Tubes 13x100 ml

7.7 Disposable pipettes.

8. Reagents and Standards

8.1 Stock Nitrate Standard, 1000mg N/L as NO_3^-: Purchased commercially prepared with certificate of analysis. Expires upon manufacturer's expiration date. There must be different manufacturers for calibration stock and ICV/LCS stock.

8.1.1 Stock Nitrate Standard, 200.0mg N/L as NO_3^-: Pipet 50mL of 1000ppm standard (Section 8.1) into 250mL volumetric flask and bring to volume with DI.

Alternately, in a 1L volumetric flask, dissolve 1.444g potassium nitrate (KNO_3) in about 600mL DI. Add 2mL chloroform. Dilute to the mark with DI and invert to mix. Refrigerate at 4±2°C. This solution is stable for six months.

8.2 Stock Nitrite Standard, 1000mg N/L as NO_2^-: Purchased commercially prepared with certificate of analysis. Expires upon manufacturer's expiration date. There must be different manufacturers for calibration stock and ICV/LCS stock.

8.2.1 Stock Nitrite Standard, 200.0mg N/L as NO_2^-: Pipet 50mL of 1000ppm standard (Section 8.2) into 250mL volumetric flask and bring to volume with DI.

Alternately, in a 1L volumetric flask, dissolve 0.986g sodium nitrite ($NaNO_2$) or 1.214g potassium nitrite (KNO_2) in approximately 800mL DI. Add 2mL chloroform. Dilute to the mark with DI and invert to mix. Refrigerate at 4±2°C. This solution is stable for six months.

8.3 Intermediate Nitrate Working Standard, 20 mg N/L as Nitrate: To a 250mL volumetric flask, add 25.0mL of the 200mg N/L NO_3^- stock standard. Dilute to the mark with DI and invert to mix. These solutions are stable for two weeks. Refrigerate at 4±2°C.

8.4 Intermediate Nitrite Working Standard, 20 mg N/L as Nitrite: To a 250mL volumetric flask, add 25.0mL of the 200mg N/L NO_2^- stock standard. Dilute to the mark with DI and invert to mix. These solutions are stable for two weeks. Refrigerate at 4±2°C.

8.5 Set of Six Calibration NO_3^- Standards, 8.0, 4.0, 1.00, 0.40, 0.20 and 0.1mg N/L as Nitrate: These standards are stable for 2 weeks. Refrigerate at 4±2°C.

To four 200mL volumetric flasks, add respectively: 8.0, 4.0, 1.0 and 0.4mL of the 200mg N/L NO_3^- stock standard. Bring to volume with DI water.

To two 200mL volumetric flasks, add respectively: 2.0 and 1.0mL of the 20mg N/L NO_3^- intermediate standard. Bring to volume with DI water.

Alternatively, an autodiluter can be used to make the standards during calibration, in which case only 8.0ppm and 1.0 ppm need to be manually prepared. If an autodiluter is used then it must be checked in an analytical tray by autodiluting 8.0mg N/L as Nitrite. The recovery for NO2 must be within 10% of the true value.

8.6 Set of Six Calibration NO_2^- Standards, 8.0, 4.0, 1.00, 0.40, 0.10 and 0.05mg N/L as Nitrite: These standards are stable for 2 weeks. Refrigerate at 4±2°C.

To three 200mL volumetric flasks, add respectively: 8.0, 4.0 and 1.0 of the 200mg N/L NO_2^- stock standard. Bring to volume with DI water.

To three 200mL volumetric flasks, add respectively:4.0, 1.0mL and 0.5mL of the 20mg N/L NO_2^- intermediate standard. Bring to volume with DI water.

Alternatively, an autodiluter can be used to make the standards during calibration, in which case only 8.0ppm and 1.0 ppm need to be manually prepared.

8.7 Ammonium Chloride Buffer, pH 8.5: In a 2L volumetric flask, dissolve 170g ammonium chloride (NH_4Cl) and 2.0g disodium ethylenediamine tetraacetic acid dihydrate ($Na_2EDTA•2H_2O$) in about 800mL water. Dilute to the mark with DI water and invert to mix. Adjust the pH to 8.5 with concentrated ammonium hydroxide or concentrated HCl. This solution is prepared monthly and stored at room temperature.

8.8 Sulfanilamide Color Reagent: To a 2L volumetric flask add about 1200mL water. Then add 200mL of 85% phosphoric acid (H_3PO_4), 80.0g sulfanilamide, and 2.0g N^-(1-naphthyl)ethylenediamine dihydrochloride (NED). Shake to wet, and stir to dissolve for 30 minutes. Dilute to the mark with DI water and invert to mix. Store in a dark bottle. This solution is stable for one month. Store at room temperature.

8.9 200ppm Nitrate Stock Standard, (for ICV/LCS): Pipet 50mL of 1000ppm standard (Section 8.1) into 250mL volumetric flask and bring to volume with DI. Store refrigerated at 4±2°C. Expires six months from preparation or upon manufacturer's expiration date.

8.10 200ppm Nitrite Stock Standard: Pipet 50mL of 1000ppm standard (Section 8.2) into 250mL volumetric flask and bring to volume with DI. Store refrigerated at 4±2°C. Expires six months from preparation or upon manufacturer's expiration date.

8.11 Initial Calibration Verification Standard (ICV)/Laboratory Control Sample (LCS): Store refrigerated at 4±2°C. Expiration is 2 weeks from date of preparation.

8.11.1 Nitrate LCS, 5.0ppm: Pipet 5.0mL of 200ppm stock (Section 8.9) into a 200mL volumetric flask and bring to volume with DI.

8.11.2 Nitrate ICV, 0.5ppm: Pipet 10.0mL of 5.0ppm standard (Section 8.11.1) into a 100mL volumetric flask and bring to volume with DI.

8.11.3 Nitrite LCS, 5.0ppm: Pipet 5.0mL of 200ppm stock (Section 8.9) into a 200mL volumetric flask and bring to volume with DI.

8.11.4 Nitrite ICV, 0.5ppm: Pipet 10.0mL of 5.0ppm standard (Section 8.11.3) into a 100mL volumetric flask and bring to volume with DI.

8.12 DPD Free Chlorine Reagent Powder Pillows: HACH brand, for 25mL sample. Store at room temperature. Expires upon manufacturer's expiration date.

8.13 1N Hydrochloric acid (HCL): To a 1L volumetric flask add about 600mL DI. Then add 83mL of concentrated hydrochloric acid (HCL) Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature.

8.14 1N Sodium Hydroxide (NaOH): To a 1L volumetric flask add about 600mL DI. Then add 40 g of sodium Hydroxide. Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature

8.15 6N Sulfuric Acid (H2SO4): To a 1L volumetric flask add about 600mL DI. Then add 140 ml of concentrated Sulfuric Acid (H2SO4). Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

One Method Blank is analyzed per batch of 20 samples or less The Method Blank consists of DI.

For soils: 5g of Ottawa sand extracted with 50 ml of DI. Results must be < 0.1mg/L. If this criterion is not met, the blank is re-analyzed. If there is still failure, the problem must be found and corrected prior to any sample analysis

9.2 Laboratory Control Sample (LCS)

The 5ppm ICV is reported as the LCS for the batch. See Section 9.3.

For soil LCS: 5g of Ottawa sand extracted with 0.25 ml of 1000 mg/l nitrate (8.1) (or 1000 mg/l Nitrite standard (8.2)) and 50 ml DI. The nitrate standard is used for spikes for Nitrate-N as well as Nitrate/Nitrite-N. LCS recoveries must be recovered within ± 10% of the true value. If these criteria are not met, LCS's must be re-analyzed. If failure continues, the batch has to be re-extracted and re-analyzed.

9.3 Initial Calibration Verification (ICV)

Two ICVs are analyzed at the beginning of the analytical sequence. One is at a concentration of 0.5ppm, and the other is at a concentration of 5.0ppm.

Both must be recovered within ± 10% of the true value. If these criteria are not met, the ICVs must be re-analyzed. If failure continues, the ICVs are to be re-made and/or a new calibration curve is to be generated.

The 5ppm ICV is reported as the LCS for the batch.

9.4 Continuing Calibration Verification (CCV)

At the beginning of the first tray, after every ten samples and at the end of every analytical sequence, a CCV and a CCB pair must be analyzed to verify both calibration curves.

1.0ppm Nitrate CCV (Section 8.5)

1.0ppm Nitrite ICV (Section 8.6)

Calibration Blank (DI)

The results of the CCVs must be within ±10% of the true value, otherwise re-calibration is required.

The results of the CCBs must be less than our standard limit of detection, otherwise the analysis is stopped and the problem corrected.

9.5 Matrix Spike

One Matrix Spike is analyzed per batch of 20 samples or less. Separate spikes are performed for Nitrate and Nitrite. In a 25mL volumetric flask, 0.5mL of 200ppm stock calibration standard (Section 8.1 or 8.2) is added to the sample. The final concentration of the matrix spike is

4.0ppm. The nitrate standard is used for spikes for Nitrate-N as well as Nitrate/Nitrite-N. The nitrite standard is used for spikes for Nitrite-N.

For soils: weigh 5.0 g of sample, add 2.0 ml of 200 mg/l Nitrate or Nitrite standard and 48 ml of DI. The final concentration of the matrix spike is 80.0 mg/kg. The nitrate standard is used for spikes for Nitrate-N as well as Nitrate/Nitrite-N. The nitrite standard is used for spikes for Nitrite-N.

% Recovery for the Matrix Spike must be within in-house control limits. If acceptance criteria are not met, the Matrix Spike is reanalyzed. If failure continues, a narrative is included with the data for inclusion on the Client report.

Note: For samples, analyzed by method **353.2** (NO2-353 and NO3-353) maximum batch size is 10 samples; every 10 samples required separate matrix spike (MS) to be analyzed. % Recovery for the Matrix Spike must be within +/- 10% of true value. If acceptance criteria are not met, the Matrix Spike is reanalyzed. If failure continues, a narrative is included with the data for inclusion on the Client report.

9.6 Laboratory Duplicate

One Duplicate sample is analyzed per batch of 20 samples or less. A separate aliquot of the sample is analyzed for this purpose.

% RPD for the Duplicate must be within in-house control limits. If acceptance criteria are not met, the Duplicate is reanalyzed. If failure continues, a narrative is included with the data for inclusion on the Client report

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Calibration
- ICV/LCS – both levels
- Sample analysis
- CCV – every ten samples and at the end of the analytical sequence

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation

10.1.1.1 Place the Nitrate+Nitrite board (containing the cadmium column) in Channel 1. Place the Nitrite board in Channel 2. Make sure the valve to the cadmium column is closed prior to starting to pump the reagents.

10.1.1.2 Commence pumping of reagents.

10.1.1.3 Once the lines are full of reagent and free of gas bubbles, open the valve to allow reagent to flow through the cadmium column.

NOTE: Be sure to switch the valve back before rinsing the manifold with DI water at the completion of the run.

NOTE: DO NOT LET AIR ENTER THE CADMIUM COLUMN.

10.1.2 Column Efficiency Procedure

10.1.2.1 Visually inspect the column. Check for air bubbles in the column or lines, gaps in the column or any change in the cadmium surface characteristics, (cadmium granules should be dark gray). If air bubbles are present in column, connect the column into the manifold, turn the pump on maximum and tap firmly with a screwdriver handle, being careful not to break the column, working up the column until all air is removed. If air cannot be removed, the column should be repacked. Cadmium columns should be stored filled with buffer. If air enters the column, efficiency will decrease. Check the flow efficiency by disconnecting the cadmium column from the manifold and reconnecting to a green pump tube. Pump buffer through the packed column and collect in a graduated cylinder. The flow rate with the column connected should be greater than 4.0 mL/minute.

10.1.2.2 **Column Efficiency – Slope Ratio Method:** Calibrate with the mid-range NO_3 – N standards. Calibrate with a matching concentration range of NO_2–N standards. The column efficiency is determined by the equation:

$$E = \frac{S_{NO3\text{-}N}}{S_{NO2\text{-}N}} \times 100$$

where:

$S_{NO3\text{-}N}$ = slope of NO_3 calibration
$S_{NO2\text{-}N}$ = slope of NO_2 calibration
E = % efficiency

10.1.2.3 **Column Efficiency – Concentration Ratio Method:** Calibrate with the mid-range NO_2-N and NO_3-N standards. Run a known concentration NO_2-N standard. Run a matching concentration NO_3-N standard. The column efficiency is determined by the following equation:

$$E = \frac{C_{NO3\text{-}N}}{C_{NO2\text{-}N}} \times 100$$

where:

$C_{NO3\text{-}N}$ = concentration of NO_3 standard
$C_{NO2\text{-}N}$ = concentration of NO_2 standard
E = % efficiency

10.1.2.4 **Column Efficiency Result:** If the efficiency is <75%, the column is repacked. All results are recorded and maintained on file in the QC department.

10.1.3 Residual Chlorine Screening

Check all wastewater and drinking water samples for residual chlorine prior to analysis.

10.1.3.1 Add 1 DPD Free Chlorine powder pillow (Section 8.12) to 25mL of sample in a centrifuge tube. An immediate color change to pink indicates residual chlorine is present. If residual chlorine is present, add a small amount of ascorbic acid to a sample aliquot (record this in logbook) and check for residual chlorine presence again. If residual chlorine remains, notify the Department Manager and/or the Laboratory Director. Results will be reported as Not Applicable (N/A).

If residual chlorine is not present, continue with sample analysis.

10.2 Initial Calibration

Calibrate the Lachat ion analyzer according to manufacturer's instructions.

10.2.1 Calibration

Two boards are used to calibrate the Lachat instrument. Each curve has seven calibration points. The correlation coefficient of each curve must be $\geq$0.995, otherwise recalibration is necessary. Prepare standard curves by plotting the peak areas of standards processed through the manifold against NO_3+NO_2 as N and NO_2 as N concentrations in standards.

10.2.1.1 Channel 1 is used to generate a calibration curve for Nitrate/Nitrite ranging from 0 to 8.0ppm.

10.2.1.2 Channel 2 is used to generate a calibration curve for Nitrite ranging from 0 to 8.0ppm.

Note: Instrument is calibrated daily, fixed calibration range is used; linearity is verified daily; three standards are used for linear calibration verification (low ICV (0.5 mg/l), High ICV (5.0 mg/l) and CCV (1.0 mg/l)). All standards must be within 10% of true value

10.2.2 Initial Calibration Verification (ICV)

10.2.2.1 Prior to sample analysis, the following ICVs must be analyzed to verify both calibration curves.

10.2.2.1.1 Nitrate ICV, 0.5ppm (Section 8.12.2)

10.2.2.1.2 Nitrate ICV, 5.0ppm (Section 8.12.1)

10.2.2.1.3 Nitrite ICV, 0.5ppm (Section 8.12.4)

10.2.2.1.4 Nitrite ICV, 5.0ppm (Section 8.12.3)

10.2.2.2 The results must be within $\pm$10% of the true value, otherwise re-calibration is required.

10.3 Equipment Operation and Sample Processing

Follow the manufacturer's directions for the operation of the Lachat 8000.

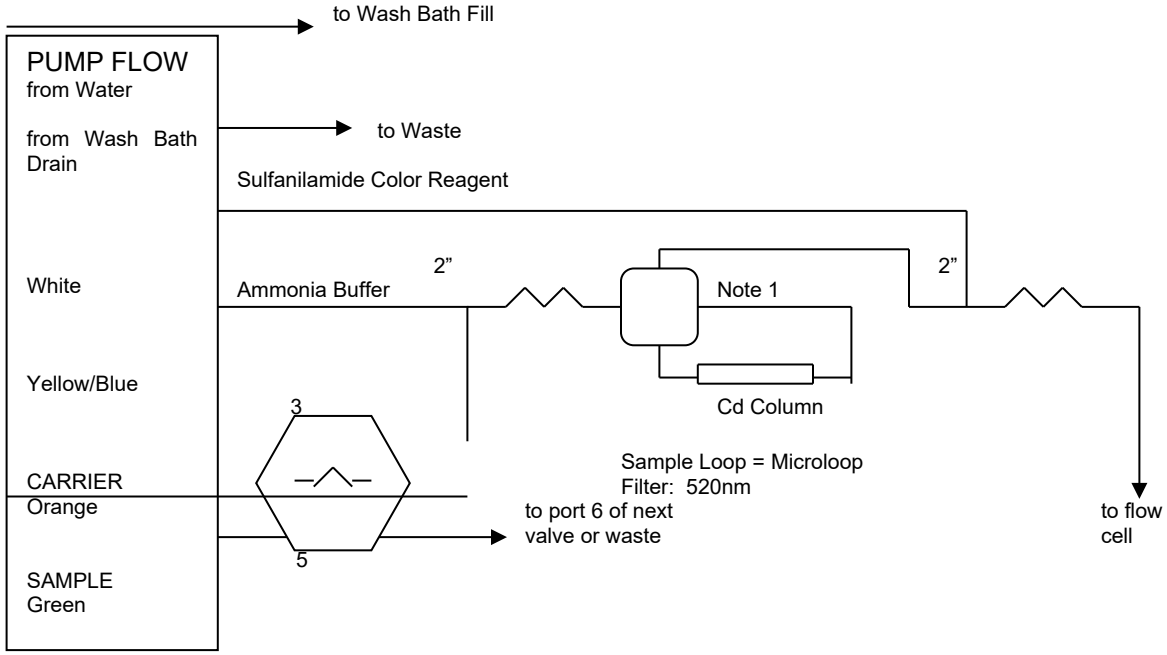
All samples have to be inspected prior to analysis. Samples that are turbid or have sediment have to be filtered prior to analysis.

Check pH of the samples. If pH is less then 5 or greater then 9, then adjust pH using 1N Hydrochloric Acid (HCl) (8.13), 6N Sulfuric Acid (H2SO4) (8.15) or 1N Sodium Hydroxide (NaOH) (8.14). Record pH adjustment in the log book.

For soils: extract soils samples prior to analysis: take 5g of sample, add 50 ml of DI, extract for 30 min, then filter thorough 0.45 nm filter. Record all weights for calculations.

Note: if samples are filtered, then Method blank also have to be filtered

The Manifold Diagram follows:



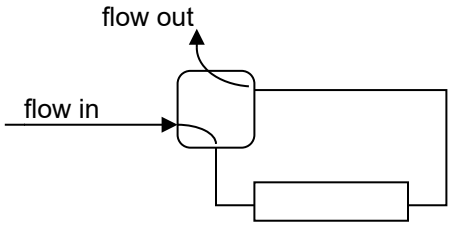
CARRIER is water.

2" is 135cm of tubing on a 2-inch coil support.
APPARATUS: Standard valve, flow cell, and detector head modules are used.

All manifold tubing is 0.8mm (0.032") i.d. This is 5.2µL/cm.

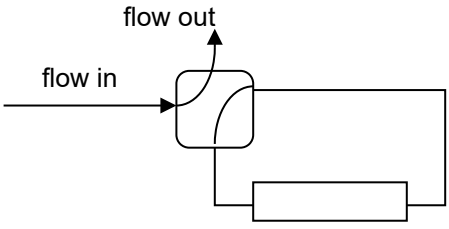
NOTE 1: This is a two-state switching valve used to place the cadmium column in line with the manifold.

State 1: Nitrate + Nitrite



Cd Column

State 2: Nitrite Only



Cd Column

10.4 Continuing Calibration

See Section 9.4

10.5 Preventive Maintenance

Tubing is changed monthly or as needed.

At the end of each analytical sequence, the valve to the column is closed. DI is rinsed through the Lachat for five minutes followed by five minutes of air.

All maintenance is documented in the Instrument Maintenance Logbook.

11. Data Evaluation, Calculations and Reporting

11.1 Nitrate/Nitrite: When the software is set up according to the manufacturer's recommendations, the concentration of nitrate plus nitrite in mg NO_3/NO_2-N/L is reported directly when the Cd column is included in the sample train in Channel 1.

11.2 Nitrite: When the software is set up according to the manufacturer's recommendations, the concentration nitrite in mg NO_2-N/L is reported directly when the Cd is not included in the sample train in Channel 2.

11.3 Nitrate: The concentration of nitrate is determined by the subtraction of the nitrite concentration, (Section 11.2 above), from the nitrate-nitrite concentration, (Section 11.1 above).

11.3.1 If the sample was preserved initially as described in Section 6.3, subtract the Nitrite value generated manually from the Nitrate/Nitrite value generated by the Lachat Instrument. This value is reported as the Nitrate result.

When the sample is preserved initially as described in Section 6.3, the value generated by the Lachat instrument for Nitrite is invalid and therefore disregarded.

11.4 If any sample exceeds a concentration of 8.0 mg/L, the sample must be diluted and re-analyzed. All sample concentrations must fall within the calibration curve.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

The quarterly method reporting limit (MRL) verification requirement is satisfied by the quarterly LOQ analysis, as per SOP/1732

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

16. Revision History

Revisions Made from Prior Version

Section	Description of Change
8.7	Added the use of Conc HCl to lower pH of buffer

17. Attachments

None

Nitrite

Manual Colorimetric Method

References: **SM 4500NO2-B**, Standard Methods for the Examination of Water and Wastewater, Online (2011).

1. Scope and Application

Matrices: This method is limited to optically clear water samples with a total concentration of nitrite below 1mg N/L. Turbid samples are filtered prior to analysis.

Definitions: See Alpha Laboratories Quality Manual Appendix A

In waters and wastewaters, the forms of nitrogen of greatest interest are, in order of decreasing oxidation state, nitrate, nitrite, ammonia, and organic nitrogen. All these forms of nitrogen, as well as nitrogen gas (N_2), are biochemically interconvertible and are components of the nitrogen cycle. They are of interest for many reasons.

Organic nitrogen is defined functionally as organically bound nitrogen in the trinegative oxidation state. It does not include all organic nitrogen compounds. Analytically, organic nitrogen and ammonia can be determined together and have been referred to as “kjeldahl nitrogen,” a term that reflects the technique used in their determination. Organic nitrogen includes such natural materials as proteins and peptides, nucleic acids and urea. Numerous concentrations vary from a few hundred micrograms per liter in some lakes to more than 20mg/L in raw sewage.

Total oxidized nitrogen is the sum of nitrate and nitrite nitrogen.

Nitrite is an intermediate oxidation state of nitrogen, both in the oxidation of ammonia to nitrate and in the reduction of nitrate. Such oxidation and reduction may occur in wastewater treatment plants, water distribution systems, and natural waters. Nitrite can enter a water supply system through its use as a corrosion inhibitor in industrial process water. Nitrite is the actual etiologic agent of methemoglobinemia. Nitrous acid, which is formed from nitrite in acidic solution, can react with secondary amines (RR’NH) to form nitrosamines (RR’N-NO), many of which are known to be carcinogens. The toxicologic significance of nitrosation reactions in vivo and in the natural environment is the subject of much current concern and research.

Within this SOP nitrite nitrogen is referred to as NO_2^--N.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Spectrometer. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The nitrite is determined by diazotizing with sulfanilamide followed by coupling with N-(1-naphthyl)ethylenediamine dihydrochloride. The resulting water-soluble dye has a magenta color, which is read at 540nm.

2.1 Method Modifications from Reference

1. Spectrophotometer reading done at 540 nm, not at 543 nm.

2. Two coloring reagents- sulfanilamide and NED used, instead of one combined coloring reagent.

3. Detection Limits

The laboratory follows the procedure found in 40CFR Part 136 to determine the MDL on an annual basis. The method detection limits determined by the laboratory are on file for review.

This method has an analytical range of 0.02 to 0.3mg N/L in the form of nitrite. The Reported Detection Limit is 0.02 mg N/L.

4. Interferences

4.1 For turbid samples, filter through 0.45µm membrane filter prior to analysis.

4.2 Sample color interferes if it is absorbed at about 540nm.

5. Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, and Handling

6.1 Sample Collection

Samples are collected in glass or plastic bottles.

6.2 Sample Preservation

Refrigerate samples at 4 ± 2 °C.

6.3 Sample Handling

Samples are stored under refrigeration at 4 ±2 °C. Analysis must be performed within 48 hours of collection.

7. Equipment and Supplies

7.1 Spectrophotometer: With infrared phototube for use at 540nm, providing a light path of 1.0cm.

7.2 Centrifuge Tubes: 50mL volume. (Must be new and disposable.)

7.3 Acrodisk Membrane Filters, 0.45µm

7.4 Acrodisk Glass Fiber Filters

7.5 Pipets: various volumes.

7.6 Syringe: 10mL and 20mL

8. Standards and Reagents

8.1 Stock Nitrite Standard, 1000mg N/L as NO_2: Purchased commercially prepared. Store in refrigerator at 4 ± 2°C. Expires upon manufacturer's specified date.

8.1.1 Intermediate Nitrite Working Standard, 5mg N/L as Nitrite: To a 200mL volumetric flask, add 1.0mL of the 1000mg N/L NO_2^- stock standard. Dilute to the mark with DI and invert to mix. This solutions are stable for two weeks. Store in the refrigerator at 4 ± 2°C.

8.2 Calibration Standards: Refer to the Table below for preparation instructions. Prepare fresh on each day of use.

Volume of 5.0 mg/L Working Standard (Section 8.3)	Final Volume (mL)	Calibration Standard Final Concentration (mgN/L)
1.0 mL	250	0.02
1.0 mL	100	0.05
2.0 mL	100	0.10
6.0 mL	100	0.30

8.3 ICV-LCS-CCV Stock Standard, 1000mg N/L: A second source standard than what is used in Section 8.1. Store in refrigerator at 4 ± 2°C. Expires upon manufacturer's specified date.

8.3.1 ICV-LCS-CCV Intermediate Standard, 8mg N/L: Add 0.8mL of 1000 mgN/L stock standard (Section 8.3) to 100mL volumetric flask and dilute to volume with DI water. Store refrigerated at 4 ±2 °C. Expires 2 weeks from date of preparation.

8.3.2 ICV-LCS-CCV Intermediate Standard, 5mg N/L: Add 0.5mL of 1000 mgN/L stock standard (Section 8.3) to 100mL volumetric flask and dilute to volume with DI water. Store refrigerated at 4 ±2 °C. Expires 2 weeks from date of preparation

8.3.2.1 ICV-LCS-CCV Working Standard, 0.16mg N/L: Add 1mL of 8 mg/L intermediate standard (Section 8.3.1) to 50mL volumetric flask and dilute to volume with DI water. Prepare fresh on each day of use.

8.3.2.2 ICV-LCS-CCV Working Standard, 0.05mg N/L: Add 0.5mL of 5 mg/L intermediate standard (Section 8.3.2) to 50mL volumetric flask and dilute to volume with DI water. Prepare fresh on each day of use.

8.3.2.3 ICV-LCS-CCV Working Standard, 0.2mg N/L: Add 2.0mL of 5 mg/L intermediate standard (Section 8.3.2) to 50mL volumetric flask and dilute to volume with DI water. Prepare fresh on each day of use.

8.4 NED coloring reagent: Dissolve 1.0g N¯(1-naphthyl)ethylenediamine dihydrochloride (NED) in 1L volumetric flask with DI. Store in a dark place at room temperature for up to 3 months.

8.5 Sulfanilamide Color Reagent: In a 500mL volumetric flask, dissolve 5g of sulfanilamide and 50 mL of 85% Phosphoric Acid. Bring to 500mL volume with DI water. Expires one month from date of preparation. Store in a dark bottle in the refrigerator for up to 1 month..

8.6 1N Hydrochloric acid (HCL): To a 1L volumetric flask add about 600mL DI. Then add 83mL of concentrated hydrochloric acid (HCL) Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature.

8.7 5N Sodium Hydroxide (NaOH): To a 1L volumetric flask add about 600mL DI. Then add 200 g of sodium Hydroxide. Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature.

8.8 6N Sulfuric acid (H2SO4): To a 1L volumetric flask add about 600mL DI. Then add 140mL of concentrated sulfuric acid (H2SO4) Stir to dissolve. Dilute to the mark with DI water and invert to mix. This solution is stable for six month. Store at room temperature.

8.9 **Phosphoric Acid 85%.** Store at room temperature. Expires upon manufacturer's specified date

9. Procedure

9.1 Set Up

Samples to be analyzed for Nitrite are filtered through a 0.45µm membrane filter prior to analysis if turbid or have color. Typically 50-100 mL is filtered. A glass fiber filter may be used to prefilter hard-to-filter samples. Clear samples don't need to be filtered.

9.2 Initial Calibration

Prepare individual calibration curve from a series of four standards (Section 8.2) and blank as per Section 9.4. Plot absorbance vs. nitrite concentration to give a linear curve. The correlation coefficient must be 0.995 or greater for the curve to be considered valid. The calibration curve is verified by a second source standard prior to performing any sample analysis. Recovery of the second source standard must be 90-110% of the true value.

All calibration points are back calculated and should be within 10% from true concentration, except 1 lowest point of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse then 0.995

An Initial Calibration Curve is generated daily.

9.3 Standardization (Continuing Calibration Verification)

The calibration curve is verified by a second source standard after every 20 samples or less. Refer to Section 10.3.

9.4 Equipment Operation

9.4.1 Spectrometer preparation

9.4.1.1 The spectrometer is set to zero absorbance using only DI in 1 cm cell at a wavelength of 540nm.

9.4.2 Sample Analysis

9.4.2.1 Pour 50mL, or a portioned dilution, of each clear sample, Duplicate sample, Method Blank, LCS/ICV and Matrix Spike sample into a corresponding new (never used) centrifuge tube.

9.4.2.2 Check pH of the samples. If pH is less then 5 or greater then 9, then adjust pH using 1N Hydrochloric Acid (HCl) (8.6), 5N Sodium Hydroxide (8.7) or 6N Sulfuric Acid (8.8). Record pH adjustment on excel.

9.4.2.3 Add 2.0mL of NED coloring (Section 8.4) reagent to each 50mL sample and QC sample aliquot and mix thoroughly.

9.4.2.4 Add 2.0mL of Sulfanilamide (Section 8.5) reagent to each 50mL sample and QC sample aliquot and mix thoroughly.

9.4.2.5 After at least 10 minutes but no more than 30 minutes, measure the absorbance of each sample on the spectrophotometer at 540nm with a 1.0cm cell. Record results in the laboratory notebook. ELN can be used for data reporting.

9.4.2.6 If the sample concentration is greater than the highest calibration standard concentration, dilute the original sample with DI water and recolor and reanalyze as outlined above (Section 9.4.2.1 – 9.4.2.5).

9.5 Preventative Maintenance

The Spectrophotometers are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file.

9.6 Calculations

mg N/L, as NO_2 = mg N (as NO_2 from calibration curve) x dilution factor

10. Quality Control and Data Assessment

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation.

10.1 Demonstration of Capability

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. Each time a method modification is made, the analyst is required to repeat the procedure.

When the parameter tested fails at least one of the acceptance criteria, the analyst must locate and correct the source of the problem and repeat the test.

Repeated failure confirms a general problem with the measurement system or analytical technique of the analyst. If the failure repeats, locate and correct the source of the problem and repeat the test.

10.2 Blanks

10.2.1 Method Blank: One method blank, which consists of 50mL DI water, must be analyzed per batch of 20 samples or less. If samples were filtered, then blank has to be filtered through a 0.45 micron filter. The method blank result must be less than the Reporting Limit. If the method blank is higher than the RL, a fresh aliquot of 50mL DI is used. If the method blank is higher than the RL again, the problem must be identified and corrected before any samples are analyzed.

10.2.2 Continuing Calibration Blank: A continuing calibration blank (CCB) consists of 50mL DI water colored and treated as per Section 9.4. A CCB must be analyzed immediately following the ICV/LCS and immediately prior to the CCV. The CCB result must be less than the Reporting Limit. If the CCB is greater than the RL, all samples analyzed since the last passing CCB must be recolored and reanalyzed.

10.3 Calibration Verification and Laboratory Control Samples (ICV-LCS-CCV)

Analyze one LCS/ICV per batch of 20 samples or less. The calibration curve must be verified by a second source standard prior to performing any sample analysis. The ICV/LCS must be recovered at 90 – 110% of the true value.

If the ICV/LCS fails, re-analyze. If failure remains, stop analysis, find and correct the problem. Re-calibrate and recolor a fresh sample aliquot and reanalyze.

Analyze a CCV after every ten samples or less. The CCV must be recovered at 90 – 110% of the true value.

If the CCV fails, re-analyze. If failure remains, stop analysis, find and correct the problem. Re-calibrate and recolor a fresh sample aliquot and reanalyze.

10.4 Matrix Spike

Analyze one matrix spike per batch of 20 samples or less. The Matrix Spike final concentration is 0.1mg N/L when prepared as follows: Add 1.0mL of the 5 mg N/L Intermediate Standard (Section 8.1.1) to 49mL of sample.

If the MS recovery is outside of acceptance limits, which are based on in-house control limits (Section 10.6), the sample and its spike are reanalyzed. If the MS failure continues, a narrative is submitted with the data for inclusion on the Client report.

10.5 Duplicates

Analyze one sample in duplicate, per batch of 20 samples or less. The RPD acceptance criteria is based on in-house control limits (Section 10.6). If the RPD is outside of acceptance limits, the sample and its duplicate are reanalyzed. If the RPD failure continues, a narrative is submitted with the data for inclusion on the Client report.

10.6 Control Limits

The laboratory maintains performance records to document the quality of data that is generated. Method accuracy for samples is assessed and records maintained.

Control limits for the method parameters are generated. The control limits are calculated based on in-house performance data. The limits are compared to the control limits found in the reference method.

10.7 Analytical Sequence

- Sample filtration, if necessary including blank.
- Calibration Curve generation
- LCS/ICV
- Method Blank
- Samples 1-10
- Sample Duplicate
- Matrix Spike
- CCB
- CCV
- Samples 11-20
- CCB
- CCV

11. Method Performance

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations were obtained using reagent water. The MDL actually achieved will vary depending on instrument sensitivity and matrix effects.

Verification of the minimum reporting limit (MRL): The minimum reporting level (MRL) is set to a concentration at or above the lowest standard used in the analysis.
Verify quantitation at the MRL initially and at least quarterly by analyzing a QC sample (subjected to all sample-preparation steps) spiked at a level 1 to 2 times the MRL. A successful verification meets the method's or laboratory's accuracy requirements at the MRL.

Method performance data is on file in the laboratory. Comparison of method performance data for the laboratory to the reference method criteria occurs when laboratory in-house acceptance limits are generated. In-house generated data must be within the specifications of the reference method or the analysis is not continued until corrective action is completed.

12. Corrective Actions

Holding time exceedences or improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CCV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Pollution Prevention

See Chemical Hygiene Plan for pollution prevention operations.

14. Waste Management

See Chemical Hygiene Plan for waste handling and disposal.

Nitrogen Ammonia

Reference: **SM4500 NH_3-BH,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 350.1, Methods for the Chemical Analysis of Water and Wastes, EPA 600/ 4-82-055, Revision 2.0, August 1993.

Method 10-107-06-1-A, Methods for Automated Ion Analyzers, May 20, 1998.

1. Scope and Application

Matrices: Ammonia nitrogen can be determined in potable, surface, and saline waters as well as domestic and industrial wastewaters and in soils.

Definitions: Refer to Alpha Analytical Quality Manual.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Tecator and/or Lachat Instrument and in the interpretation of Lachat data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

In waters and wastewaters the forms of nitrogen of greatest interest are, in order of decreasing oxidation state: nitrate, nitrite, ammonia, and organic nitrogen. All these forms of nitrogen, as well as nitrogen gas (N_2), are biochemically interconvertible and are components of the nitrogen cycle. They are of interest for many reasons. Organic nitrogen is defined functionally as organically bound nitrogen in the trinegative oxidation state. It does not include all organic nitrogen compounds. Analytically, organic nitrogen and ammonia can be determined together and have been referred to as “kjeldahl nitrogen,” a term that reflects the technique used in their determination. Organic nitrogen includes such natural materials as proteins and peptides, nucleic acids and urea, and numerous synthetic organic materials. Typical organic nitrogen concentrations vary from a few hundred micrograms per liter in some lakes to more than 20mg/L in raw sewage.

Ammonia is present naturally in surface and wastewaters. Its concentration generally is low in groundwaters because it adsorbs to soil particles and clays and is not leached readily from soils. It is produced largely by deamination of organic nitrogen containing compounds and by hydrolysis of urea. At some water treatment plants ammonia is added to react with chlorine to form a combined chlorine residual.

In the chlorination of wastewater effluents containing ammonia, virtually no free residual chlorine is obtained until the ammonia has been oxidized. Rather, the chlorine reacts with ammonia to form mono- and dichloramines. Ammonia concentrations encountered in water vary from less than 10µg ammonia nitrogen/L in some natural surface and groundwaters to more than 30mg/L in some wastewaters.

In this discussion, organic nitrogen is referred to as organic N, nitrate nitrogen as NO_3-N, nitrite nitrogen as NO_2-N, and ammonia nitrogen as NH_3-N.

2. Summary of Method

The sample is buffered at pH 9.5 with a borate buffer to decrease hydrolysis of cyanates and organic nitrogen compounds. It is distilled into a solution of boric acid. The ammonia in the distillate is determined colorimetrically by the phenate method.

The phenate method is base on the Berthelot reaction. Ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity. The absorbance of the reaction product is measured at 630nm, and is directly proportional to the original ammonia concentration

2.1 Method Modifications from Reference

Method was modified to include soil sample distillation. Samples are buffered at pH 9.5. pH is checked using pH paper 7.5-14 (not pH meter).

3. Reporting Limits

The RL is determined to be 0.075mg/L based on a 100mL sample size for waters and 7.5 mg/kg based on 1g of soil material used for distillation.

4. Interferences

4.1 Most of the following interferences refer to the direct analysis of a water sample. The distillation steps remove most of these interferences. Residual chlorine must be removed prior to distillation by pretreatment of the sample with sodium thiosulfate.

4.2 Samples with a high concentration of NH_3 may carry-over into the next sample and therefore yield false high results in that next sample. If a sample with a low concentration follows a sample with a high concentration, re-analyze the low sample to ensure results are accurate.

4.3 Glycine, urea, glutamic acid, cyanates, and acetamide hydrolyze very slowly in solution on standing but, of these, only urea and cyanates will hydrolyze on distillation at pH of 9.5. Hydrolysis amounts to about 7% at this pH for urea and about 5% for cyanates. Some of the organic compounds that may interfere, such as formaldehyde, may be eliminated by boiling off at a low pH before nesslerization.

4.4 Color and turbidity of the sample distillate may interfere with analysis. These interferences are removed by manual filtration of the distillate prior to Lachat analysis, or by dilution of the sample prior to distillation.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

Phenol used in this method is hazardous and general laboratory safety practices must be observed.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

The most reliable results are obtained on fresh samples. Use plastic or glass containers.

6.2 Sample Preservation

If prompt analysis is impossible, preserve samples with approximately 0.8mL concentrated H_2SO_4/L sample. The pH of the acid-preserved samples should be <2. (Some wastewaters may require more concentrated H_2SO_4 to achieve this pH.) Store samples at 4ºC.

Soils samples are received without preservation and stored at 4ºC

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Samples (both waters and soils) are analyzed within 28 days after collection.

7. Equipment and Supplies

7.1 Distillation apparatus: Tecator Instruments Automatic Distillation Unit. Follow the instrument manufacturer's instructions for proper operation.

7.2 pH Strips: Range 7.5 to 14 units.

7.3 250mL Disposable Polypropylene cups with covers.

7.4 Automated Ion Analyzer: Lachat Instruments.

7.5 Helium gas, He: 140kPa (20 lb/in^2).

7.6 Volumetric flasks, various volumes.

7.7 Pipets, various volumes.

7.8 Analytical Balance

7.9 Centrifuge Tubes: 50mL volume.

7.10 Potassium Iodide Starch Paper: Fisher brand 14-860

8. Reagents and Standards

8.1 Borate buffer solution: Add 88mL 0.1N NaOH solution to 500mL of approximately 0.025M sodium tetraborate ($Na_2B_4O_7$) solution (9.5g $Na_2B_4O_7 \cdot 10\ H_2O$/L) and dilute to 1L with DI water. Store at room temperature. Expires one month from date of preparation.

8.2 Sodium thiosulfate (dechlorinating reagent): Dissolve 0.35g $Na_2S_2O_3 \cdot 5H_2O$ in DI water and dilute to 100mL. Prepare fresh daily as needed.

8.3 Neutralization agents: Prepare with DI water:

8.3.1 Sodium hydroxide, NaOH, 6N: In a 1L volumetric flask, carefully mix 240g of NaOH (Section 8.11) pellets with 500mL DI water. Dissolve and bring to volume with DI water. Store at room temperature. Expires one month from date of preparation.

8.3.2 Sulfuric acid, H_2SO_4, 10%: To a 1L volumetric flask, add 500mL DI water. Carefully add 100mL of concentrated Sulfuric Acid (Section 8.12). Bring to volume with DI water. Store at room temperature. Expires one month from date of preparation.

8.4 Sodium Phenolate: CAUTION! Wear gloves. Phenol causes severe burns and is rapidly absorbed into the body through the skin. In a 1L volumetric flask, dissolve 88mL of 88% liquified phenol or 83g crystalline phenol (C_6H_5OH) in approximately 600mL DI water. While stirring, slowly add 32g sodium hydroxide (NaOH). Cool, dilute to the mark, and invert three times. Do **not** degas this reagent. Store at room temperature. Expires one month from date of preparation.

8.5 Sodium Hypochlorite (approximate 2.6%): In a 500mL volumetric flask, dilute 250mL Regular Chlorine bleach [5.25% sodium hypochlorite (NaOCl)] to the mark with DI water. Invert three times to mix. Store at room temperature. Expires one month from date of preparation.

8.6 Sodium Nitroprusside (coloring agent): In a 1L volumetric flask, dissolve 3.50g sodium nitroprusside (Sodium Nitroferricyanide [$Na_2Fe(CN)_5NO_2 \cdot H_2O$]) dilute to the mark with DI water. Degas with helium to prevent bubble formation. Use He at 140kPa (20 lb/in^2) through a helium degassing tube. Bubble He vigorously through the solution for one minute. Store refrigerated at 4 ± 2 °C. Expires one month from date of preparation.

8.7 Boric 1.5% Boric Acid Solution: To a 1000mL volumetric flask add 15g Boric Acid. Dilute to the mark with DI water. Store at room temperature. Expires one month from date of preparation.

8.8 0.2% Boric Acid Solution (Carrier Solution): To a 2L volumetric flask, dissolve 4g Boric Acid (H_3BO_3) in DI water. Degas by bubbling vigorously with Helium for one minute. Store at room temperature. Expires one month from date of preparation.

8.9 Stock Standard, 1000ppm as NH_3 (for calibration and spike solutions): Commercially prepared. Certificate of analysis is required. Store refrigerated at 4 ± 2 °C. Expires upon manufacturer's specified date.

8.9.1 Intermediate Calibration Stock Standard, 100ppm as NH_3: To a 100mL volumetric flask, add 10.0mL of Stock Standard (Section 8.9) and dilute to the mark with 0.2% Boric Acid solution (Section 8.8). Invert three times. Store refrigerated at 4 ± 2 °C. Expires one month from date of preparation.

8.9.1.1 Nine Working Calibration Standards: The following standards are prepared in volumetric flasks fresh each day of use:

8.9.1.1.1 20.0ppm: 40mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.2 10.0ppm: 20mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.3 **8.00ppm:** 8mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.4 **4.00ppm:** 8mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.5 **2.00ppm:** 2mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.6 **1.00ppm:** 1mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.7 **0.400ppm:** 4mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.1.8 **0.200ppm:** 1mL of 20ppm standard (Section 8.9.1.1.1) to 100mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler.

8.9.1.1.9 **0.100ppm:** 1mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler.

8.9.1.1.10 **0.050ppm:** 20mL of 0.100ppm standard (Section 8.9.1.1.8) to 40mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler.

8.9.1.2 Continuing Calibration Standards:

8.9.1.2.1 **0.400ppm Low CCV:** 4mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.2.2 **4.00ppm Hi CCV:** 8mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.2 Spiking solution, 200ppm as NH_3: To a 100mL volumetric flask add 20mL of 1000ppm Stock Standard (Section 8.9) and dilute to the mark with DI water.

8.10 Stock Standard Solution, 1000ppm as NH_3 (for ICV and LCS): Commercially prepared. Certificate of analysis is required. This must be from a different source than that used for Stock Standard (Section 8.9). Store refrigerated at 4 ± 2 °C. Expires upon manufacturer's specified date.

8.10.1 Initial Calibration Verification Standards (ICV):

8.10.1.1 Hi ICV, 10ppm: To a 100mL volumetric flask add 1mL of 1000ppm standard (Section 8.10). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation.

8.10.1.2 Hi ICV, 8.0ppm: To a 100mL volumetric flask add 0.8mL of 1000ppm standard (Section 8.10). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation

8.10.1.3 Low ICV, 1.0ppm: To a 100mL volumetric flask add 10mL of 10ppm ICV (Section 8.10.1.1). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation.

8.10.2 LCS solution, 200ppm as NH_3: To a 100mL volumetric flask add 20mL of 1000ppm Stock Standard (Section 8.10) and dilute to the mark with DI water. Store refrigerated at 4 ± 2 °C. Expires one month from date of preparation.

8.11 NaOH Pellets: ACS Grade. Store well sealed at room temperature. This salt does not have an expiration date.

8.12 Sulfuric Acid (Concentrated): Store at room temperature. Expires upon manufacturer's specified date. Use extreme caution when handling.

8.13 Ottawa sand: store at room temperature. Expires upon manufacturer's specified date.

8.14 Thymol Blue Sodium Salt 0.04% water solution: used as indicator during pH adjustment prior distillation. Store at room temperature. Expires upon manufacturer's specified date.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

9.1.1 The Distillation Blank is 100 mL DI for waters or 1g of Ottawa sand for soils. Distill one per batch of 20 samples or less. Results for the Distillation Blank must be less than the Reporting Limit. If results for the Distillation Blank are greater than the reporting limit, and all sample results for the batch are greater than 10x the Blank result, sample results may be reported with a narrative to be included on the final report. Otherwise, all samples must be redistilled and reanalyzed.

9.1.2 The Initial Calibration Blank (ICB) and Continuing Calibration Blank (CCB) for the Lachat analysis are not distilled and are 0.2% Boric Acid Solution (Section 8.8).

The ICB is run after the initial calibration verification standards (ICV) and CCB is run after the continuing calibration standards (CCV).

If results for the ICB are greater than the reporting limit, the ICB is reinjected. If failure continues the instrument must be recalibrated. Samples are not to be analyzed until an acceptable ICB is obtained.

If results for the CCB are greater than the reporting limit, the CCB is reinjected. If failure continues the instrument must be recalibrated. All samples that were analyzed since the last passing CCB have to be reanalyzed.

9.2 Laboratory Control Sample (LCS)

9.2.1 Distillation: Distill a Low and a Hi LCS with each batch of 20 samples or less. Recovery of the LCSs must be within +/- 10% of true value for method 350.1 for waters, and within in-house Control Limits for method 4500 and soils. If recovery is outside of this range, the standard is reanalyzed once. If recovery still fails criteria, the entire sample batch must be redistilled and reanalyzed.

9.2.1.1 Low 2ppm LCS: Add 1mL of 200ppm LCS solution (Section 8.10.2) to

100mL DI for waters or for 1.0g of Ottawa sand for soils. This is used for the Low 0 – 2ppm curve

9.2.1.2 **Hi 20ppm LCS:** Add 10mL of 200ppm LCS solution (Section 8.10.2) to 100mL DI for waters or to 1.0g of Ottawa sand for soils. This is used for the Hi 2-20ppm cuve.

9.3 Initial Calibration Verification (ICV)

9.3.1 **Lachat Analysis:** The ICVs are not distilled. Analyze the following Hi and Low ICVs after calibration of the Lachat instrument. Recoveries must be within 10% of the true value. If results are outside of this range, the ICV may be reinjected once. If failure continues, recalibration of the instrument is necessary. Samples are not to be analyzed until acceptable ICV results are obtained.

9.3.1.1 **Low ICV, 1.0ppm** (Section 8.10.1.2)

9.3.1.2 **Hi ICV, 10ppm (**Section 8.10.1.1). **HI ICV** will be **8.0** ppm (Section 8.10.1.2) in case of one board calibration.

9.4 Continuing Calibration Verification (CCV)

9.4.1 **Lachat Analysis:** The CCVs are not distilled. Analyze the following Low and Hi CCVs after every ten samples and at the completion of analysis. Recoveries must be within 10% of the true value.

If recoveries fall outside of this range, the CCV is reanalyzed. If recovery is acceptable, the analysis may continue. If recovery is again unacceptable, the cause for the failure is determined and corrected, and the instrument is recalibrated. All samples that were analyzed since the last CCV that was within range are reanalyzed.

9.4.1.1 **0.4ppm Low CCV** (Section 8.9.1.2.1)

9.4.1.2 **4.0ppm Hi CCV** (Section 8.9.1.2.2)

9.5 Matrix Spike

One matrix spike is prepped and analyzed per batch of **20 samples** or less for **method 4500**. Matrix spike has to be analyzed **for every 10 samples** or **less for method 350.1**. Prior to distillation, use 2mL of 200ppm Intermediate Stock Standard (Section 8.9.2) per 100mL of sample for waters or 1.0g of sample for soils. Recovery of the Matrix Spike must be within +/- 10% of true value for waters and within in-house Control Limits for soils. If the recovery is outside of acceptance criteria, and the LCS is within acceptance criteria, matrix interference may be assumed. The associated data is reported with a narrative to be included on the final report.

9.6 Laboratory Duplicate

Distill one duplicate sample per batch of 20 samples or less. The %RPD for the duplicate sample must be within in-house Control Limits. If results are outside of the acceptance criteria, a narrative to be included on the final report.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

9.8.1 Distillation Sequence

3 Rinse tubes
Blank
Low LCS
Hi LCS
Rinse
Samples
Rinse after suspected high samples
Duplicate
Spike
Rinse
Shut-Down

9.8.2 Lachat Analytical Sequence

Instrument Calibration
CCV: Low, Hi,CCB
Low ICV
Hi ICV
IC Blank
Samples
CCV: Low, Hi, CC Blank every 10 samples and at completion of analysis
Shut-Down

10. Procedure

10.1 Equipment Set-up

10.1.1 Prior to analysis, organize samples in order from low to high odor.

10.1.2 Clean 250mL Tecator tubes by rinsing twice with approximately 0.5mL of 6N NaOH solution and 100mL RO water. Rinse twice again with DI water.

10.2 Initial Calibration

Calibrate the Lachat ion analyzer according to manufacturer's instructions.

10.2.1 Calibration

Two boards are used to calibrate the Lachat instrument. Each curve has 7 calibration points. The correlation coefficient of each curve must be $\geq$ 0.995, otherwise re-calibration is necessary. Prepare standard curves by plotting the peak areas of standards processed through the manifold against NH_3-N concentrations in standards.

10.2.1.1 Channel 1 is used to generate a calibration curve on the low range from 0.00 – 2ppm.

10.2.1.2 Channel 2 is used to generate a calibration curve on the high range from 0 – 20ppm.

Alternative method: One board can be used to calibrate the Lachat instrument. 10 point calibration curve will be used with calibration standards 10.0, 8.0, 4.0, 2.0, 1.0,04, 0.2, 0.1, 0.05 mg/l each and blank. The correlation coefficient must

be ≥ 0.995, otherwise re-calibration is necessary. Prepare standard curves by plotting the peak areas of standards processed through the manifold against NH_3-N concentrations in standards.

Calibration coefficient will be calculated using Lachat software. All calibration points are back calculated by Lachat software and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse than 0.995.

10.2.2 Initial Calibration Verification (ICV)

Prior to sample analysis, an ICV is analyzed at 1.0ppm to verify the low calibration curve on Channel 1. Another ICV is analyzed at 10ppm to verify the high calibration curve on Channel 2. Both ICVs must yield results ± 10% of their true value, otherwise re-calibration is necessary.

Note: if instrument is calibrated using one board calibration, then both ICV's (Low and High), will be evaluated and high ICV will be 8.0 mg/l ICV standard (Section 8.10.1.2).

10.2.3 Initial Calibration Blank (ICB)

Following the ICV is the analysis of an ICB. The ICB consists of an aliquot of 0.2% Boric Acid (Section 8.8). Results must be less than the Reporting Limit.

10.3 Equipment Operation and Sample Processing

10.3.1 Sample Preparation

10.3.1.1 Sample Aliquots

10.3.1.1.1 Aqueous Samples

Add 100mL dechlorinated sample or a portion diluted to 100mL with DI water, to pre-washed Tecator tubes that are numbered to correspond with the samples.

10.3.1.1.2 Soil Samples

One gram of a well-homogenized sample is weighed out into a centrifuge tube (record weight in notebook). The sample is then transferred into a Tecator tube with 100 mL of DI.

10.3.1.1.3 QC Samples

In a similar manner, prepare the QC samples to be distilled with the batch (refer to Sections 9.1.1, 9.2, 9.4 and 9.6).

10.3.1.2 Add approximately 1.0mL of $Na_2S_2O_3$ to each tube in order to remove chlorine, followed by 5.0mL of Borate Buffer solution.

10.3.1.2.1 Aqueous Samples

Add small amounts of 6N NaOH until the pH is adjusted to 9.5. 2-3 drops of indicator (sec 8.14) can be used for pH adjustment. Color of solution will change from pink to blue at pH 9.5. Use pH paper to confirm pH reading. Check each sample with Potassium Iodide Starch paper (7.10) for chlorine presence. If starch paper showing presence of chlorine, add extra 1.0mL of $Na_2S_2O_3$ and check again.

10.3.1.2.2 Soil Samples

No pH adjustment is needed.

10.3.2 Distillation

10.3.2.1 **Equipment preparation:** To clean any prior contamination, first rinse the Tecator distillation unit by running a series of 3 rinse tubes. The first rinse containing 90mL DI and 10mL 6N NaOH. The second and third rinses are with100mL DI and 0.5mL 6N NaOH.

10.3.2.2 **Distillation:** Begin by distilling rinse tubes, Blank, Low and Hi LCS, and then Samples and QC Samples using the Tecator Instruments Automatic Distillation Unit according to the manufacturer's instructions.

Distillate is collected from the Tecator unit in 250mL disposable polypropylene cups that contain 20mL of 1.5% Boric acid (Section 8.7). Distillation is complete after collection of approximately 140mL. Fill each cup to 150mL with DI water. Cover and store at 1-4º C if Lachat analysis is to be delayed

10.3.3 Ammonia analysis of distillate: Follow the manufacturer's instructions for the proper operation of the Lachat ion analyzer. The following are specific notes for this analysis.

Sample throughput:	90 samples/hr; 40sec/sample
Pump speed:	35
Cycle period:	40 s
Inject to start of peak period:	25 s
Inject to end of peak period:	63 ± s

10.3.4 System Notes:

10.3.4.1 Allow 15 minutes for heating unit to warm up to 60ºC.

10.3.4.2 System IV GAIN: 175 X 1

10.3.4.3 If standards are not distilled, samples should be multiplied by 1.5 to correct for the increase in volume after the distillation.

10.3.4.4 If baseline drifts, peaks are too wide, or other problems with precision arise, clean the manifold by the following procedure:

10.3.4.4.1 Place all reagent lines in deionized water and pump to clear reagents (2-5 minutes).

10.3.4.4.2 Place reagent lines and carrier in 1M hydrochloric acid (1 volume concentrated HCl added to 11 volumes of deionized water) and pump for several minutes.

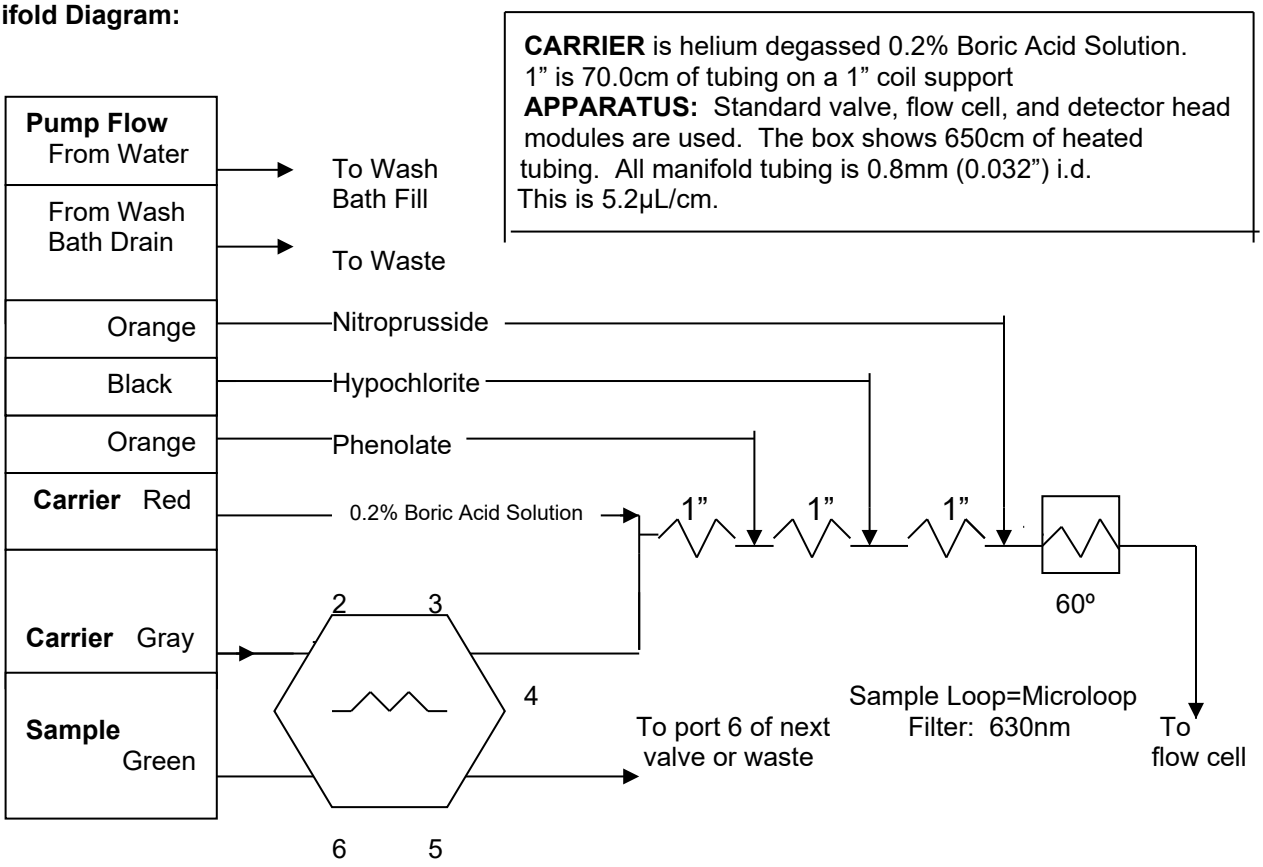
10.3.4.4.3 Place all lines in deionized water and pump until the HCl is thoroughly washed out.

10.3.4.4.4 Resume pumping reagents.

10.3.4.4.5 If sample distillate is colored or turbid, discard the sample distillate and re-distill the original sample at a higher dilution (Sections 10.3.1 and 10.3.2).

If there is not sufficient original sample volume for re-distillation, the colored/turbid distillate may be filtered through a 0.45µm filter and diluted on the Lachat instrument.

Manifold Diagram:



10.4 Continuing Calibration

Analyze the following after every 10 samples and at the completion of analysis:

0.400ppm Low CCV, (Section 8.9.1.2.1)

4.0ppm Hi CCV, (Section 8.9.1.2.2)

Blank, 0.2% Boric Acid Solution. (Section 8.8)

10.5 Preventive Maintenance

10.5.1 All lines are flushed at the end of each run.

10.5.2 All equipment is kept clean.

11. Data Evaluation, Calculations and Reporting

Compute sample NH_3-N concentration by comparing sample peak areas with standard curve, as determined by the Lachat instrument software.

11.1 If the sample has a concentration of less than 2ppm, calculate results by using the low curve generated on Channel 1.

11.2 If the sample concentration is greater than 2ppm but less than 20ppm, calculate results by using the high curve generated on Channel 2.

11.3 If the concentration is greater than 20ppm, dilute the sample and reanalyze.

NOTE: For soil samples Lachat reading is multiplied by extraction volume (150ml) and divided by sample weight (the exact weight is used as recorded in the laboratory notebook). Calculations are done using Lachat software.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If ICV/CCV and/or ICB/CCB fail acceptance criteria, check sections 9.1.2, 9.3.1 and 9.4.1 for correction actions.

If the Method Blank result exceeds the Reporting Limit (RL) for the analyte, the Blank is reanalyzed. If reanalysis will confirm positive Method blank result, data has to be evaluated and all associated samples have be re-distilled and reanalyzed.

If lab doesn't have samples for reanalysis, then samples with results below RL or more then 10x of reporting limit may be reported with narrative; samples with results between ND and 10x of reporting limit have to be B qualified and reported with narrative

If the LCS recovery falls outside the designated acceptance range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

If the Matrix Spike acceptance criteria is not met, the spiked sample is reanalyzed (if possible). If failure continues and if all other QC performance criteria are met, the data is reported and a narrative is included with the final report.

If the RPD for the Duplicate measurements falls outside the Laboratory defined control limits, the sample is reanalyzed (if possible). If failure continues, and if all other QC performance criteria are met, the data is reported and a narrative is included with the final report.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.
The quarterly method reporting limit (MRL) verification requirement is satisfied by the quarterly LOQ analysis, as per SOP/1732.

Note: Method **350.1** required MDL studies to be performed and evaluated every 6 month.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

NOTE: Lachat Phenol waste is hazardous and must be disposed of in "Hazmat" Lachat waste drums

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

16. Attachments

None.

Nitrogen, Total Kjeldahl

References: **Method 351.1,** Methods for Chemical Analysis of Water and Waste, EPA-600/4-79-020, U.S. Environmental Protection agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268 (March 1979)

Method SM 4500N_{org}-C, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method SM 4500NH3-H, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 10-107-06-2-D, Methods for Automated Ion Analyzers, May 20, 1998.

1. Scope and Application

Matrices: Total Kjeldahl nitrogen can be determined in potable, surface, and saline waters as well as domestic and industrial wastewaters.

Definitions: See Alpha Laboratories Quality Manual Appendix A

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Tecator and/or Lachat Instrument and in the interpretation of Lachat data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

This method covers the determination of total kjeldahl nitrogen in drinking, surface and saline waters, and domestic and industrial wastes. The procedure converts nitrogen components of biological origin such as amino acids, proteins and peptides to ammonia, but may not convert the nitrogenous compounds of some industrial wastes such as amines, nitro compounds, hydrazones, oximes, semicarbazones and some refractory tertiary amines.

In waters and wastewaters the forms of nitrogen of greatest interest are, in order of decreasing oxidation state, nitrate, nitrite, ammonia, and organic nitrogen. All these forms of nitrogen, as well as nitrogen gas (N_2), are biochemically interconvertible and are components of the nitrogen cycle. They are of interest for many reasons.

Organic nitrogen is defined functionally as organically bound nitrogen in the trinegative oxidation state. It does not include all organic nitrogen compounds. Analytically, organic nitrogen and ammonia can be determined together and have been referred to as “Kjeldahl nitrogen”, a term that reflects the technique used in their determination. Organic nitrogen includes such natural materials as proteins and peptides, nucleic acids and urea, and numerous synthetic organic materials. Typical organic nitrogen concentrations vary from a few hundred micrograms per liter in some lakes to more than 20mg/L in raw sewage.

Ammonia is present naturally in surface and wastewaters. Its concentration generally is low in groundwaters because it adsorbs to soil particles and clays and is not leached readily from soils. It is produced largely by deamination of organic nitrogen containing compounds and by hydrolysis of

urea. At some water treatment plants ammonia is added to react with chlorine to form a combined chlorine residual.

In the chlorination of wastewater effluents containing ammonia, virtually no free residual chlorine is obtained until the ammonia has been oxidized. Rather, the chlorine reacts with ammonia to form mono- and dichloramines. Ammonia concentrations encountered in water vary from less than 10µg ammonia nitrogen/L in some natural surface and groundwaters to more than 30 mg/L in some wastewaters.

In this discussion, organic nitrogen is referred to as organic N, nitrate nitrogen as NO_3–N, nitrite nitrogen as NO_2 - - N, and ammonia nitrogen as NH_3–N.

Total Kjeldahl nitrogen is defined as the sum of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate $(NH_4)_2SO_4$, under the conditions of digestion described below.

Organic Kjeldahl nitrogen is defined as the difference obtained by subtracting the free-ammonia value from the total Kjeldahl nitrogen value. This may be determined directly by removal of ammonia before digestion.

2. Summary of Method

The organic nitrogen is converted to ammonia via heating in the presence of concentrated sulfuric acid, K_2SO_4, $HgSO_4$, and evaporated until SO_3 fumes are obtained and the solution becomes colorless or pale yellow. The residue is cooled, diluted, and treated and made alkaline with a hydroxide-thiosulfate solution. The digestate is distilled at high pH into a solution of boric acid. The ammonia in the distillate is determined colorimetrically by the phenate method.

The phenate method is based on the Berthelot reaction. Ammonia reacts with alkaline phenol, then with sodium hypochlorite to form indophenol blue. Sodium nitroprusside (nitroferricyanide) is added to enhance sensitivity. The absorbance of the reaction product is measured at 630nm, and is directly proportional to the ammonia concentration in the digestate.

2.1 Method Modifications from Reference

This method has been modified for soil digestion, Section 9.4.1.

3. Detection Limits

The RDL is determined to be 0.3 mg/L for liquid samples and 150 mg/Kg for soil or solid samples.

4. Interferences

4.1 Instrumental

Samples with a high concentration of TKN may carry-over into the next sample and therefore yield false high results in that next sample. If a sample with a low concentration follows a sample with a high concentration, re-analyze the low sample to ensure results are accurate.

4.2 Parameters

High nitrate concentrations (10X or more than the TKN level) result in low TKN values. The reaction between nitrate and ammonia can be prevented by the use of an anion exchange resin (chloride form) to remove the nitrate prior to the TKN analysis.

5. Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

NOTE: Both Phenol and Mercury used in this method are hazardous and general laboratory safety practices must be observed. Due to the Mercury used in this procedure, digestion must be done under a hood.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, and Handling

6.1 Sample Collection

The most reliable results are obtained on fresh samples. Use plastic or glass containers.

6.2 Sample Preservation

Samples may be preserved by addition of 2mL of concentrated H_2SO_4 per liter if sample cannot be analyzed immediately. Refrigerate at 4ºC.

6.3 Sample Handling

Even when properly preserved, conversion of organic nitrogen to ammonia may occur. Therefore samples should be analyzed as soon as possible.

7. Equipment and Supplies

7.1 Digestion apparatus: Kjeldahl Digestion System 20, Model 1015 Digester. Follow the instrument manufacturer's instructions.

7.2 Distillation apparatus: Tecator Instruments Automatic Distillation Unit: Follow the instrument manufacturer's instructions for proper operation.

7.3 Automated Ion Analyzer: Lachat Instruments

7.4 Disposable polypropylene cups: 250mL with covers.

7.5 Glass Tuttlecaps: For digestion.

7.6 Glass Pipets: Various volumes.

7.7 Auto-pipettor with tips: For 10mL capability.

7.8 Analytical balances: capable to weigh 0.1000g aliquots.

7.9 Dilu Vials

7.10 Boiling chips

8. Standards and Reagents

8.1 Sodium Phenolate: **CAUTION!** Wear gloves. Phenol causes severe burns and is rapidly absorbed into the body through the skin. In a 1L volumetric flask, dissolve 88mL of 88% liquified phenol or 83g crystalline phenol (C_6H_5OH) in approximately 600mL DI water. While stirring, slowly add 32g sodium hydroxide (NaOH). Cool, dilute to the mark, and invert three times. Do **not** degas this reagent. Expires one month from date of preparation.

8.2 Sodium Hypochlorite (approximate 2.6%): In a 500mL volumetric flask, dilute 250mL Regular Chlorine bleach [5.25% sodium hypochlorite (NaOCl)] to the mark with DI water. Invert three times to mix. Expires one month from date of preparation.

8.3 Sodium Nitroprusside (coloring agent): In a 1L volumetric flask, dissolve 3.50g sodium nitroprusside (Sodium Nitroferricyanide $[Na_2Fe(CN)_5NO_2 \cdot H_2O]$) dilute to the mark with DI water. Degas with helium to prevent bubble formation. Use He at 140kPa (20 lb/in^2) through a helium degassing tube. Bubble He vigorously through the solution for one minute. Expires one month from date of preparation.

8.4 Boric 1.5% Boric Acid Solution: To a 1000mL volumetric flask add 15g Boric Acid. Dilute to the mark with DI water. Expires one month from date of preparation.

8.5 Mercuric Sulfate Solution: Dissolve 8g red mercuric oxide (HgO) in 50mL of l:4 sulfuric acid (10.0mL concentrated H_2SO_4 : 40mL distilled water) and dilute to 100mL with distilled water. Expires one month from date of preparation.

8.6 Digestion Solution (Sulfuric Acid-Mercuric Sulfate-Potassium Sulfate): Dissolve 267g K_2SO_4 in 1300mL distilled water and 400mL concentrated H_2SO_4. Add 50mL mercuric sulfate (Section 8.5) solution and dilute to 2L with distilled water. Expires one month from date of preparation.

8.7 Sodium Hydroxide-Sodium Thiosulfate Solution: Dissolve 500g NaOH and 25g $Na_2S_2O_3 \cdot 5H_2O$ in distilled water and dilute to 1L. Expires one month from date of preparation.

8.8 0.2% Boric Acid Solution (Carrier Solution): To a 2L volumetric flask, dissolve 4g Boric Acid (H_3BO_3) in DI water. Degas by bubbling vigorously with Helium for one minute. Expires one month from date of preparation.

8.9 Stock Standard, 1000ppm as NH_3 (for calibration solutions): Commercially prepared. Certificate of analysis is required.

8.9.1 Intermediate Calibration Stock Standard, 100ppm as NH_3: To a 100mL volumetric flask, add 10.0mL of Stock Standard (Section 8.9) and dilute to the mark with 0.2% Boric Acid solution (Section 8.8). Invert three times. Expires one month from date of preparation.

8.9.1.1 Nine Working Calibration Standards: Prepare the following standards in volumetric flasks:

8.9.1.1.1 20.0ppm: 40mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.2 10.0ppm: 20mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.3 8.00ppm: 8mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.4 4.00ppm: 8mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.5 2.00ppm: 2mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.6 1.00ppm: 1mL of 100ppm standard (Section 8.9.1) to 100mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.7 0.400ppm: 4mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Prepare fresh each day of use.

8.9.1.1.8 0.200ppm: 1mL of 20ppm standard (Section 8.9.1.1.1) to 100mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler. Prepare fresh each day of use.

8.9.1.1.9 0.100ppm: 1mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler. Prepare fresh each day of use.

8.9.1.1.10 0.050ppm: 20mL of 0.100ppm standard (Section 8.9.1.1.9) to 40mL with 0.2% Boric Acid solution (Section 8.8). Alternately, this standard may be prepared utilizing autodilution of the 20ppm standard on the autosampler. Prepare fresh each day of use.

8.9.1.2 Continuing Calibration Standards:

8.9.1.2.1 0.400ppm Low CCV: 4mL of 20ppm standard (Section 8.9.1.1.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.9.1.2.2 4.00ppm Hi CCV: 8mL of 100ppm standard (Section 8.9.1) to 200mL with 0.2% Boric Acid solution (Section 8.8).

8.10 Stock Standard, 1000ppm as TKN (for spike): Commercially prepared. Certificate of analysis is required

8.10.1 Intermediate spike Stock Standard, 200ppm as TKN: To a 100mL volumetric flask, add 20.0mL of Stock Standard (Section 8.10) and dilute to the mark with 0.2% Boric Acid solution (Section 8.8). Invert three times. Expires one month from date of preparation.

8.11 Stock Standard Solution, 1000ppm as NH_3 (for ICV only): Commercially prepared. Certificate of analysis is required. This must be from a different source than that used for Stock Standard (Section 8.9).

8.11.1 Initial Calibration Verification Standards (ICV):

8.11.1.1 Hi ICV, 10ppm: To a 100mL volumetric flask add 1mL of 1000ppm standard (Section 8.11). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation.

8.11.1.2 Hi ICV, 8.0ppm: To a 100mL volumetric flask add 0.8mL of 1000ppm standard (Section 8.11). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation.

8.11.1.3 Low ICV, 1.0ppm: To a 100mL volumetric flask add 10mL of 10ppm ICV (Section 8.11.1.1). Dilute to the mark with 0.2% Boric Acid Solution (Section 8.8). Expires one month from date of preparation.

8.12 Stock Standard Solution, 1000ppm as TKN (for LCS): Commercially prepared. Certificate of analysis is required. This must be from a different independent source than that used for Stock Standard (Section 8.10).

8.12.1 LCS solution, 200ppm as TKN: To a 100mL volumetric flask add 20mL of 1000ppm Stock Standard (Section 8.12) and dilute to the mark with DI water. Expires one month from date of preparation.

8.13 6N Sodium Hydroxide Solution : Dissolve 240g NaOH in distilled water and dilute to 1L. Expires six month from date of preparation.

9. Procedure

9.1 SET-UP

9.1.1 Clean 250mL Tecator tubes by rinsing twice with approximately 0.5mL of 6N NaOH solution and 100mL RO water. Rinse twice again with DI water.

9.1.2 Rinse glass tuttlecaps under the hood in a 1000mL beaker with approximately 500mL DI and 1.0 mL of 6N NaOH. Allow to sit in this solution until use.

9.2 Initial Calibration

Calibrate the Lachat ion analyzer according to manufacturer's instructions.

9.2.1 Calibration

Two boards are used to calibrate the Lachat instrument. Each curve has 7 calibration points. The correlation coefficient of each curve must be $\geq$ 0.995, otherwise re-calibration is necessary. Prepare standard curves by plotting the peak areas of standards processed through the manifold against NH_3-N concentrations in standards.

9.2.1.1 Channel 1 is used to generate a calibration curve on the low range from 0.00 – 2ppm.

9.2.1.2 Channel 2 is used to generate a calibration curve on the high range from 0 – 20ppm.

Alternative method: One board can be used to calibrate the Lachat instrument. Ten point calibration curve will be used with calibration standards 10.0, 8.0, 4.0, 2.0, 1.0, 0.4, 0.2, 0.1, 0.05 mg/l each and blank. The correlation coefficient must be $\geq$ 0.995, otherwise re-calibration is necessary. Prepare standard curves by plotting the peak areas of standards processed through the manifold against NH_3-N concentrations in standards

Calibration coefficient will be calculated using Lachat software. All calibration points are back calculated by Lachat software and should be within 10% from true concentration, except 2 lowest points of calibration curve. % recoveries for the low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse than 0.995. The standards for the calibration curve are recalculated manually to prove the software calculations are within +/- 10% of the true concentrations.

9.2.2 Initial Calibration Verification (ICV)
Prior to sample analysis, an ICV is analyzed at 1.0ppm (Section 8.10.1.2) to verify the low calibration curve on Channel 1. Another ICV is analyzed at 10ppm (Section 8.10.1.1) to verify the high calibration curve on Channel 2. Both ICVs must yield results ± 10% of their true value, otherwise re-calibration is necessary.

Note: if instrument is calibrated using one board calibration, then both ICV's (Low and High), will be evaluated and high ICV will be 8.0 mg/l ICV standard (Section 8.11.1.2)

9.2.3 Initial Calibration Blank (ICB)
Following the ICV is the analysis of an ICB. The ICB consists of an aliquot of 0.2% Boric Acid (Section 8.8). Results must be less than < 0.05mg/L.

9.3 Standardization (Continuing Calibration Verification)

Analyze the following after every 10 samples and at the completion of analysis:

0.400ppm Low CCV, (Section 8.9.1.2.1)

4.0ppm Hi CCV, (Section 8.9.1.2.2)

Blank, 0.2% Boric Acid Solution. (Section 8.8)

9.4 Equipment Operation and Sample Analysis

9.4.1 Aqueous Sample Digestion: Add 50mL of homogenized sample or a portion diluted to 50mL with DI water, to pre-washed Tecator tubes that are numbered to correspond with the samples.

Soil/Solid Sample Digestion: Weigh 0.1g of homogenized soil/solid sample into dilu vial and record the weight in the laboratory notebook. Transfer to a pre-washed, pre-numbered Tecator tube and add 50mL of DI water.

In a similar manner, for each matrix, prepare the QC samples to be digested with the batch (refer to Sections 10.2.1, 10.3.1, 10.7 and 10.8)

Then add approximately 1g of black boiling chips to each tube. Move to hood before adding 10mL of Digestion Solution (Section 8.6) to each tube with a calibrated pipettor.

Rinse glass tuttlecaps with DI and place one onto the top of each Tecator tube. Place Tecator tube rack onto Tecator digestion block and heat to 380°C. Cook for approximately 2 hours to SO_3 fumes. The remaining mixture will be clear or pale yellow in color. Remove tube from digestion block and allow to cool to ~80 °C before adding DI to the 90mL mark on the tube.

9.4.2 Distillation: To minimize contamination, leave distillation apparatus assembled after steaming out and until just before starting sample distillation. Make the digestate alkaline by careful addition of 20mL of sodium hydroxide-thiosulfate solution (Section 8.7) without mixing. Do not mix until the digestion tube has been connected to the distillation apparatus. Connect the Tecator tube to the Tecator distillation unit's stopper as defined in the manual. Distill for 3 minutes 40 seconds as defined in the manual and collect distillate in 20mL boric acid solution (Section 8.4). Distill at maximum rate with the tip of the delivery tube below the surface of boric acid receiving solution. Collect at least 90mL distillate. Dilute to 150mL with DI water. Refrigerate at 0-4ºC if Lachat analysis is delayed.

9.4.3 Ammonia analysis of distillate: Follow the manufacturer's instructions for the proper operation of the ion analyzer. The following are specific notes for this analysis.

Sample throughput:	90 samples/hr; 40 sec/sample
Pump speed:	35
Cycle period:	40 s
Inject to start of peak period:	25 s
Inject to end of peak period:	63 s

9.4.4 System Notes:

9.4.4.1 Allow 15 minutes for heating unit to warm up to 60ºC.

9.4.4.2 System IV GAIN: 175 x 1.

9.4.4.3 If standards are not distilled, samples should be multiplied by procedure dilution (final volume 150mL) divided by initial volume.

9.4.4.4 If baseline drifts, peaks are too wide, or other problems with precision arise, clean the manifold by the following procedure:

9.4.4.4.1 Place all reagent lines in deionized water and pump to clear reagents (2-5 minutes).

9.4.4.4.2 Place reagent lines and carrier in 1M hydrochloric acid (1 volume concentrated HCl added to 11 volumes of deionized water) and pump for several minutes.

9.4.4.4.3 Place all lines in deionized water and pump until the HCl is thoroughly washed out.

9.4.4.4.4 Resume pumping reagents.

9.4.4.5 If samples are colored or are suspected to show a background absorbance, this interference should be subtracted. This can be done by diluting or by the following procedure:

9.4.4.5.1 Calibrate the system in the normal manner.

9.4.4.5.2 Disable the check standard or DQM features and analyze the samples.

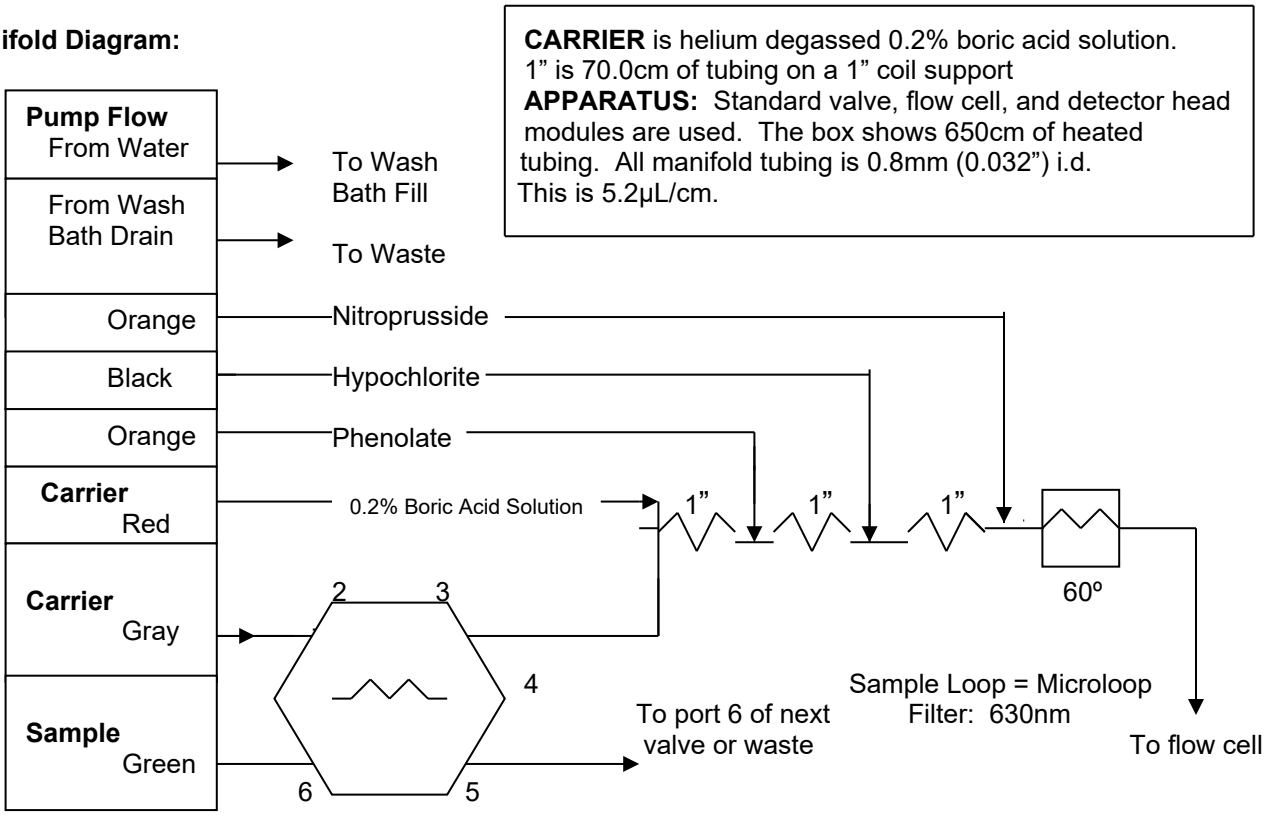
9.4.4.5.3 Place reagent and carrier lines in DI water and allow the baseline to stabilize.

9.4.4.5.4 Inject samples again without recalibrating.

9.4.4.5.5 Subtract the "background" concentration from the original concentration to give the corrected concentration.

Original Concentration – Background Concentration = Corrected Concentration.

Manifold Diagram:



9.5 Preventative Maintenance

9.5.1 All lines are flushed at the end of each run.

9.5.2 All equipment is kept clean.

9.6 Calculations

Prepare standard curves by plotting peak areas of standards processed through the manifold against NH_3 -N concentrations in standards. Compute sample NH_3 -N concentration by comparing sample peak areas with standard curve, as determined by the Lachat instrument software.

9.6.1 If the sample has a concentration of less than 2ppm, calculate results by using the low curve generated on Channel 1.

9.6.2 If the sample concentration is greater than 2ppm, calculate results by using the high curve generated on Channel 2.

Alternative method: use only one Channel and calculate all results from one channel.

To compute final results for aqueous samples, multiply the direct reading by the dilution factor based on the initial preparation volume.

TKN mg/L = mg/L direct reading x dilution factor

To compute results for soil/solid samples, multiply the direct reading by the extraction final volume (150mL) and then divide by the weight of the sample used for extraction (Section 9.4.1), and multiply by a dilution factor as necessary.

$$\text{TKN mg/Kg} = \left[\frac{(\text{mg/L direct reading}) \times \text{extraction final volume (150mL)}}{\text{Sample weight (g)}} \right] \times \text{dilution factor}$$

10. Quality Control and Data Assessment

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation.

10.1 Demonstration of Capability

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. Each time a method modification is made, the analyst is required to repeat the procedure.

When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must locate and correct the source of the problem and repeat the test for failed parameters of the method.

Repeated failure confirms a general problem with the measurement system or analytical technique of the analyst. If the failure repeats, locate and correct the source of the problem and repeat the test for all parameters listed in the method.

10.2 Blank

10.2.1 The Distillation Blank is 50mL of DI. Distill one per batch of 20 samples or less. Subtract any blank greater than 0.3mg/L from all samples and QC.

10.2.2 The Analytical Blank (ICB) for the Lachat analysis is not distilled and is 0.2% Boric Acid Solution (Section 8.8).

The ICB is run after the initial calibration verification standards (ICV) and another is run after the continuing calibration standards (CCV).

10.3 Laboratory Control Samples (LCS)

10.3.1 Distillation: Distill a Low and a Hi LCS with each batch of 20 samples or less. The results from the Hi LCS are reported for the batch. The Low LCS is used to verify the low curve, but is not reported.

10.3.1.1 Low 4.0ppm LCS: Add 1mL of 200ppm LCS solution (Section 8.10.2) to 50 mL DI. This is used for the Low 0 - 6 ppm curve.

10.3.1.2 Hi 40ppm LCS: Add 10mL of 200ppm LCS solution (Section 8.10.2) to 50 mL DI. This is used for the Hi 0 - 60 ppm curve.

10.4 Initial Calibration Verification Standards

10.4.1 Lachat Analysis: The ICVs are not distilled. Analyze the following after calibration of the Lachat instrument. Recoveries must be within 10% of the true value, otherwise recalibration of the instrument is necessary.

10.4.1.1 Low ICV, 1.0ppm (Section 8.10.1.2)

10.4.1.2 Hi ICV, 10ppm (Section 8.10.1.1); **HI ICV** will be 8.0 ppm (Section 8.10.1.2) in case of one board calibration.

10.5 Continuing Calibration Verification Standards

10.5.1 Lachat Analysis: The CCVs are not distilled. Analyze the following after every ten samples and at the completion of analysis. Recoveries must be within 10% of the true value.

If recoveries fall outside of this range, the cause for the failure is determined and corrected, and the instrument is recalibrated. All samples that were analyzed since the last CCV that was within range are reanalyzed.

10.5.1.1 0.4ppm Low CCV (Section 8.9.1.2.1)

10.5.1.2 4.0ppm Hi CCV (Section 8.9.1.2.2)

10.6 Interference Check Standards

None.

10.7 Matrix Spike

One per batch of 20 samples or less. Prior to distillation, add 2mL of 200ppm Spiking Solution (Section 8.10.1) per 50mL of sample.

10.8 Duplicates

Distill one duplicate sample per batch of 20 samples or less.

10.9 Control Limits

The laboratory maintains performance records to document the quality of data that is generated. Method accuracy for samples is assessed and records maintained.

Control limits for the method parameters are generated by the QC staff. The control limits are calculated based on in-house performance data. The limits are compared to the control limits found in the reference method.

10.10 Analytical Sequences

10.10.1 Distillation Sequence:

3 Rinse tubes
Blank 1
Low LCS
Hi LCS
Rinse
Samples (each sample must be followed by rinse, except between sample and its spike or sample and its duplicate)
Duplicate
Spike
Rinse
Shut-down

10.10.2 Lachat Analytical Sequence:

Instrument Calibration
DQM = Hi 4.0ppm CCV
Low 0.4ppm CCV
CC Blank
Low 1.0ppm ICV
Hi 10ppm ICV or 8.0 ppm ICV
IC Blank
Samples
DQM : Run after every 10 samples and at completion of analysis
Rinse reagent lines with 1M HCl for 5 to 10 minutes
DI water rinse for 5 to 10 minutes
Air rinse 5 to 10 minutes.
Shut-Down.

11. Method Performance

11.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732 These studies performed by the laboratory are maintained on file for review.

The quarterly method reporting limit (MRL) verification requirement is satisfied by the quarterly LOQ analysis, as per SOP/1732.

11.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734, 1739 for further information regarding IDC/DOC Generation.

11.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

11.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

12. Corrective Actions

Holding time exceedence, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the ICV/CCV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Pollution Prevention

See Chemical Hygiene Plan for pollution prevention operations.

14. Waste Management

See Chemical Hygiene Plan SOP/1728 for waste handling and disposal.

NOTE: TKN Lachat waste contains Mercury and must be deposited into TKN/Lachat waste stream in the Waste Room.

Oil and Grease

Total Petroleum Hydrocarbons by

n-Hexane Extraction and Gravimetric Method

References: EPA 9071B: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGTHEM; Non-polar Material) by Extraction and Gravimetry. April 1998.

EPA1664 A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGTHEM; Non-polar Material) by Extraction and Gravimetry. EPA-821-R-98-002. February 1999.

1. Scope and Application

Matrices: This method, as outlined, is limited to soils, sludges, sediment, and solid samples.

Definitions: See Alpha Analytical Quality Manual Appendix A

This method is for the determination of n-hexane extractable material (HEM) in soil, sediment, sludges, and other solid material amenable to chemical drying and solvent extraction with n-hexane. Extractable materials that may be determined are relatively non-volatile hydrocarbons, vegetable oils, animal fats, waxes, soaps, greases and related materials. Method 9071 employs n-hexane as the extraction solvent with Soxhlet extraction.

This method is not applicable to measurement of materials that volatilize at temperatures below approximately 85ºC. Petroleum fuels from gasoline through #2 fuel oil may be partially lost in the solvent removal operation.

Some crude oils and heavy fuel oils contain a significant percentage of materials that are not soluble in n-hexane. Accordingly, recoveries of these materials may be low.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

2. Summary of Method

A representative portion is mixed with anhydrous sodium sulfate to dry the sample.

The sample, after drying, is extracted with n-hexane using a Soxhlet apparatus. The n-hexane extract is then distilled from the extract and the HEM is desiccated and weighed.

A separate portion is evaluated for percent solids to make a dry-weight HEM concentration.

The amount of oil and grease is calculated gravimetrically.

For TPH-1664 determination, an amount of silica gel proportionate to the oil and grease result is added to the solution containing the redissolved oil and grease. The solution is then stirred and filtered through sodium sulfate. The solution is then dried and the amount of TPH is calculated gravimetrically.

2.1 Method Modifications from Reference

The laboratory does not use magnesium sulfate monohydrate to dry acidified samples. Samples are used as they are received. In place of evaporating the extract in a waterbath, the use of a Speedvap 9000 has been substituted. MDL Studies utilizing the Speedvap 9000 show no quantitative difference with the boil-down in a waterbath. Using Speedvap technology, the laboratory proved that samples have constant weight during one drying cycle; laboratory doesn't repeat drying, cooling, desiccating and weighing process multiple times

3. Reporting Limits

The reporting limit is 200 mg/Kg when analyzing a 10 g sample.

4. Interferences

4.1 Instrumental

Improperly prepared glassware may result in interferences.

Sodium sulfate may cause interferences, if it is powder form instead of granular.

4.2 Parameters

Samples containing particulates or detergents may cause interferences. Smaller sample volumes may need to be collected in these cases.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The Hexane used in this method is hazardous, as well as flammable. This chemical should be used under a laboratory hood and stored in a flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

A minimum 100 g of sample should be collected using a metal spatula, spoon or equivalent device. Samples should be collected into a pre-cleaned wide-mouth glass container fitted with a TFE-lined screw cap.

6.2 Sample Preservation

Not applicable.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

A holding time has not been established, but it is recommended to analyze as soon as possible.

7. Equipment and Supplies

7.1 Soxhlet extraction apparatus

7.2 Heating mantle- explosion proof, with temperature control

7.3 Boiling flask- 125mL or appropriate size

7.4 Chiller

7.5 Speedvap Evaporator: With spark-proof pump.

7.6 Aluminum Weighing Dishes: Disposable.

7.7 Paper extraction thimble for Soxhlet apparatus.

7.8 Fume Hood

7.9 Desiccator

7.10 Analytical Balance: Capable or weighing to 0.1mg.

7.11 Glass wool

7.12 Porcelain mortar and pestle

7.13 Pipets: 5mL and 10 mL

7.14 Erlenmeyer flasks: 125mL, 250mL

7.15 Glass fiber filter paper – Whatman No. 40 or equivalent

7.16 Boiling chips – Silicon carbide or fluoropolymer

7.17 Glass funnel

7.18 pH Paper: Range 1 – 12 pH units

7.19 Magnetic Stirring Bar

7.20 Filter Paper

7.21 Stirring Plate

8. Reagents and Standards

8.1 Hexadecane / Stearic Acid Stock 4mg/mL and 8 mg/mL (LCS Standard and Spiking Solution): Commercially prepared and purchased. (Available from Environmental Express and Horizon.) Maintain capped and stored in the dark at room temperature. Expires upon manufacturer's specified date

NOTE: The LCS Standard must be from a different source than that used for the Spiking Solution.

8.2 Acetone: ACS residue less than 1mg/L. Store in flammables cabinet. Expires upon manufacturer's specified date.

8.3 Hydrochloric Acid: HCl, 1:1. Store in flammables cabinet. Expires upon manufacturer's specified date.

8.4 n-Hexane: 85% purity, 99.0% minimum saturated C_6 isomers, residue less than 1mg/L. Boiling point, 69°C. Store in flammables cabinet. Expires upon manufacturer's specified date.

8.5 Sodium Sulfate: Na_2SO_4, anhydrous granular crystals. Store in flammables cabinet. Expires upon manufacturer's specified date.

8.6 Magnesium sulfate monohydrate: Prepare $MgSO_4 \cdot H_20$ by spreading a thin layer in a dish and drying in an oven at 150°C overnight. Store in a tightly sealed glass container until used.

8.7 Silica Gel: JT Baker 60-200 mesh, chromatography grade. Activated by baking at 140 °C for a minimum of 14 hours in a shallow tray. Stored in closed glass containers, in a desiccator. All references to silica gel in this method refer to this prepared reagent.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Analyze one Blank per batch of 20 samples or less. The method blank must be <40 mg/Kg. If the method blank does not meet this criterion, the entire batch is suspect and must be re-extracted.

9.2 Laboratory Control Sample (LCS)

Analyze one LCS per batch of 20 samples or less. The percent recovery must be within the acceptance range of 79-114% for oil and grease, and 64 – 132% for TPH, per the reference method. If this criterion is not met the LCS is reanalyzed. If failure continues a narrative is submitted with the data for inclusion on the Client report.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Analyze one matrix spike sample per batch of 20 samples or less. A matrix spike concentration must be approximately 1-5 times greater than the concentration of the sample being spiked. The percent recovery must be within the acceptance range of 79% to 114% for oil and grease, and 64 – 132% for TPH, per the reference method. If the percent recovery is outside of these limits, a narrative is submitted with the data for inclusion on the Client report.

9.6 Laboratory Duplicate

Analyze one sample in duplicate per batch of 20 samples or less. The relative difference must be equal or less than 18% for oil and grease, and equal or less than 34% for TPH. If the %RPD is outside of this criterion, a narrative is submitted with the data for inclusion on the Client report.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

1. Weigh out the sample and air dry overnight.
2. Blend with sodium sulfate and add to extraction thimble.
3. Extract in Soxhlet apparatus for 4 hours.
4. Filter the extract into a beaker and rinse flask with solvent.
5. Pour the sample into a weighed aluminum dish.
6. Evaporate the sample using the Speedvap 9000.
7. Place the aluminum dishes in a desiccator for 10 minutes.
8. Re-weigh the aluminum dishes.
9. Calculate sample results.
10. TPH-1664
    - Clean-up the sample and QC sample extracts with silica gel.
    - Repeat steps 4 – 8.

10. Procedure

10.1 Equipment Set-up

10.1.1 Clean all glassware.

10.1.2 Turn on the Soxhlet apparatus. Then turn on the chiller.

10.1.3 Check fittings to ensure tightness.

10.1.4 Add boiling chips to boiling flasks containing 90 mL of n-hexane.

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Weigh out 10g of sample and dry overnight.

10.3.2 Mix with 10g of sodium sulfate to remove any remaining liquid.

10.3.3 Transfer homogenized mixture into an extraction thimble and than into the Soxhlet.

10.3.4 Preparation of QC Samples:

10.3.4.1 **Blank:** Add 10g of sodium sulfate to a clean extraction thimble.

10.3.4.2 **LCS:** Add 10g of sodium sulfate to a clean extraction thimble. Slowly add 5mL of LCS Stock Standard (Section 8.1), by touching the pipet tip to the side of the Soxhlet. Release the contents of the pipet very slowly. (For best recoveries use a warmed LCS Solution.)

10.3.4.3 **Matrix Spike:** To a second aliquot of a chosen sample, slowly add 10mL of the Spiking Solution (Section 8.1) directly onto the sample. Use the same procedure described above in Section 10.3.4.2.

10.3.5 Connect the Soxhlet to the cold fingers and boiling flasks with the sample.

10.3.6 Heat the samples until a cycle rate of 20 cycles/hr is obtained and extract for 4 hours

10.3.7 At the end of the 4 hour extraction period, filter the hexane phase through a filter funnel lined with filter paper that contains sodium sulfate, and into an Erlenmeyer flask. Rinse the boiling flask and sodium sulfate with n-hexane and add to the flask. Rinse the filter funnel with hexane.

NOTE: Sometimes a slightly cloudy white layer will appear between the hexane layer and the colored water layer. Do not transfer this layer to the weighing dish.

10.3.8 Pre-weigh an empty aluminum weighing dish to the nearest 0.0001g. Record weight in the Laboratory Notebook. Transfer the filtered hexane sample into the weighed aluminum dish.

10.3.9 Turn Speedvap 9000. (Switch is on back of instrument.) Turn dial all the way to the right. Push <Release Cover> button and put weighing dishes containing the extracted oil and grease in hexane in the Speedvap.

10.3.10 Allow the evaporator to continue to run until the dish reaches dryness. This takes about 8-10 minutes. **Do Not Over-dry. Do this in a fume hood.**

10.3.11 Turn off Speedvap using button on back. Place the dishes in a desiccator for about 10 minutes.

10.3.12 Re-weigh the aluminum dishes and record weight in the Laboratory Notebook.

Note 1: Record lot numbers for SPE disks, reagents and solvents used for extraction in Laboratory Notebook.

Note 2: On each day of use, the laboratory balances are verified to be accurate at weights that bracket the weight of use. The balance verification performed both before and after a sample batch must pass within the acceptance criteria outlined in the Balance Calibration Verification SOP (SOP ID 1730). If the balance verification is outside of acceptance criteria, the problem must be corrected and the sample extracts re-weighed.

10.3.13 Clean-up Procedure for TPH-1664 Analysis

Using Hexane, rinse the oil and grease from the aluminum dish into a 250mL Erlenmeyer flask. Add a stir bar and silica gel. The amount of silica gel is dependent upon the concentration of oil and grease determined in the sample. For every 100ppm of oil and grease, add 3 grams of silica gel. Place the flask on the stirplate and stir for 5 minutes. Repeat Sections 10.3.7 – 10.3.12.

NOTE: All QC samples in the batch must be treated in the same manner as samples. Therefore, if TPH-1664 is to be determined, the Blank, LCS, matrix spike and duplicate must also go through the TPH-1664 clean-up procedure along with the samples.

10.4 Continuing Calibration

Not applicable.

10.5 Preventive Maintenance

Preventative maintenance is conducted per the manufacturer's instructions. All maintenance is recorded in the Instrument Maintenance Logbook. Rinsing of the lines with a 50/50 Acetone and Hexane mixture is done daily to keep the lines clean.

11. Data Evaluation, Calculations and Reporting

$$\text{mgHEM / Kg} = \frac{\text{Final weight of dish – tared weight}}{\text{weight}} \times 1000$$

Where: weight = dry weight of sample in grams

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the LCS recovery falls outside the designated acceptance range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

Organochlorine Pesticides
By Capillary Column Gas Chromatography

Reference Method No.: **Method 8081B**

References: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, 2007.

Mass DEP CAM QC Requirements and Performance Standards for the Analysis of Chlorinated Pesticides by Gas Chromatography (GC), Revision 2, April 12, 2024.

SW-846 Pesticides by 8081, State of Connecticut Department of Environmental Protection Reasonable Confidence Protocol (RCP), Version 3.0, May 2024

1. Scope and Application

Method 8081B is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices. This SOP details the analysis for these compounds using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).

Matrices: Extracts from solid and liquid matrices.

Definitions: See Pace Analytical Quality Manual

Regulatory Parameter List: The compounds listed below are determined by this method:

Parameter	CAS
Aldrin	309-00-2
Alpha-BHC	319-84-6
Beta-BHC	319-85-7
Gamma-BHC (Lindane)	58-89-9
Delta-BHC	319-86-8
Alpha-chlordane	5103-71-9
Gamma-chlordane	5103-74-2
4,4'-DDD	72-54-8
4,4'-DDE	72-55-9
4,4'-DDT	50-29-3
Dieldrin	60-57-1
Endosulfan I	959-98-8
Endosulfan II	33213-65-9
Endosulfan Sulfate	1031-07-8
Endrin	72-20-8
Endrin Aldehyde	7421-93-4
Endrin Ketone	53494-70-5
Heptachlor	76-44-8
Heptachlor Epoxide	1024-57-3
Methoxychlor	72-43-5
Toxaphene	8001-35-2
Chlordane	57-74-9
Alachlor [+]	15972-60-8
Hexachlorobenzene [++]	118-74-1

[+] CT-RCP only compound [++] MCP/CT only compound

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer

This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph (GC) and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (see section 13), analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the QA Officer and/or Laboratory Director on a case-by-case basis.

2. Summary of Method

A measured volume or weight of sample (for liquids: 100mL, for solids: 15g) is extracted using the appropriate matrix-specific sample extraction technique.

Liquid samples are extracted at neutral pH with methylene chloride using Method 3510C (separatory funnel), or other appropriate technique.

Solid samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet), Method 3546 (microwave extraction), or other appropriate technique.

Wipe samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet) or other appropriate technique.

Oil samples are diluted with hexane following the procedure outlined in the extraction SOP.

After cleanup, the extract is analyzed by injecting a 1µL sample into a gas chromatograph equipped with narrow- or wide-bore fused silica capillary columns and electron capture (GC/ECD) detectors.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 1 lists our routine reporting limits.

4. Interferences

4.1 Only high purity gases are used in the GC system to eliminate this source of possible contamination. Both the hydrogen (carrier gas – 99.999%) and argon-methane (detector make-up gas) are certified by the gas supplier.

4.2 Preventive instrument maintenance is performed routinely, and whenever highly contaminated extracts are analyzed that could result in chromatographic interferences or result in degradation of system performance. Section 9.5 details the maintenance steps.

4.3 Glassware must be scrupulously cleaned. This procedure is detailed in the extraction SOPs. Store dry glassware in a clean environment.

4.4 All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free. All of these materials are routinely determined to be free of interferences by analysis of extraction blanks with every extraction batch performed.

4.5 Certain compounds (i.e. phthalates) can be extracted from the sample matrix and be detected by the ECD that could possibly result in false positive results or complicate the data interpretation. The use of the cleanup procedures detailed in the extraction SOPs minimize these possible interferences. Analyst experience is also crucial in making compound determinations.

4.6 Interferences co-extracted from the samples will vary considerably from waste to waste. While general cleanup techniques are referenced or provided as part of the method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

4.7 Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

4.7.1 Common flexible plastics contain varying amounts of phthalate esters which are easily extracted or leached from such materials during laboratory operations.

4.7.2 Cross-contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled.

4.7.3 Interferences from phthalate esters are minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination.

4.8 The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination is often seen in sediment and some soil samples.

4.9 Other halogenated pesticides or industrial chemicals may interfere with the analysis of pesticides. Coeluting chlorophenols are eliminated by using Method 3620B (Florisil).

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. The following analytes covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: 4,4'-DDT, 4,4'-

DDD, and BHCs. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in either two 500 ml amber glass jars or two 1L amber glass jars with teflon-lined lids. Solid samples are collected in one 250 mL wide-mouth glass jar with a teflon-lined lid. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ± 2^{o} C. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ± 2^{o} C.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Aqueous samples must be extracted within 7 days of sample collection, solid samples within 14 days of collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph, Hewlett Packard 6890 (or equivalent): An analytical system completes with gas chromatograph configured for split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system.

7.1.1 Data System: A computer system is interfaced to the GC for collection of data. Data acquisition is performed using Agilent Technologies GC ChemStation (Rev. A.10.02 [1757]).

7.2 GC Columns: Pace utilizes dual-column analyses. The dual-column approach involves either a single injection that is split between two columns that are mounted in a single gas chromatograph, or dual injections of the split extract on a single GC equipped with two

columns. Typical column pairs used are listed below. Other columns may be used as long as method performance criteria can be met.

7.2.1 Column pair 1

30m x 0.32mm ID fused silica capillary column (RTX-clpesticide), 0.32µm film thickness.

30m x 0.32mm ID fused silica capillary column (RTX-clpesticideII) 0.25µm film thickness.

7.2.2 Column pair 2

30m x 0.32mm ID fused silica capillary column (STX-clpesticide), 0.32µm film thickness.

30m x 0.32mm ID fused silica capillary column (STX-clpesticideII) 0.25µm film thickness.

7.3 Guard Column: Cat. #10027 from Restek or equivalent: 5m, 0.32mm

7.4 Gooseneck splitless injection liner: Cat. #23303 from Restek or equivalent

7.5 Universal "Y" Press-tight tee split: Cat. #20406 from Restek or equivalent **/ Siltek MXT Connector:**

7.6 Class "A" Volumetric Flasks: 10mL, 25mL, 50mL, 100mL, 200mL, and 2000mL, for the preparation of standards.

7.7 Microsyringes/Wiretrol syringes: 10 µL – 1000 µL

7.8 Disposable Borosilicate Pipets

7.9 Vials: 2 mL clear glass, crimp-top and screw-cap.

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4 ± 2°C in Teflon(R)-sealed containers in the dark. When a lot of standards is prepared, aliquots of that lot are stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC tests indicate a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

8.1 n-Hexane: Pesticide quality or equivalent.

8.2 Acetone: Pesticide quality or equivalent.

8.3 Methylene chloride: Pesticide quality or equivalent.

8.4 Organic-free Reagent Water: All references to water in this method refer to organic-free reagent water from Alpha's RO water treatment system.

8.5 Stock Standard Solutions: All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is opened, it is transferred to a labeled amber screw-cap vial. The expiration date of the stock solution is either the vendor specified expiration date, or 6 months from the date the ampule was opened, whichever is sooner. Typical stock standard concentrations are listed in Table 2.

8.6 Calibration Standards: Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the calibration standard. Calibrations are typically performed at 11 concentration levels for individual pesticides, Chlordane and Toxaphene; concentrations for all levels are listed in Table 2. Below are suggested procedures for preparation of individual pesticides (Sec. 8.6.1), Chlordane (Sec. 8.6.2), and Toxaphene (Sec. 8.6.3) initial calibration standards.

8.6.1 Preparation of individual pesticides initial calibration standards;

(syringes needed: *100µL [A], 500µL [B], 1000µL [C]*)
(solvent: *Hexane)*
(glassware: *9x10mL, 2x25mL, 1x50mL, 2x100mL, volumetric flasks*)

ICAL (Initial Calibration) STOCK #1: *2000µg/L; 25mL*
50µL[A] of AccuStandard M-8081-SC (Pesticides mix; 1000µg/mL)
250µL[B] of AccuStandard CLP-032-R (TCMX, Deca; 200µg/mL)
500µL[B] of AccuStandard P-102S (Alachlor; 100µg/mL)
500µL[B] of AccuStandard APP-9-112 (Hexachlorobenzene; 100µg/mL)

ICAL STOCK #2 (=LEVEL 10)**:** *200µg/L; 25mL.*
2500µL[B] of STOCK #1
…..

LEVEL 1, 0.5 µg/L:	25µL[A] of ICAL STOCK #2, 10mL
LEVEL 2, 1.0 µg/L:	50µL[A] of ICAL STOCK #2, 10mL
LEVEL 3, 2.0 µg/L:	100µL[A] of ICAL STOCK #2, 10mL
LEVEL 4, 3.0 µg/L:	150µL[B] of ICAL STOCK #2, 10mL
LEVEL 5, 4.0 µg/L:	200µL[B] of ICAL STOCK #2, 10mL
LEVEL 6, 5.0 µg/L:	250µL[B] of ICAL STOCK #2, 10mL
LEVEL 7, 10 µg/L:	500µL[B] of ICAL STOCK #2, 10mL
LEVEL 8, 50 µg/L:	2500µL[C] of ICAL STOCK #2, 10mL
LEVEL 9, 100 µg/L:	5000µL[C] of ICAL STOCK #2, 10mL
LEVEL 10, 200 µg/L:	2500µL[C] of ICAL STOCK #1, 25mL
LEVEL 11, 400 µg/L:	2000µL[C] of ICAL STOCK #1, 10mL

ICV (Initial Calibration Verification) STOCK #1: *2000µg/L; 100mL*
1000µL[C] of Restek 579104 (Custom Pesticides Mix; 200µg/mL)

ICV STOCK #2: *1000µg/L; 50mL*
250µL[B] of Agilent ISM-320-1 (TCMX, Deca; 200µg/mL)
…..

ICV, 50 µg/L:	2500µL[C] of ICV STOCK #1 with 5000µL[C] of ICV STOCK #2, 100mL

8.6.2 Preparation of Chlordane initial calibration standards:

(syringes needed: *100µL [A], 500µL [B], 1000µL [C]*)
(solvent: *Hexane)*
(glassware: *11x10mL, 1x25mL, 1x50mL, volumetric flasks*)

ICAL STOCK #1: *50µg/mL (Chlordane);10mL*
100µL[A] of Agilent EPA-1086-1 (Chlordane Standard; 5000µg/mL)

ICAL STOCK #2 (=LEVEL 9)**:** *2000µg/L; 25mL.*
1000µL[C] of STOCK #1

LEVEL 1, 2µg/L:	10µL[A] of ICAL STOCK #2, 10mL
LEVEL 2, 5µg/L:	25µL[A] of ICAL STOCK #2, 10mL
LEVEL 3, 10µg/L:	50µL[A] of ICAL STOCK #2, 10mL
LEVEL 4, 20µg/L:	100µL[A] of ICAL STOCK #2, 10mL
LEVEL 5, 100µg/L:	500µL[B] of ICAL STOCK #2, 10mL
LEVEL 6, 250µg/L:	1250µL[B] of ICAL STOCK #2, 10mL
LEVEL 7, 500µg/L:	2500µL[C] of ICAL STOCK #2, 10mL
LEVEL 8, 1000µg/L:	5000µL[C] of ICAL STOCK #2, 10mL
LEVEL 9, 2000µg/L:	1000µL[C] of ICAL STOCK #1, 25mL
LEVEL 10, 5000µg/L:	1000µL[C] of ICAL STOCK #1, 10mL

ICV STOCK: *50µg/mL; 10mL*
500µL[B] of Restek 32021 (Chlordane Standard; 1000µg/mL)
…..
ICV, 1000µg/L: 1000µL[C] of ICV STOCK, 50mL

8.6.3 Preparation of Toxaphene initial calibration standards;

(syringes needed: *100µL [A], 500µL [B], 1000µL [C]*)
(solvent: *Hexane)*
(glassware: *11x10mL, 1x50mL, volumetric flasks*)

ICAL STOCK #1: *100µg/mL (Toxaphene);10mL*
1000µL[C] of Absolute Standards 20021 (Toxaphene; 1000µg/mL)

ICAL STOCK #2 (=LEVEL 8)**:** *2000µg/L; 10mL.*
200µL[A] of STOCK #1

LEVEL 1, 5 µg/L:	25µL[A] of ICAL STOCK #2, 10mL
LEVEL 2, 10 µg/L:	50µL[A] of ICAL STOCK #2, 10mL
LEVEL 3, 20 µg/L:	100µL[A] of ICAL STOCK #2, 10mL
LEVEL 4, 100 µg/L:	500µL[B] of ICAL STOCK #2, 10mL
LEVEL 5, 200 µg/L:	1000µL[C] of ICAL STOCK #2, 10mL
LEVEL 6, 500 µg/L:	50µL[A] of ICAL STOCK #1, 10mL
LEVEL 7, 1000 µg/L:	100µL[A] of ICAL STOCK #1, 10mL
LEVEL 8, 2000 µg/L:	200µL[B] of ICAL STOCK #1, 10mL
LEVEL 9, 5000 µg/L:	500µL[B] of ICAL STOCK #1, 10mL
LEVEL 10, 10000 µg/L:	1000µL[C] of ICAL STOCK #1, 10mL

ICV: *2000µg/L; 50mL*
1000µL[C] of Agilent PP-271-1 (Toxaphene Standard; 100µg/mL)

8.7 Internal Standard Solution: 1-Bromo-2-nitrobenzene is used as the internal standard and is added to all single-component calibration standards and sample extracts to achieve a concentration of 0.25 µg/mL. Standard solution expires 6 months after the date of preparation. Below are suggested procedures for preparation of PCB Internal Standard which is diluted to prepare Pesticides Internal Standard.

8.7.1 Preparation of Internal Standard solution:

(syringe needed: *1000µL [A]*)
(solvent: *Hexane)*
(glassware: *2x200mL, volumetric flasks*)

PCB INTERNAL STANDARD: *25µg/mL; 200mL*
1000µL[A] of Agilent PPS-351-1 (1-Bromo-2-nitrobenzene Standard; 5000µg/mL)

PESTICIDES INTERNAL STANDARD: *2.5µg/mL; 200mL*
20mL[A] of PCB INTERNAL STANDARD

8.8 PEM/DEG Solution: is prepared volumetrically by diluting stock standard in Hexane. Solution expires 6 months from the date of preparation, or on the earliest expiration date of any of the stock solution used to prepare the standard.

- 5mL of Pesticides Deg. Check Solution, Ultra. #ISM-450-1 into 100mL of Hexane

8.9 Surrogate Standards: Tetrachloro-m-xylene and decachlorobiphenyl are used as surrogates. They are added to the calibration standards at the concentrations listed in Table 2, and are spiked into all samples and QC samples prior to extraction. The spiking solution is prepared in acetone at the concentrations listed in Table 2. Standard expires 6 months after the date of preparation. Below are suggested procedures for preparation of surrogate standard.

8.9.1 Preparation of surrogate standard:

(syringe needed: *1000µL [A]*)
(solvent: *Acetone)*
(glassware: *1x2000mL, volumetric flasks*)

- **EXTRACTION SURROGATES:** *2000µg/L; 2000mL*
  20mL[A] of AccuStandard CLP-032-R (Deca. & TCMX; 0.2mg/mL)
- **ICV Surrogate Stock:** is prepared by diluting of 250µL of Pesticides Surrogate Standard (Ultra, Cat. #CS-1947) to 50mL of Hexane to achieve concentration of TCMX and DCB at 1.0µg/mL.

8.10 LCS/MS Spiking Solutions: The LCS/MS spiking solutions are prepared volumetrically by diluting the appropriate stock standards in acetone. The spiking solution concentrations are listed in Table 2. Spiking solution expires 6 months after the date of

preparation. Below are suggested procedures for preparation of individual pesticides, Chlordane, and Toxaphene spiking solutions.

8.10.1 Preparation of spiking solutions:

(syringes needed: *250μL [A], 1000μL [B]*)
(solvent: *Acetone)*
(glassware: *1x25mL, 1x50mL, 1x200mL, volumetric flasks*)

INDIVIDUAL PESTICIDES SPIKE: *2000μg/L; 200mL*
2000μL[B] of Restek 579104 (Custom Pesticides Mix; 200μg/mL)

CHLORDANE SPIKE: *25μg/mL; 50mL*
250μL[A] of Agilent EPA-1086-1 (Chlordane Standard; 5000μg/mL)

TOXAPHENE SPIKE: *25μg/mL; 25mL*
625μL[A] of Absolute Standards 20021 (Toxaphene; 1000μg/mL)

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Extraction blanks are performed with each extraction batch of 20 or less samples, according to the extraction SOPs. The extraction blank must not contain any of the reportable analytes above the reporting limit. If any reportable analytes are detected in the blank, the entire extraction batch is suspect and re-extraction of all associated samples is required. Actions associated with blank failures are listed in Table 5.

9.2 Laboratory Control Sample (LCS)

A Laboratory Control Sample (LCS) is extracted with each analytical batch. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with single component pesticide analytes. The concentration of the spiking solution is listed in Table 2. The recovery acceptance criteria are listed in Table 3. If any recovery criteria are not met, the extract should be reanalyzed. If the criteria are still not met, the entire batch should be re-extracted. If this is not possible, due to insufficient sample or holding time exceedance, the analyst must write up the failure on a narrative sheet for inclusion in the client report. Actions associated with LCS/LCSD failures are listed in Table 5.

9.2.1 LCS Duplicate (LCSD)

A Laboratory Control Sample Duplicate is extracted with each analytical batch. The LCSD consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCSD is spiked with the single component pesticide analytes. The concentration of the spiking solution is listed in Table 2. The recovery acceptance criteria are listed in Table 3. If Recovery limits are outside acceptance criteria, the non-conformance is narrated.

9.2.2 Connecticut RCP/MCP/NJ LCS Requirements

For CT- Reasonable Confidence Protocol (RCP) 8081B samples, Massachusetts Contingency Plan (MCP) samples, and samples with a state of origin of New Jersey, the LCS recovery criteria is 40 – 140%. MCP allows for up to 10% of reported spike compounds to fail in the LCS/LCSD and still report.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4

9.5 Matrix Spike

A matrix spike (MS)/matrix spike duplicate (MSD) is extracted and analyzed upon Client request for each batch of 20 or less samples. MS and MSDs are field samples spiked with a known quantity of the target analyte(s). They are prepared by taking additional sample aliquots of the same weight or volume and adding the appropriate amounts of surrogate and spiking solutions. The spike compounds and levels are listed in Table 2. The recovery acceptance criteria are 30-150%. If the recovery criteria are not met, but are met in the LCS, the failure may be attributed to sample matrix effects. Actions associated with MS/MSD failures are listed in Table 5.

9.6 Laboratory Duplicate (DUP)

A duplicate sample is extracted and analyzed upon Client request for each batch of 20 or less samples. Duplicates are laboratory selected replicate samples, prepared by taking an additional sample aliquot of the same weight or volume of a sample. The % RPD criteria are listed in Table 3. If the %RPD is not met, the failure may be attributed to sample matrix effects. Actions associated with duplicate failures are listed in Table 5.

For samples with a state of origin of New Jersey, a MS and MSD/DUP must be extracted for every twenty samples within a 24hr period.

9.7 Surrogates:

All extracted samples and associated QC are spiked with surrogates at the levels listed in Table 2. The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate control limits listed in Table 3. If the surrogate limits are not met, the extract should be reanalyzed to determine if the failure was due to an instrument problem. If the criteria are still not met, the affected samples should be re-extracted to confirm that the failure was due to sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

9.8 Method Sequence

<u>Typical Initial Calibration</u> (each level to identify the standard lot number)

1. Instrument Blank (may be omitted to conserve tray space)
2. Degradation check standard
3. Std Level 1

4. Std Level 2
5. Std Level 3
6. Std Level 4
7. Std Level 5
8. Std Level 6
9. Std Level 7
10. Std Level 8
11. Std Level 9
12. Std Level 10
13. Std Level 11 (Individual Pesticides only)
14. Initial Calibration Verification Standard (ICV)
15. Repeat std Levels 1 –10 for other mixes until curve is complete.

    [**NOTE:** If multiple calibration mixtures are analyzed, it is acceptable to analyze appropriate ICVs after all calibration standards have been injected.]

Typical Daily sequence (each standard must be identified with lot number)

1. Degradation Check Standard
2. Pesticide Continuing Calibration Standard
3. Chlordane Continuing Calibration Standard
4. Toxaphene Continuing Calibration Standard
5. Extraction Blank
6. Laboratory Control Sample
7. Laboratory Control Sample Duplicate
8. Matrix Spike (Upon Client Request)
9. Duplicate/Matrix Spike Duplicate (Upon Client Request)
10. Samples (up to **20 field samples may be analyzed**)
11. Cal check
12. Repeat 1 – 10 (as needed)

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Extraction

Water samples are extracted at a neutral pH with methylene chloride using a separatory funnel (Method 3510C). See extraction SOP for details.

Solid samples are extracted with methylene chloride: acetone (1:1) using Soxhlet extraction (Method 3540C) or microwave extraction (Method 3546). See extraction SOP for details.

10.1.2 Extract Cleanup

Cleanup procedures may not be necessary for a relatively clean sample matrix, but most extracts from environmental and waste samples will require additional preparation before analysis. The specific cleanup procedure used will depend on the nature of the sample to be analyzed and the data quality objectives for the measurements. See extraction SOPs for details.

10.1.3 GC Conditions:

The dual-column / dual-detector approach involves the use of the columns listed in section 7.2. The columns are connected to an injection tee or dual injection GC, and separate electron capture detectors. Typical GC conditions for the HP 6890 instruments are listed below but may be altered as long as method performance criteria are met.

Temperature 1:	120 °C
Time 1:	0 minute
Ramp 1:	45 °C/minute
Temperature 2	200 °C
Time 2:	0 minutes
Ramp 2:	15 ° C/minute
Temperature 3:	230 °C
Time 3:	0 minutes
Injector temperature:	250 °C
Ramp 3:	30 °C/minute
Final Temperature:	330 °C
Final Time:	2.0 minutes

Injector mode:	Pulsed Split
	4:1 split
Injector Flow:	20 mL/min split flow
Detector temperature:	350 °C
Carrier gas:	Hydrogen
Carrier flow:	28 mL/min
Carrier mode:	Constant flow
Makeup gas:	Nitrogen
Total detector flow:	60 mL/min
Injection volume:	1 µL

10.1.4 DDT and Endrin Breakdown

The breakdown of DDT and Endrin must be measured before samples are analyzed and at the beginning of each 12-hour shift. Injector maintenance must be completed if the breakdown is greater than 15% for either compound (See Section 10.5.1). Both analytical columns must pass DDT/Endrin breakdown criteria prior to sample analysis.

10.2 Initial Calibration

10.2.1 Prepare calibration standards using the procedures in Section 8.6 and the concentrations listed in Table 2. The calibration standards are aliquoted into autosampler vials and capped prior to loading onto the autosampler tray.

10.2.2 Single point calibration might be employed for analysis of the multi-component analytes (Chlordane, Toxaphene). A single calibration standard near the mid-point of the expected

calibration range of each multi-component analyte is included with the initial calibration of the single component analytes for pattern recognition, so that the analyst is familiar with the patterns and retention times on each column. The calibration standard may be at a lower concentration than the mid-point of the expected range, if appropriate for the project.

10.2.3 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in section 10.1.3. The same operating conditions are used for calibrations and sample analyses. Create the analytical sequence using the Agilent Chemstation data acquisition software.

10.2.3.1 Record the calibration standard, unique lab identifier code (lot), the analytical sequence list.

10.2.4 A 1µL injection volume of each calibration standard is used. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume must be used for all standards and samples.

10.2.5 Because of the low concentration of pesticide standards injected on a GC/ECD, column adsorption may be a problem when the GC has not been used for a day or more or after system maintenance. The GC column may be primed (or deactivated) by injecting a pesticide standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

Alternately, the system may be primed by baking at the final analytical temperature for approximately 30 minutes.

Several analytes may be observed in the injection just following system priming. Always run an instrument blank after system priming.

10.2.6 Calibration Factors

10.2.6.1 Internal Standard Procedure

Internal standard calibration techniques are employed in this method. In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formulae listed in Pace's Quality Manual.

10.2.7 Initial Calibration Criteria

- If the **RSD for an analyte is < 20%,** then the response of the instrument for this compound is considered linear over the range and the mean calibration factor can be used to quantitate sample results.

- If the **RSD for any analyte is > 20%,** then linearity through the origin cannot be assumed. The mean response factor cannot be used for quantitation. An

alternative calculation may be done using **linear regression or quadratic regression** as long as the correlation coefficient is >0.990. If both quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated, and a new calibration curve must be analyzed. If quadratic regression is used for calibration, this must be noted in the laboratory narrative (MCP/RCP-CT only).

- **Percent Relative Error (%RE):** For linear and quadratic fit compounds, the %RE must be calculated for the standard at or near the midpoint and at the lowest level of the initial calibration. The %RE between the calculated and expected concentration of an analyte must be ≤ 30%, otherwise recalibration is necessary.

- **Chlordane/Toxaphene peaks requirements**:

Analyte	Number of Peaks
Chlordane	3-5
Toxaphene	4-6

- **The minimum number of calibration standards based on the type of calibration curve is listed below**:

Type of Calibration Curve	Minimum Number of Calibration Standards
Linear Fit/Avg Response	5
Quadratic Fit	6

- **Removal and replacement of the calibration standards:**

    - The Analyst may remove individual analyte from the lowest (cannot remove RL level) and or the highest calibration levels of the curve. Removal of the individual analyte of interior levels is not permitted.

    - The Analyst may remove an entire single standard calibration level from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. The Analyst who chooses to remove a calibration standard from the interior of the calibration shall remove that particular standard calibration level for all analytes.

    - The Analyst may replace a calibration standard provided that:

        - the replacement standard will be analyzed within **twenty-four (24) hours** of the original calibration standard analysis for that particular calibration level;

- all analytes of the replacement calibration standard must be replaced if a standard within the interior of the calibration is replaced;

- the laboratory limits the replacement of calibration standards to one calibration standard concentration.

10.2.8 Retention Time Windows

10.2.8.1 The retention time windows used for the identification of target analytes are calculated using the procedure recommended in Method 8000 and were found to be ± 0.015 minutes.

10.2.8.2 The windows listed above are used as guidance; however, the experience of the analyst weighs heavily in the interpretation of the chromatograms. For example, it has been observed that certain oil matrices can cause the retention times to shift more dramatically. Additionally, if any positive results are questionable and at sufficiently high concentration, GC/MS analysis is used for confirmation.

10.2.9 Initial Calibration Verification

An **Initial Calibration Verification** standard must be run immediately after each initial calibration, near the midpoint of the curve. This standard must be prepared using a second source that is different than the source used for the initial calibration. The **%D** for each analyte to be quantitated must not exceed a **± 20%** difference when compared to the initial calibration curve.

If recovery is outside the 80-120% criteria, the following steps should be taken:

- Analyze the replacement standard. The cause of the failure of the first calibration verification result should be documented.
- If the reanalysis still didn't achieve the criteria, the source of the problem must be investigated, and the instrument must be recalibrated.

10.3 Equipment Operation and Sample Processing

10.3.1 The same GC operating conditions used for the initial calibration must be employed for sample analyses, including sample injection volume (Section 10.1.3).

10.3.2 Tentative identification of an analyte occurs when a peak from a sample extract falls within the retention time window for the compound. Each tentative identification is confirmed using a second GC column of dissimilar stationary phase. Confirmation is positive when the %RPD is $\leq$ 40% from the results of the two columns. In particularly difficult matrices, confirmation by GC/MS may be advisable (see Section 10.3.11).

10.3.3 The concentration reported for an identified target analyte in an extract is calculated using the Enviroquant data processing software. The Enviroquant methods have been configured to utilize the quantitation formulas found in Quality Manual. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined. See the Manual Integration SOP for integration guidelines.

10.3.3.1 If the responses exceed the calibration range of the system, dilute the extract and reanalyze.

10.3.4 Each sample analysis must begin with an acceptable initial calibration, breakdown standard (PEM) calibration verification standard(s) (each **12-hour analytical shift**), or calibration standards interspersed within the samples.

The **Internal Standard area of the samples must be within 50%-200% of the starting CCV.** When a calibration verification standard fails to meet the QC criteria, all samples that were injected after the last standard that last met the QC criteria must be re-injected.

10.3.5 Sample injections may continue as long as the calibration verification standards and standards interspersed with the samples meet instrument QC requirements. The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria are exceeded.

10.3.6 Use the calibration standards analyzed during the sequence to evaluate retention time stability. The retention time windows are established using the absolute retention time of each analyte in the mid-concentration standard during the initial calibration as the mid-point of the window. The widths of the windows are defined in Section 10.2.7.

10.3.7 Each subsequent injection of a standard during the 12-hour analytical shift (i.e., those standards injected every 20 field samples, or more frequently) must be checked against the retention time windows. If any of these subsequent standards fall outside their absolute retention time windows, the GC system is out of control. Determine the cause of the problem and correct it. If the problem cannot be corrected, a new initial calibration must be performed.

10.3.8 Identification of mixtures (i.e. Chlordane and Toxaphene) is based on the characteristic 'fingerprint' retention time and shape of the indicator peak(s); and quantitation is based on the area under the characteristic peaks as compared to the area under the corresponding calibration peaks(s) of the same retention time and shape generated using internal calibration procedures.

10.3.9 If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be needed. If instrument problems are suspected, rerun the extract on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to the extraction SOPs for the procedures to be followed in sample cleanup.

10.3.10 For secondary column analysis, a second dissimilar column is utilized to confirm positive pesticide results. The laboratory must report the **higher of the two results** unless obvious interference is present on one of the columns. All required QA/QC parameters (e.g. calibrations, LCSs, etc.) must be met on the second column as well.

10.3.10.1 The **Relative Percent Difference (RPD)** should be 40 or lower. If the RPD exceeds 40, this will be denoted with a P flag. If the RPD exceedance is due to interference, the lower of the dual column values can be reported with I and P flags.

10.3.11 GC/MS Confirmation

GC/MS confirmation may be used in conjunction with either single-column or dual-column analysis if the concentration is sufficient for detection by GC/MS.

10.3.11.1 Full-scan GC/MS will normally require a concentration of approximately 10ng/µL in the final extract for each single-component compound.

10.3.11.2 The GC/MS must be calibrated for the specific target pesticides when it is used for quantitative analysis.

10.3.11.3 GC/MS may not be used for confirmation when concentrations are below the sensitivity of the instrument.

10.3.11.4 GC/MS confirmation should be accomplished by analyzing the same extract that is used for GC/ECD analysis.

10.3.11.5 The base/neutral/acid extract and the associated blank may be used for GC/MS confirmation if the surrogates and internal standards do not interfere and if it is demonstrated that the analyte is stable during acid/base partitioning. However, if the compounds are not detected in the base/neutral/acid extract, then GC/MS analysis of the pesticide extract should be performed.

10.3.11.6 A QC reference sample containing the compound should also be analyzed by GC/MS. The concentration of the QC reference sample must demonstrate that those pesticides identified by GC/ECD can be confirmed by GC/MS.

10.4 Continuing Calibration Verification

10.4.1 Verify calibration each 12-hour shift by injecting calibration verification standards (CCV) prior to conducting any sample analyses. A calibration standard must be injected at intervals of not less than once every twenty field samples.

10.4.1.1 The calibration factor (for external standard compounds) and response factor (for internal standard compounds) for each analyte to be quantitated must not exceed a **± 20%** difference when compared to the initial calibration curve. The Enviroquant data processing software automatically calculates the %D for all analytes according to the formulae in Quality Manual. **The Internal Standard area of the CCV must be within 50%-150%** of the Initial Calibration Internal Standard area.

10.4.1.2 If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.4.1.3 If routine maintenance does not return the instrument performance to meet the QC requirements (Section 10) based on the last initial calibration, then a new initial calibration must be performed. Due to the large number of analytes present, allowances may be made for a CF or RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples. Any QC failures must be written up by the analyst on narrative sheets for inclusion with the sample data.

10.4.2 Compare the retention time of each analyte in the calibration standard with the absolute retention time windows described in section 10.2.7. The center of the absolute retention time window for each analyte is its retention time in the mid-concentration standard analyzed during the initial calibration. Each analyte in each standard must fall within its respective retention time window. If not, the gas chromatographic system must either be adjusted so that a second analysis of the standard does result in all analytes falling

within their retention time windows, or a new initial calibration must be performed and new retention time windows established.

10.5 Internal Standard

The use of internal standard calibration does not require that all sample results be bracketed with CCV standard. However, when internal standard calibration is used, the retention times of internal standards and the area response of internal standards should be checked for each analysis.

10.5.1 **IS in CCAL** – The measured area of the internal standard must be no more than ± 50% different from the average area calculated during initial calibration (-50 to 150%).

10.5.2 **IS in samples** - The measured area of the internal standard must be no more than -50% to +100% different from the area calculated from opening CCV (-50 to 200%)

Retention time shifts of more than 30 seconds from the retention time of the most recent calibration standard are cause for concern and must be investigated.

10.6 Preventive Maintenance

Routine preventive maintenance should be performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and replacement of injector seals.

10.6.1 Other Maintenance

Additional maintenance may be required if system performance degrades.

10.6.1.1.1 GC injector ports are of critical concern, especially in the analysis of DDT and Endrin.

10.6.1.1.1.1 Injectors that are contaminated or chemically active can cause the degradation (“breakdown”) of the analytes. Endrin and DDT breakdown to Endrin aldehyde, Endrin ketone, DDD, or DDE.

Check for degradation problems by injecting a standard containing only 4,4’-DDT and Endrin. The presence of 4,4’-DDE, 4,4’-DDD, Endrin ketone or Endrin indicates breakdown. If degradation of either DDT or Endrin exceeds 15%, take corrective action before proceeding with calibration.

When such a breakdown is observed, replacement of the injector liner and seal may solve the problem. If not, clip approximately 3 – 6 inches from the injector end of the GC column. If the degradation does not improve, it may be necessary to replace the column(s).

Calculate percent breakdown as follows:

$$\%\ breakdown\ of\ DDT = \frac{sum\ of\ degradation\ peak\ areas\ (DDE + DDD)}{sum\ of\ all\ peak\ areas\ (DDT + DDE + DDD)} * 100$$

$$\%\ breakdown\ of\ Endrin = \frac{sum\ of\ degradation\ peak\ areas\ (Aldehyde + Ketone)}{sum\ of\ all\ peak\ areas\ (Endrin + Aldehyde + Ketone)} * 100$$

10.6.1.1.2 ECD detectors may also become contaminated, requiring bake out at elevated temperatures, or repair by the manufacturer.

11. Data Evaluation, Calculations and Reporting

11.1 Quantitation of Single Component Pesticides

The single component pesticide compounds are calculated as described in Section 10.3.3 and reported in µg/L or µg/Kg units. After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews should be done by two separate individuals.

11.1.1 Quantitation of Multiple-Component Analytes

Quantitation is based on use of 3-5 of the major peaks present for chlordane and 4-6 of the major peaks present for Toxaphene. Each of these peaks is individually calibrated with a 10-point calibration based on average response factors. The %RSD must meet the criteria of ≤20%. The 3 to 5 or 4 – 6 major peaks are calculated as described in Section 10.3.3. After individual calculation meets criteria, the average of the major 3-5 or 4 – 6 peaks is used to determine the final concentration.

11.1.1.1 Toxaphene

Toxaphene is quantitated by the internal standard method, using the 4 – 6 largest peaks found in the standard and averaging the resulting concentrations.

11.1.1.2 Chlordane

Chlordane is a mixture of at least 11 major components and 30 or more minor components. Trans- and cis-Chlordane (alpha and gamma, respectively), are the two major components of Chlordane. However, the exact percentage of each in the chlordane material is not completely defined and is not consistent from batch to batch.

11.1.1.2.1 The GC pattern of a Chlordane residue may differ considerably from that of the chlordane standard. Depending on the sample substrate and its history, residues of Chlordane can consist of almost any combination of constituents from the Chlordane, plant and/or animal metabolites, and products of degradation caused by exposure to environmental factors such as water and sunlight.

11.1.1.2.2 Whenever possible, when Chlordane residue does not resemble Chlordane, the analyst should quantitate the peaks of alpha-Chlordane, gamma-

Chlordane, Heptachlor, and trans-Nonachlor separately against the appropriate reference materials, and report the individual residues.

11.1.1.2.3 When the GC pattern of the residue resembles that of Chlordane, the analyst may quantitate Chlordane residues by comparing the total area of the Chlordane chromatogram using the 3-5 major peaks.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and/or improper preservation are noted on the nonconformance report form.

All non-conformances, sample preparatory extraction methods and clean-up methods must be addressed in a case narrative and included in the data report.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Qualtrax ID 1732. These studies performed by the laboratory are maintained on file for review

13.2 Demonstration of Capability Studies

Refer to Qualtrax ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

Qualtrax ID 1732 MDL/LOQ Generation

Qualtrax ID1739 IDC/DOC Generation

Qualtrax ID 1728 Waste Management and Disposal SOP

16. Attachments

Table 1: REPORTING LIMITS

Table 2: STANDARD SOLUTIONS

Table 3: QC ACCEPTANCE CRITERIA

Table 4: MCP/CT/NJ SPECIFIC ACCEPTANCE CRITERIA

Table 5: QC FAILURE ACTION PLAN

TABLE 1

REPORTING LIMITS*

	RL (Aqueous)	RL (Soil)
Pesticides		
Alpha-BHC	0.02 µg/L	3.33 µg/Kg
Gamma-BHC (Lindane)	0.02 µg/L	2.67 µg/Kg
Heptachlor	0.02 µg/L	4 µg/Kg
Endosulfan I	0.02 µg/L	10 µg/Kg
Dieldrin	0.04 µg/L	5 µg/Kg
Endrin	0.04 µg/L	3.33 µg/Kg
4, 4'-DDD	0.04 µg/L	10 µg/Kg
4, 4'-DDT	0.04 µg/L	15 µg/Kg
Methoxychlor	0.2 µg/L	15 µg/Kg
Aldrin	0.02 µg/L	10 µg/Kg
Beta-BHC	0.02 µg/L	10 µg/Kg
Delta-BHC	0.02 µg/L	10 µg/Kg
Heptachlor Epoxide	0.02 µg/L	15 µg/Kg
trans-Chlordane	0.02 µg/L	10 µg/Kg
cis-Chlordane	0.02 µg/L	10 µg/Kg
4, 4'- DDE	0.04 µg/L	15 µg/Kg
Endosulfan II	0.04 µg/L	15 µg/Kg
Endrin Aldehyde	0.04 µg/L	10 µg/Kg
Endosulfan Sulfate	0.04 µg/L	3.33 µg/Kg
Endrin Ketone	0.04 µg/L	10 µg/Kg
Chlordane	0.2 µg/L	65 µg/Kg
Toxaphene	0.2 µg/L	150 µg/Kg
Alachlor +	0.1 µg/L	10 µg/Kg
Hexachlorobenzene ++	0.02ug/L	8 µg/Kg

*** RL:** Typical laboratory reporting limit. Actual RLs may be higher depending on sample matrix. RLs are not adjusted for % Moisture.

+ CT-RCP only compound.
++ MCP/RCP-CT only compound.

TABLE 2

STANDARD SOLUTIONS

	Stock 1 solution (µg/mL)	Stock 2 solution (µg/mL)	Level 11 (µg/L)	Level 10 (µg/L)	Level 9 (µg/L)	Level 8 (µg/L)	Level 7 (µg/L)	Level 6 (µg/L)	Level 5 (µg/L)	Level 4 (µg/L)	Level 3 (µg/L)	Level 2 (µg/L)	Level 1 (µg/L)	Std. Spike Solution (µg/mL)	Std. LCS Solution (µg/mL)
Hexachlorobenzene	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Alpha-BHC	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Lindane	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Heptachlor	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endosulfan I	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Dieldrin	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endrin	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
4, 4'-DDD	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
4, 4'-DDT	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Methoxychlor	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Tetrachloro-m-Xylene *	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Decachloro-Biphenyl *	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Aldrin	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Beta-BHC	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Delta-BHC	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Heptachlor Epoxide	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Trans-Chlordane	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Cis-Chlordane	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
4, 4'- DDE	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endosulfan II	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endrin Aldehyde	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endosulfan Sulfate	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Endrin Ketone	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Alachlor	2	0.1	400	200	100	50	10	5	4	3	2	1	0.5	2.0	2.0
Chlordane	50000	1000	10000	5000	2000	1000	500	250	100	20	10	5	2	25	25
Toxaphene	100000	2000	20000	10000	5000	2000	1000	500	200	100	20	10	5	25	25
Internal Standard															
1-Bromo-2-Nitrobenzene	5000	5000	25	25	25	25	25	25	25	25	25	25	25		

* - surrogates

TABLE 3

QC ACCEPTANCE CRITERIA**

	Aqueous		Soil	
Surrogate % Recovery*	**Lower Control Limit**	**Upper Control Limit**	**Lower Control Limit**	**Upper Control Limit**
2,4,5,6-Tetrachloro-m-xylene	30%	150%	30%	150%
Decachlorobiphenyl	30%	150%	30%	150%

*One surrogate on one column is allowed to be outside of the control limits and still be within criteria.

Compound	LCS % Recovery (Aqueous)		LCS % Recovery (Soil)		Duplicate and/or MSD %RPD	
	Lower Control Limit	**Upper Control Limit**	**Target Analyte Limits**	**Marginal Exceedance Limits**	**Aqueous RPD**	**Soil RPD**
Target Analytes LCS/LCSD	30%	150%	30%	150%	≤ 30%	≤ 50%
Target Analytes MS/MSD	30%	150%	30%	150%	≤ 30%	≤ 50%

TABLE 4

MCP/CT/NJ SPECIFIC QC ACCEPTANCE CRITERIA

Compound	LCS % Recovery (Aqueous)		LCS % Recovery (Soil)		Duplicate and/or MSD %RPD	
	Lower Control Limit	**Upper Control Limit**	**Target Analyte Limits**	**Marginal Exceedance Limits**	**Aqueous RPD**	**Soil RPD**
Target Analytes LCS/LCSD	40%	140%	40%	140%	≤ 20%	≤ 30%
Target Analytes MS/MSD	40%	140%	40%	140%	≤ 20%	≤ 30%

TABLE 5

QC FAILURE ACTION PLAN

Blank Failures	Failure Situation	Action(s)
Surrogate Failure	1 of 4 high or low	Report as is
	2 or more high	Report as is
	2 or more low, samples are non-detect with passing surrogates	Report as is
	2 or more low, samples have hits and/or failing surrogates	Revial/rerun Re-extract if still failing
IS Failure	IS not present (less than 10% recovery)	Add IS Rerun
	IS out high or low	Revial/rerun Re-extract if still failing
Contamination	Below RL; sample(s) non-detect, below RL, or concentration 10 times or more above the blank	Report as is
	Below RL, sample(s) concentration above RL	See team leader/manager
	Above RL, sample(s) concentration 10 times or more above the blank	Report as is
	Above RL with non-detect sample(s)	Report as is
	Above RL, sample(s) concentration less than 10 times above the blank and volume available	Revial/rerun Re-extract if contamination still present
	Above RL, sample(s) concentration less than 10 times above the blank and no volume available	Revial/rerun B-flag affected data

LCS/LCSD Failures	Failure Situation	Action(s)
Surrogate Failure	1 or more high or low	Report as is
IS Failure	IS not present (less than 10% recovery)	Add IS Rerun
	IS out high or low	Revial/rerun Re-extract if still failing
Spike Failure	Spike compounds out high and sample(s) are non-detect with passing surrogates	Report as is
	Spike compounds out high and sample(s) have hits	Revial/rerun Re-extract if still high
	Spike compounds out low	Revial/rerun Re-extract if still low
	RPD failure	Report as is

MS/MSD/DUP Failures	Failure Situation	Action(s)
Surrogate Failure	(MS/MSD) 1 or more high or low	Report as is
	(MS/MSD) 2 or more high or low	Report as is
	(DUP) 1 or more high, parent sample non-detect	Report as is
	(DUP) 1 or more low, parent sample matches	Report as is
	(DUP) 1 or more low, parent sample doesn't match	Revial/rerun Re-extract if still failing
IS Failure	IS not present (less than 10% recovery)	Add IS Rerun
	IS out high or low	Revial/rerun Re-extract if still failing
Spike Failure (MS/MSD only)	Compounds out high or low due to sample matrix or hits	Report as is
	Compounds out high or low due to extraction error	Revial/rerun Re-extract if still failing
	RDP failure	Report as is

Oxidation – Reduction Potential (ORP)

References: **ASTM D1498 (Modified),** Annual Book of ASTM Standards, Vol 11.01. September 15, 1993.

1. Scope and Application

Matrices: Water and Soils/Solids

Definitions: Refer to Alpha Analytical Quality Manual.

This practice covers the apparatus and procedure for the electrometric measurement of oxidation-reduction potential (ORP) in water. It does not deal with the manner in which the solutions are prepared, the theoretical interpretation of the oxidation-reduction potential, or the establishment of a standard oxidation-reduction potential for any given system. The practice described has been designed for the routine and process measurement of oxidation-reduction potential.

Oxidation-reduction potential: The electromotive force, Em, developed between a noble metal electrode and a standard reference electrode. This oxidation-reduction potential (ORP) is related to the solution composition by:

$$E_m = E^o + 2.3 \frac{RT}{nF} \log A_{ox}/A_{red}$$

Where:

Em = ORP,
E^o = constant that depends on the choice of reference electrodes.
F = Faraday constant,
R = gas constant,
T = absolute temperature, °C + 273.15,
n = number of electrons involved in process reaction, and
A_{ox} and A_{red} = activities of the reactants in the process.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

This is a practice designed to measure the ORP which is defined as the electromotive force between a noble metal electrode and a reference electrode when immersed in a solution. The practice describes the electronic equipment available to make the measurement and describes how to determine the sensitivity of the electrodes as well as the calibration of equipment to solutions

having a known potential. The ORP electrodes are inert and measure the ratio of the activities of the oxidized to the reduced species in the process reactions.

2.1 Method Modifications from Reference

Modified. One sample is measure in duplicate. Not all samples are read in successive portions until they differ by less than 10 mV.

3. Reporting Limits

Not applicable to this method.

4. Interferences

4.1 The ORP electrodes reliably measured ORP in nearly all aqueous solutions and in general are not subject to solution interference from color, turbidity, colloidal matter, and suspended matter.

4.2 The ORP of an aqueous solution is sensitive to change in temperature of the solution, but temperature correction is rarely done due to its minimal effect and complex reactions. Temperature corrections are usually applied only when it is desired to relate the ORP to the activity of an ion in the solutions.

4.3 The ORP of an aqueous solution is almost always sensitive to pH variations even to reactions that do not appear to involve hydrogen or hydroxyl ions. The ORP generally tends to increase with an increase in hydrogen ions and to decrease with an increase in hydroxyl ions during such reactions.

4.4 Reproducible oxidation-reduction potentials cannot be obtained for chemical systems that are not reversible. Most natural and ground waters do not contain reversible systems, or may contain systems that are shifted by the presence of air. The measurement of end point potential in oxidation-reduction titration is sometimes of this type.

4.5 If the metallic portion of the ORP electrode 1s sponge-like, materials absorbed from solutions may not be washed away, even by repeated rinsings. In such cases, the electrode may exhibit a memory effect, particularly if it is desired to detect a relatively low concentration of a particular species immediately after a measurement has been made in a relatively concentrated solution. A brightly polished metal electrode surface is required for accurate measurements.

4.6 The ORP resulting from interactions among several chemical systems present in mixed solutions may not be assignable to any single chemical.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by

whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in plastic or glass containers.

6.2 Sample Preservation

Samples are not preserved.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Liquid samples are analyzed within 24 hours of collection; soil samples are analyzed within 28 days of collection.

The exception is MCP-Hexachrome package soils require ORP soils to have a 24 hour hold time and NJ-Hexachrome package soils need to be analyzed within 24h of HexCr preparation time

7. Equipment and Supplies

7.1 ORP Meter with Oxidation-Reduction Electrode: Orion 555A PH/ORP/Conductivity meter with ORION sure flow combo REDOX/ORP electrode.

7.2 Glass Beakers or plastic cups

7.3 Stir bars

7.4 Stir plate

7.5 Ag/AgCl Reference Electrode Filling Solution

8. Reagents and Standards

8.1 Purity of Reagents: Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended 'hat all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society., Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 Purity of Water: DI water from Alpha's water system.

8.3 Redox Standard Solution; Ferrous-Ferric Reference Solution: Dissolve 0.745g of potassium chloride (KCL) (EMD), 0.140 g of potassium ferrocyanide (K4(Fe(CN)6)*3H20) and 0.110g of Potassium Ferriccyanide (K3Fe(CN)6) in water and dilute to 100mL. It is necessary to prepare the solution using reagent grade chemicals that have an assay confirming them to be within 1% of the nominal composition. The solution is stored in a closed glass or plastic container. Expiration date is 3 months from the date of preparation.

8.3.1 The ferrous-ferric reference solution is a reasonably stable solution with a measurable oxidation – reduction potential of 230mV.

8.3.2 Zobell Solution: Commercially available Redox standard solution. Same as 8.3 but from a different source. Expires at manufactures date.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Laboratory Control Sample (LCS)

LCS result must be within +/-10% of the true value.

9.2 Initial Calibration Verification (ICV)

ICV result must be within +/-10% of the true value.

9.3 Continuing Calibration Verification (CCV)

CCV result must be within +/-10% of the true value.

9.4 Laboratory Duplicate

% RPD must be within $\leq$ 20%. If %RPD fails, the analyst must check samples for homogeneity and matrix interferences. % RPD failure may be narrated.

9.5 Method Sequence:

- Turn on instrument
- Weigh out soils and spin with DI
- Read ICV
- Read LCS
- Read samples and duplicate
- Read closing CCV

10. Procedure

10.1 Equipment Set-up

10.1.1 Electrode Treatment: Condition and maintain ORP electrodes as recommended by the manufacturer.

10.1.2 ORP Electrode Cleaning: Rinse with DI water.

10.2 Initial Calibration

10.2.1 Before using electronic type meters, turn them on and alloy them *to* warm up thoroughly. Bring them to electrical balance by carefully following the manufacturer's instructions. Set the scale or range to the desired millivolt level expected in the test solution.

10.2.2 Once the instrument is ready. Read the ICV standard, if the standard is within 10% of the true value, proceeds with the analysis.

10.3 Equipment Operation and Sample Processing

10.3.1 Place the sample in a clean glass beaker or sample cup and insert the electrodes. Provide adequate agitation throughout the measurement period. Read the millivolt potential of the solution allowing sufficient time for the system to stabilize.

10.3.2 For Soil samples: Weigh 10g into a cup and add a stir bar. 100mL DI is added and the solution is stirred for 1 hour. After 1 hour, decant out the liquid portion and follow 10.3.1.

10.4 Continuing Calibration

CCV analyzed after every 10 samples and at the end of analytical run.

10.5 Preventive Maintenance

Replace electrode as needed.

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11. Data Evaluation, Calculations and Reporting

11.1 Report the oxidation-reduction potential in millivolts directly from the meter. The electrode system employed must also be reported.

11.2 If it is desired to report the oxidation-reduction potential referred to the hydrogen electrode, calculate as follows:

$$E_h = E_{obs} + E_{ref}$$

Where:

E_h = oxidation-reduction potential referred to the hydrogen scale, mV,

E_{obs} = observed oxidation-reduction potential of the noble metal-reference electrode employed, mV, and

E_{ref} = oxidation-reduction potential of the reference electrode as related to the hydrogen electrode, mV.

11.3 Report the oxidation-reduction potential to the nearest I mV, interpolating the meter scale as required. When considered appropriate, the temperature at which the measurement was made and the pH at the time of measurement, may also be reported.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

If the ICV, CCV or LCS fails, remake the solution and reanalyze.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/ 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/ 1734 and 1739for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732	MDL/LOD/LOQ	Generation
SOP/1734,1739	IDC/DOC	Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

Table 1: National Institute of Standards and Technology (NIST) Materials for Reference Buffer Solutions

Table 2: Potential of the Platinum Electrode for Several Reference Electrodes at 25 °C in Ferrous-Ferric Reference Solution

Table 3: Nominal ORP of Reference Quinhydrone Solutions

TABLE 1 National Institute of Standards and Technology (NIST) Materials for Reference Buffer Solutions

NIST Standard Sample Designation[A]	Buffer Salt	Drying Procedure
186-II-e	disodium hydrogen phosphate	2 h in oven at 130°C
186-I-e	potassium dihydrogen phosphate	2 h in oven at 130°C
185-g	potassium hydrogen phthalate	drying not necessary

[A] The buffer salts listed can be purchased from the Office of Standard Reference Materials, National Institute of Standards and Technology (NIST), Gaithersburg, MD 20899.

TABLE 2 Potential of the Platinum Electrode for Several Reference Electrodes at 25°C in Ferrous-Ferric Reference Solution

Reference Electrode	Potential EMF, mV
Hg, Hg_2 Cl_2, satd KCl	+430
Ag, AgCl, 1.00 M KCl	+439
Ag, AgCl, 4.00 M KCl	+475
Ag, AgCl, satd KCl	+476
Pt, H_2 (p = 1), H (a = 1)	+675

TABLE 3 Nominal ORP of Reference Quinhydrone Solutions

	ORP, mV					
Buffer solution, nominal pH	4			7		
Temperature, °C	20	25	30	20	25	30
Reference Electrode						
Silver/silver chloride	268	263	258	92	86	79
Calomel	223	218	213	47	41	34
Hydrogen	470	462	454	295	285	275

Analysis of Polynuclear Aromatic Hydrocarbons and PCB Congeners by Gas Chromatography/Mass Spectrometry with Selected Ion Monitoring

References: Method EPA 8270E, SW-846,Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision VI (Phase II), June 2018.

Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, Compendium Method TO-13A – Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Ambient Air Using GC/MS-SIM, January 1999.

USEPA, Superfund Analytical Services/Contract Laboratory Program (CLP), Multi-Media, Multi-Concentration Organics Analysis, SOM01.1, Exhibit D - Analytical Methods, "Analytical Method for the Analysis of Semivolatile Organic Compounds," May, 2005.

1. Scope and Application

Matrices: Solid, waste, soil, sediment, tissue, oil, water and Air PUF.

Definitions: Refer to Alpha Analytical Quality Manual.

This standard operating procedure (SOP) is applicable to the quantification of polynuclear aromatic hydrocarbons (PAHs) and PCB Congeners by gas chromatography/mass spectrometry with selected ion monitoring (GC/MS-SIM). Method is modified from USEPA method 8270D for the application of SIM following the SW846 sited CLP SOW reference.

The sample preparation methods described herein are: 3510C (water), 3540 (Air PUF), 3580 (oil) and 3570 (solid/waste, soil, sediment and tissue) from the above reference.

The individual target compounds are found in Table B.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/MS instrumentation and in the interpretation of GC/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Aqueous samples are serially extracted with methylene chloride in a 2 liter separatory funnel at the required pH. Soil, sediment, solid/waste and tissue samples are extracted by micro scale solvent extraction (MSE) 3570 modified. Oil samples are diluted with methylene chloride (1mL sample to 9mL of solvent) following method 3580. Air PUF samples are extracted via soxhlet method 3540 in hexane/ether (90/10). The extracts are concentrated to the required final volume and spiked with internal standards in preparation for analysis. All extraction procedures are documented in the associated sample preparation SOPs. The extracts are analyzed by GC/MS-SIM. The target analytes are resolved on the GC column and detected using a mass selective detector (MSD). Concentrations are determined using mean relative response factors from a multi-level calibration

curve. Response factors for target analytes and surrogate compounds are determined relative to the internal standards.

2.1 Method Modifications from Reference

Section 7.5.5 of EPA SW846 8270C is referred to for the application of SIM, along with EPA SOM01.1, Exhibit D for Semivolatiles. This method has been modified to include a sub list of PCB Congeners that are co-analyzed along with the PAHs. These PCB Congeners are commonly referred to the NOAA 18, or NOAA 22 Congener List. If analyzing for the PCB Congeners in conjunction with the PAHs, the Mass Spectrometer DFTPP tuning criteria for Maximum Sensitivity described in Section 10.1.4.4 must be utilized.

3. Reporting Limits

Reporting limits for individual PAH compounds can range from 10ng/L to 10,000ng/L for water samples, and a range of 8μg/Kg to 8000μg/Kg for soil/sediment, solid/waste and tissue samples. Oil samples can range from 100ug/Kg to 100,000ug/Kg. Air PUF samples can range from 200ng/PUF to 40,000ng/PUF. PCB Congener compounds can range from 1.0ng/L to 500ng/L for water samples and can range from 0.133ug/Kg to 66.7ug/Kg for soil/sediment, solid/waste and tissue samples. Oil samples can range from 5ug/Kg to 5000ug/Kg. Air PUF samples are not analyzed for PCB Congeners following this SOP. Specific reporting limits for each matrix per analyte can be viewed within the LIMS under Applications/Product Catalog.

Project-specific reporting limits may be determined based on the sample size extracted, the extract final volume and the lowest concentration standard included in the initial calibration.

4. Interferences

Phthalate esters can be a major source of contamination if any material containing plasticizers (phthalates) comes in contact with the sample during the extraction process. Use of plastic or any material containing plasticizers (phthalates) should be avoided during extraction or analysis.

When emulsions are formed during the separatory funnel extraction process, centrifuging the extract may be necessary to break down the emulsion before continuing the serial extractions. If water does filter into the collected extract, the extract should be re-filtered through sodium sulfate before the extract is concentrated to its final volume.

Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

The following cleanup methods can be used to help minimize co-extracted matrix/interferences: Method 3610 Alumina Cleanup, Method 3630 Silica Cleanup, Method 3660 Sulfur Removal using Copper and Method 3640 GPC Cleanup.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Water samples are collected in pre-cleaned 1-L amber glass bottle with a Teflon lined screw cap. Approximately 1 L of sample is required for the analysis. Soil/Sediment/Solid/Waste/Oil samples are collected in a pre-cleaned 250 mL glass jar with Teflon lined screw cap. Approximately 100g of soil/sediment is required for the analysis although more may be collected to provide a representative sample. Solid/Waste and Oil samples require half this amount, approximately 50g. Tissue samples are typically received from the field wrapped in foil and inside Ziploc baggies. As much tissue sample as possible should be provided. Air PUF samples are collected per the sited TO-13A reference method.

6.2 Sample Preservation

Water, soil/sediment, solid, waste, oil, Air PUF and tissue samples are preserved by cooling to 4°C $\pm$ 2°C and should remain at this temperature until extraction. No chemical preservative is required, and may interfere with the analysis. Sediment and Tissue samples may also be frozen to -20°C $\pm$ 2°C per client specific instructions.

6.3 Sample Shipping

No special sample shipping requirements.

6.4 Sample Handling

Water and Air PUF samples are extracted within seven days of collection. Soil, sediment, solid/waste, oil and tissue samples are extracted within 14 days of collection. Sediment and tissue samples may be frozen to extend holding times per client request. All sample extracts are analyzed within 40 days of sample preparation.

7. Equipment and Supplies

7.1 Gas chromatograph/Mass Spectrometer and Autosampler: Programmable; heating range from 40°C to 350°C; split/splitless-type inlet system; (Agilent 6890 or similar); mass selective detector (Agilent 5973, or similar); automatic injector (Agilent 7683B, or similar)..

7.2 Chromatography Column: Fused silica, capillary column, 0.25 mm ID x 30m length, 0.25µm film thickness (ZB-5, Phenomenex, 5% Phenyl-95% dimethypolysiloxane, Restek RTX5 30m x 0.25mm ID x 0.25um film thickness, or equivalent).

7.3 Data Acquisition System: Computerized system for collecting, storing, and processing detector output (Agilent Enviroquant target software or equivalent

7.4 Gases: BIP Ultra high purity helium (99.9995%), Compressed Nitrogen for N-Evap.

7.5 Hamilton Gas tight Syringes – 10uL to 1.0mL.

7.6 Vials including 2mL, 4mL, 10mL, and 40mL.

8. Reagents and Standards

Use reagent grade chemicals for all reagents. Deionized (DI) water is ASTM Type II laboratory reagent grade water.

8.1 Reagents: All solvent expirations determined as indicated by manufacturer guidelines.

8.1.1 Methylene Chloride: ACS approved, Pesticide grade, see SOP *Reagent, Solvent and Standard Control* (G-008) for additional details regarding solvent purity. Used to extract samples and prepare instrument/analytical standards

8.1.2 Acetone: ACS approved, Pesticide grade, see SOP *Reagent, Solvent and Standard Control* (G-008) for additional details regarding solvent purity. This water soluble solvent is used for surrogate and LCS/MS preparation.

8.1.3 Methanol: ACS approved, Pesticide grade, *Reagent, Solvent and Standard Control SOP* (G-008) for additional details regarding solvent purity.

8.1.4 Hexane: ACS approved, Pesticide grade, see SOP *Reagent, Solvent and Solvent Control* (1816) for additional details regarding solvent purity. This solvent is used in the extraction of Air PUF samples.

8.1.5 Ether: see SOP *Reagent, Solvent and Solvent Control* (1816) for additional details regarding solvent purity. This solvent is used in the extraction of Air PUF samples.

8.2 Analytical Standards

Standards should be stored at -10 or less, away from light when not in use. They should be discarded after 1 year unless the vendor expiration date states otherwise or if degradation is observed. Stock standards are given a 1 year expiration from the preparation date or the expiration of the primary vendor solution, whichever occurs first. Working standards are given a 6 month expiration from the preparation date or the expiration of the primary solution whichever occurs first. All analytical standards are made up in Methylene Chloride. All prep standards are made up in Acetone.

8.3 Primary Standards

8.3.1 PAH Custom Mix – Supelco 21353385 (100ug/mL)

8.3.2 Custom PCB Standard – Accustandard S-7911-2X (NOAA22 Mix 100ug/mL)

8.3.3 3,3',4,4'-Tertachlorobiphenyl – Accustandard C-077S-TP (BZ#77 100ug/mL)

8.3.4 2,3,3',4,6-Pentachlorobiphenyl – Accustandard C-110S-TP (BZ#110 100ug/mL)

8.3.5 3,3',4,4',5-Pentachlorobiphenyl – Accustandard C-126S-TP (BZ#126 100ug/mL)

8.3.6 4,4'-Dibromooctafluorobiphenyl – Ultra PPS-172 (DBOB 5000ug/mL)

8.3.7 2,2',3,3',4,5,5',6-Octachlorobiphenyl – Ultra RPC-075S (BZ#198 100ug/mL)

8.3.8 Custom Deuterated PAH Standard – Restek 563160 (2000ug/mL)

8.3.9 PAH Two Component Mixture – Cambridge Isotope Laboratory ES-5498 (2000ug/mL)

8.3.10 EPA PCB Congener Calibration Check solution – Ultra Scientific RPC-EPA2 (100ug/mL)

8.3.11 **Custom PAH Standard** - Restek 567608 (1000ug/mL)

8.3.12 **MA EPH Aromatic Hydrocarbon Standard** - Restek 31458 (1000ug/mL)

8.3.13 2**,2',3,4,4',5,5'-Heptachlorobiphenyl** – Cambridge Isotope Laboratory EC-1407-3 (BZ#180 13C12 40ug/mL)

8.3.14 **4,4'-Dichlorobiphenyl** – Cambridge Isotope Laboratory EC-1407-3 (BZ#15 13C12 40ug/mL)

8.3.15 **Semi-Volatile GCMS Tuning Standard** – Ultra GCM-150 (1000ug/mL)

8.3.16 **SV Internal Standard Mix** – Restek 31206 (2mg/mL)

8.4 Stock Solutions

8.4.1 **NOAA22 + BZ#198 Stock** – Prepared by Diluting 1.25mL of **Custom PCB Standard** (Accustandard S-7911-2X 100ug/mL), 1.25ml of **2,2',3,3',4,5,5',6-Octachlorobiphenyl** (Ultra RPC-075S BZ#198 100ug/mL), and 25ul of **4,4'-Dibromooctafluorobiphenyl** (Ultra PPS-172 DBOB 5000ug/mL) to 25mL in Hexane. Final concentration 5000ng/mL.

8.4.2 **BZ77/110/126 Stock** – Prepared by diluting 0.5ml of each **3,3',4,4'-Tertachlorobiphenyl** (Accustandard C-077S-TP BZ#77 100ug/mL), **2,3,3',4,6-Pentachlorobiphenyl** (Accustandard C-110S-TP BZ#110 100ug/mL), and **3,3',4,4',5-Pentachlorobiphenyl** (Accustandard C-126S-TP BZ#126 100ug/mL) to 10ml in hexane. Final concentration 5000ng/mL.

8.4.3 **BZ#198 and DBOB Stock** - Prepared by diluting 1.25ml of **2,2',3,3',4,5,5',6-Octachlorobiphenyl** (Ultra RPC-075S BZ#198 100ug/mL), and 25ul of **4,4'-Dibromooctafluorobiphenyl** (Ultra PPS-172 DBOB 5000ug/mL) to 25mL in Hexane. Final concentration 5000ng/mL.

8.4.4 **PCB Congener ICV Stock –** Prepared by diluting 0.020mL of **EPA PCB Congener Calibration Check solution** (Ultra Scientific RPC-EPA2 100ug/mL) to a final volume of 10mL in methylene chloride. Final concentration 200ng/mL.

8.4.5 **PAH ICV Stock –** Prepared by diluting 0.1 mL of **Custom PAH Standard** (Restek 567608 1000ug/mL), and 0.1mL of **MA EPH Aromatic Hydrocarbon Standard** (Restek 31458 1000ug/mL), to a final volume of 10mL in Methylene chloride. Final concentration 10ug/mL.

8.5 Spiking Solutions

8.5.1 **PAH Extraction Surrogate** – Prepared by diluting 1.25mL of **Custom Deuterated PAH Standard** (Restek 563160 2000ug/mL) to 500ml in Methylene Chloride. Final Concentration 5.0ug/ml.

8.5.2 **PAH Field Surrogate** – Prepared by diluting 1.0mL of **PAH Two Component Mixture** (C.I.L. ES-5498 2000ug/ml) to 10ml in Methylene Chloride. Final concentration 200ug/ml. 20ul of this resulting solution is spiked into all PUF samples prior to shipment to the client.

8.5.3 **PAH Laboratory Control Standard** – Prepared by diluting 0.5mL of **Custom PAH Standard** (Restek 567608 1000ug/mL), and 0.5ml of **MA EPH Aromatic Hydrocarbon Standard** (Restek 31458 100ug/mL) to 100mL in Acetone. Final concentration 100ug/mL.

8.5.4 Pest/Cong Surrogate Spike – Prepared by diluting 2.0mL of **BZ#198 and DBOB Stock** to 100ml in Acetone. Final concentration 100ug/mL.

8.5.5 PCB Laboratory Control Standard – Prepared by diluting 0.1mL of **Custom PCB Standard** (NOAA22 Mix, Accustandard S-7911-2X 100ug/mL), and 10mL of **Pesticide LCS Solution** (1000ng/mL) to a final volume of 100mL in Acetone. Final concentration 100ng/mL). Information for the formulation of the Pesticide LCS Solution can be found in SOP (2158_ Determination of Organochlorine Pesticides by Gas Chromatography/Electron Capture Detection. Pesticides are not reported by the method outlined in this SOP but combined extraction is sometimes performed.

8.5.6 PAH SIM Internal Standard - Prepared by diluting 0.125mL of **SV Internal Standard Mix** (Restek 31206 2000ug/ml) to 10ml in Methylene Chloride. Final Concentration 25ug/ml. 20ul of this solution is spiked into every 1ml of sample extract prior to instrument analysis. This solution can also be prepared from a serial dilution of the **8270 Internal Standard Mixture** (250ug/mL). Information for the preparation of the 8270 Internal Standard Mixture can be found in SOP (2155) Semivolatile Organic Compounds by Gas Chromatography/Mass Spectroscopy.

8.5.7 Internal Standard Primary Standard for PCB Congeners – Prepared by diluting 2.5ml of 2**,2',3,4,4',5,5'-Heptachlorobiphenyl** (Cambridge Isotope Laboratory EC-1407-3 BZ#180 13C12 40ug/mL), and **4,4'-Dichlorobiphenyl** (Cambridge Isotope Laboratory EC-1407-3 BZ#15 13C12 40ug/mL) to 10ml in Hexane. Final concentration 10ug/mL. 20ul of this solution is spiked into every 1ml of sample extract prior to instrument analysis.

8.6 Calibration Solutions

8.6.1 PAH/PCB Combo ICAL /Stock Solution – Prepared by following the recipe outlined in the following table, bringing the components to either a final volume of 1mL or 10mL in methylene chloride. The final concentration is 5,000ng/ml for PAHs and 500ng/mL for PCB congeners.

Standard/Solution	Volume Added
PAH Custom Mix (Supelco 21383385 100ug/ml	1.0mL
Custom Deuterated PAH Standard (Restek 563160 2000ug/mL)	50uL
PAH Two Component Mixture (C.I.L. ES-5498 2000ug/ml	50uL
NOAA 22 + Surrogates Stock	1.0mL
BZ77/110/126 Stock	1.0mL

The following calibration levels are prepared by diluting the indicated volume of the above stock solution to 10mL in methylene chloride.

8.6.1.1 Level 1 (10/0.5ng/mL): 0.010mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.1.2 Level 2 (20/1.0ng/mL): 0.020mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.1.3 Level 3 (100/5ng/mL): 0.100mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.1.4 Level 4 (200/10ng/mL): 0.200mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.1.5 Level 5 (500/25ng/mL): 0.500mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.1.6 Level 6 (1000/50ng/mL): 1.0mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.1.7 Level 7 (2000/100ng/mL): 0.200mL of Stock diluted to a final volume of 1.0mL in methylene chloride.

8.6.1.8 Level 8 (5000/250ng/mL): 5.0mL of Stock diluted to a final volume of 10mL in methylene chloride.

8.6.2 Initial Calibration Verification Solutions (ICV)

8.6.2.1 Initial Calibration Verification PAH SIM Working Solution (500ng/ml): Created by diluting 0.5ml of **PAH ICV Stock** solution (10,000ng/ml) to 10ml in methylene chloride

8.6.2.2 PCB Congener ICV Working (25ng/mL): 1.25mL of **PCB Congener ICV Stock** solution (200ng/mL) diluted to a final volume of 10mL in methylene chloride.

8.6.3 DFTPP Tuning Solution – Prepared by diluting 1.25mL of **Semi-Volatiles GCMS Tuning Standard (**Ultra GCM-150 1000ug/mL) diluted to a final volume of 25mL in methylene chloride.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A method blank (must be extracted with every batch of samples extracted or every 20 samples, whichever is more frequent. Organic compounds of interest must not be detectable in the method blank at a concentration greater than the reporting limit.

Corrective Action: Extraction of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. The results are qualified with a “B” flag for any associated sample concentrations that are less then 10x the blank concentration for the analyte. Exceptions may be made with if the samples associated with the out of control method blank are non-detect for the compound of interest, or if sample concentrations are greater than 10x the blank levels. In such cases when the sample results are accepted, the client is notified in a project narrative associated with the sample results.

9.2 Laboratory Control Sample (LCS)

Laboratory control samples (LCS/LCSD) must be prepared once per every 20 samples or per extraction batch, whichever is more frequent, and spiked with PAH SIM/PCB Congener spike solution, or TO-13 spike solution.

Acceptable Recovery limits are 40% - 140% for all matrices, except for TO-13 Air PUF - see Table D for TO13 LCS recovery criteria. The acceptable relative percent difference (RPD) between the LCS/LCSD is ≤30%. ≤10% of target compounds are allowed out provided recovery is >10%.

PAH and PCB portions need to be evaluated separately

Limits are adapted from MADEP-MCP protocol and/or TO-13A.

Corrective Action: Analysis must be repeated if an analytical error is suspected. If the LCS/LCSD recoveries and/or %RPD are still out of control, re-extract and re-analyze the LCS/LCSD and all associated samples. Samples cannot be reported until an acceptable LCS is obtained.

9.3 Initial Calibration Verification (ICV)

The initial calibration for each compound of interest must be verified prior to sample analysis. This is accomplished by analyzing a second source calibration standard (Section 8.2.10).

Calculate the % D for each analyte. If the % D for each analyte is ≤30% then the calibration is assumed to be valid. If this criterion is not met, the standard is reanalyzed once. If the standard failure continues, corrective action must be taken prior to sample analysis.

A new initial calibration must be performed and acceptable ICV results obtained prior to any sample analysis.

9.4 Continuing Calibration Verification (CCV)

On a daily basis after the DFTPP has passed, a mid-level (500/25ng/mL) continuing calibration verification standard which contains all of the analytes of interest is analyzed.

9.5 Matrix Spike/ Matrix Spike Duplicate (MS/MSD)

Matrix spike / matrix spike duplicate (MS/MSD) samples will be performed upon client and/or work plan request. MS/MSD is not performed for Method TO-13A.

See Section 12 for MS/MSD recovery limits and %RPD limits.

Calculate the %RPD as described in Section 9.6.

Corrective Action: If the % recovery and/or %RPD exceeds the control limits and the LCS is compliant; include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the reported results as evidenced by MS/MSD recoveries and/or %RPD outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses are performed upon client and/or work plan request. Acceptable relative percent differences (RPD) of duplicates are listed in Section 12. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit.

Calculate the RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:
R1 = sample Replicate #1
R2 = sample Replicate #2

Corrective Action: If the % RPD exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the reported organic results as evidenced by the matrix duplicate % RPD exceedance.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogate Spikes: Surrogate spikes must be added to QC and field samples to evaluate the extraction method performance.

See Section 12 for surrogate recovery acceptance criteria.

Corrective Action: Up to one surrogate can be out in the PAH portion and in the PCB portion, but not less than 10% recovery, before any corrective action is necessary. Otherwise, analysis must be repeated if an analytical is suspected. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm the sample matrix. If *obvious* matrix interferences are noted, consultation with the Department Manager, Laboratory Director or QA Officer may be necessary to confirm the need for sample re-extraction. If no re-extraction occurs, the surrogate results and reasons for the no re-extract decision must be discussed in the project narrative to the client.

9.7.2 Standard Reference Materials (SRMs): Standard reference materials (SRMs) are available from the National Institute of Standards and Technology (NIST) and are extracted and analyzed with samples on a project-specific basis. These are not used as controls, but rather to evaluate potential matrix effects in associated samples for the target compounds being evaluated.

See Section 12 for SRM recovery acceptance criteria.

Corrective Action: Analysis must be repeated if an analytical error is suspected. If the % recovery and/or %D still exceeds the control limits, and the LCS and MS/MSD pair are compliant, include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the reported results as evidenced by SRM % recoveries and/or %D values outside of QC limits. These cases are normally isolated to the SRM, if all other controls are within limits.

9.7.3 Internal Standards: Internal standards must be added to all sample extracts, QC samples and standards for quantitation purposes.

The internal standards in the samples should remain at constant area counts with respect to the continuing calibration analyzed at the beginning of the run. Sample IS areas must be 50% to 200% of the Internal Standards in the Continuing Calibration.

Additionally, the IS retention times should not differ more than +0.06 RRT from the Continuing Calibration.

Corrective Action: Analysis must be repeated once unless there are obvious samples matrix interferences, e.g. If the sample extract was very colored and viscous, or there are obvious chromatographic interferences. If *obvious* matrix interferences are noted, consultation with the SemiVolatiles Department Manager, Laboratory Director or QA Officer may be necessary to confirm the need for sample re-analysis.

9.8 Method Sequence

- Tune
- CCV
- Method Blank
- LCS
- LCSD
- Samples

10. Procedure

10.1 Equipment Set-up

10.1.1 The **basic GC parameters** are as follows:

Injection Port Temp: 300°C
Oven Equib Time: 0.50 min
Oven Max: 350°C
Oven: On
Cryo: Off
Ambient: 45°C
Cryo Blast: Off
Initial Temp.: 45°C
Initial Time: 3.00 min

Level	Rate (°C /min)	Final Temp. (°C)	Final Time (min)
1	25.00	250	0
2	10.00	310	5.0
3	0.0off		

Final Time: 22.2min

10.1.2 The **basic injection port parameters** are as follows:

"Split" mode
Equilibrium Time: 0.50 minute
Temp: 300°C
Pressure: 8.28 psi (10.5 psi) Total Flow: 69.3mL/min)
Split: 50:1
Split Flow: 65 mL/min

Injection volume: 3uL

Please note that this may differ slightly from one instrument to another.

10.1.3 Column conditions parameters are as follows:

Column flow: 1.3mL/min
Linear Velocity: 42 cm/sec

10.1.4 Tuning:

10.1.4.1 Tune acceptance should be verified at the beginning of every 12 hour analytical shift. Before the analytical standards are analyzed the mass spectrometer must be adjusted to meet the proper ion criteria for DFTPP. This is demonstrated by injecting into the GC/MS system 1uL of a 50ug/mL DFTPP solution.

10.1.4.2 Within the Enviroquant software, "Autofind" is used first to evaluate Tune. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction is required, and must be accomplished using a single scan acquired within 20 scans of the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or any other discrete peak that does not coelute with DFTPP.

10.1.4.3 If the "AutoFind" tune evaluation does not meet the criteria below, manual evaluation of the tune can be performed by attempting either of the options below:

- Blow up the DFTPP peak on the screen and select either one single scan at the apex of the peak, or a scan immediately preceding or following the apex.
- Take the average of the scans across the entire peak.

The following DFTPP mass intensity criteria should be used:

- **for PAH SIM analysis:**

The response of Benzidine and Pentachlorophenol, when monitored on a daily basis, will indicate the efficiency of the chromatographic system. Benzidine and Pentachlorophenol should be present at their normal responses and no peak tailing should be visible. The tailing factor of Benzidine and Pentachlorophenol must not exceed 2. DDT breakdown must be <20%. Although moderate tailing may indicate maintenance on the instrument will be required soon, these additional compounds in the DFTPP standard are included as an aid in instrument performance evaluation

DFTPP KEY MASSES AND ABUNDANCE CRITERIA

Mass	m/z Abundance criteria
68	Less than 2 percent of mass 69.
69	confirmed to be present
70	Less than 2 percent of mass 69.
197	Less than 2 percent of mass 198.
198	Base peak, or >50 percent of Mass 442.
199	5-9 percent of mass 198.
365	Greater than 1 percent of mass 198.
441	Present but less than 24 percent of mass 442.
442	Base Peak, or > 50 percent of mass 198.
443	15-24 percent of mass 442.

- For PAH/PCB combined analysis:

DFTPP KEY MASSES AND ABUNDANCE CRITERIA
(Maximum Sensitivity)

Mass	m/e Abundance criteria
51	N/A
68	N/A
70	N/A
127	30-80 percent of mass 198.
197	Less than 3 percent of mass 198.
198	Greater than 40 percent of mass 442.
199	5-15 percent of mass 198.
275	15-50 percent of mass 198.
365	Greater than 3 percent of mass 198.
441	Present but less than mass 443.
442	Base peak, 100 percent relative abundance.
443	18-30 percent of mass 442.

10.2 Initial Calibration

10.2.1 After the DFTPP passes criteria, a set of multi-level calibration standards listed in Section 8.2.9 are analyzed, from low concentration to high. A minimum of five calibration levels are analyzed. The calibration standards are stored in the standards freezer. When the Initial Calibration is typed into a sequence, the standard ID for each of the levels must be noted in the “comments” of the sequence. (Refer to the Reagent Solvent and Standard Control SOP (SOP 1816) for further information regarding standard labeling convention.

10.2.2 Once the calibration curve is accepted and prior to processing any samples, the analyst must demonstrate through the analysis of a MB or instrument blank that equipment and reagents are free from contaminants and interferences. If a peak is found in the blank that would prevent the identification or bias the measurement of an analyte, the analyst should determine the source of the contaminant peak and eliminate it, if possible. Blanks

are generally considered to be acceptable if target analyte concentrations are less than one half the LLOQ or are less than project specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected

10.2.3 Once the standards have been analyzed, they are reduced by the search software of the Enviroquant data system. Once all the components are identified, a linear curve is calculated for the components. The criteria for evaluation are as follows:

The RSD of each target analyte, for each level in the ICAL, must meet the minimum response factor criteria found in Table C. The %RSD for each target analyte must not exceed 20%. If the %RSD for any target exceeds 20%, then the linear calibration model must be employed. A linear fit is acceptable if the correlation coefficient is greater than 0.99. A minimum of 5 levels are required for a linear calibration model and a minimum of 6 levels are required for a quadratic calibration model. When calculating calibration curves using the linear regression model, a minimum quantitation check must be performed on the lowest calibration point. The recalculated concentration of the lowest calibration point should be within 30% of the true value. If this criterion is not met then corrective action must take place and a new initial calibration must be performed prior to sample analysis.

10.3 Equipment Operation and Sample Processing

10.3.1 Sample Extraction and Cleanup

Samples for PAH SIM/ PCB Congeners are generally extracted using one of the following SOPs:

- *Separatory Funnel Extraction* 3510C (SOP 2165)
- *Microscale Solvent Extraction* 3570 (SOP 2172)
- *Soxhlet Extraction for PUF* 3540C (SOP 2174)
- *Waste Dilution and Oil Prep* 3580 (SOP 2265)

Samples for PAH SIM/ PCB Congeners may be cleaned up before transfer to the instrument laboratory using one of the following SOPs:

- *Gel Permeation Column Cleanup* SOP (SOP 2167)
- *Silica Gel Clean-up* SOP (SOP 2170)
- *Sulfur Cleanup* SOP (SOP 2168)

10.3.2 The prep lab staff will transfer the samples to the instrument laboratory. The samples are generally brought to a 1.0mL to 4.0mL final volume; 1.0mL is transferred and any remaining sample is put into archive. One aliquot of each sample is then logged into the Internal Chain of Custody book and placed in the sample extract holding refrigerator located in the instrument laboratory.

10.3.3 All samples and standards are spiked with Internal Standards (IS) before analysis. The IS is intended to be used for both quantification and the establishment of relative retention times.

10.3.4 The analyst may determine to screen any samples for needed dilution. A sample that will need a dilution either for target analytes that are over calibration, or samples may need to

be diluted solely for matrix issues when the chromatogram shows a large UCM (unresolved complex mixture) or other non-target interferences, or when the extract itself is very colored and viscous. All of the samples at 500uL (including the QC samples of a method blank and LCS) are spiked with 10uL internal standard (see section 10.3.2 for specifics regarding the internal standard) for a concentration of 5ug/mL. The samples are shaken briefly after the internal standard is added to ensure mixing.

10.3.5 After the samples have been analyzed, the data files from the instrument are transferred to the server. The samples are quantified versus the proper method. The QCPRN1.MAC macro creates a form with which to easily check internal standard and surrogate criteria. The ISTDRPT.MAC macro may also be utilized to check the internal standard and surrogate criteria. The following should be reviewed initially:

10.3.5.1 Are all the surrogates within QC criteria? See Section 12 and Table A for surrogate recovery ranges.

10.3.5.2 Are all the internal standards within 50-200% and within ±0.06 RRT of the daily CCAL? If not, the samples should be checked for matrix interferences that may be causing these issues. The IS peaks should also be evaluated for peak splitting or incorrect integration by the software. A sample may not need to be reanalyzed if it can be determined (with guidance from a supervisor) that the QC is exceeded due to matrix interference.

10.3.5.3 Are all target analytes within calibration range? If not, the sample must be diluted and re-analyzed. If a dilution is performed after the internal standard has already been added, it will be necessary to add additional IS in order to provide a concentration of 5ug/mL. Conversely, if a sample has been over-diluted, it may need to be analyzed at less of a dilution to detect target analytes that may have been diluted out.

10.3.5.4 Are all analyses within 12 hour tune time? If a sample is analyzed outside tune time, it must be re-analyzed within another tune clock.

10.3.6 The sequence should also be printed out from Chemstation, initialed and dated, placed in the working logbook.

10.3.7 If anything in the initial review of the data indicates that there should be a re-analysis or a re-extract, the reason for re-analysis or re-extract should be noted on the sequence.

10.3.8 If a re-extract is required, the “Request for Reanalysis/Repreparation” book should be filled out and a photocopy of the appropriate page should be given to the Preparation Group leader or the SemiVolatile Organics Department Manager.

10.3.9 A standard sequence (saved as S5073001;”S” for semivolatile;”5” for PAH5; 0730 for July 30, and 01 is the first sequence that day) would appear as follows:

Tune: WG#-1, -4, etc. This is the Analytical working group number generated by the Alpha LIMS that denotes the Tune.

CCV: WG#-2, -3, etc. This is the Analytical working group number generated by the Alpha LIMS that denotes the calibration verification.

10.4 Continuing Calibration

10.4.1 On a daily basis after the DFTPP has passed, a mid-level (500/25ng/mL) continuing calibration standard which contains all of the analytes of interest is analyzed. The criteria for acceptance are :

10.4.1.1 The %D for all targets must be ≤20%. Up to 20% of all targets are allowed to have %D > 20% with high bias as long as all associated samples are non-detect for those analytes. If any of the targets have %D > 20% with low bias, corrective action must take place and all associated samples must be reanalyzed.
PAH and PCB portions need to be evaluated separately.

10.4.1.2 The retention times of the internal standards must be within ±0.05 RRT of the previous daily standard.

10.4.1.2 The area counts of the internal standards within the continuing calibration must be within 50 to 200% of the mid-level in previous ICAL

10.5 Preventive Maintenance

All repair and non-routine maintenance records including outside service visits are maintained in the instrument maintenance logbooks.

Injection Port Maintenance: Maintenance should be done when the daily CCAL starts to demonstrate degradation. The type of samples analyzed will have an effect on how soon maintenance should be performed.

Septum Maintenance: The septum needs to be changed approximately every two-hundred injections. Unscrew the top septum nut, remove the pierced septum, and replace with a new 11mm Thermolite green septum (Restek) or equivalent. Screw the top septum nut back on.

Column Maintenance: Maintenance should be done when the daily CCAL starts to demonstrate degradation. The type of samples analyzed will have an effect on how soon maintenance should be performed. Generally maintenance is performed by trimming 6 cm off the front of the column. The column is then installed into the injection port liner, and the inlet nut is tightened.

11. Data Evaluation, Calculations and Reporting

11.1 Qualitative Analysis

11.1.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as three ions of greatest relative intensity, or any ions over 20% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

11.1.2 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. A peak selected by the data system, based on the presence of target specific ions at a target specific retention time will be accepted as meeting this criteria.

11.1.3 The relative retention time of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

11.1.4 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%). Outlying abundances maybe included due to the presence of non-target interference. The relative intensities are monitored daily. The relative intensities will be updated when they exceed established values from the reference spectrum.

11.1.5 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

11.1.6 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e. a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

11.1.7 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes coelute, (i.e. only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum may contain extraneous ions contributed by the coeluting compounds.

11.2 Response factors and %RSD to evaluate initial calibration acceptability

Calculate RF by:

$$RF = \frac{area_{cmp}}{area_{is}} \times \frac{conc_{is}}{conc_{cmp}}$$

where:

area cmp = Area of the characteristic ion for the compound being measured.
area is = Area of the characteristic ion for the specific internal standard.
conc is = Concentration of the specific internal standard.
conc cmp = Concentration of the compound being measured.

Calculate %RSD by:

$$\%RSD = \frac{SD}{\bar{x}} \times 100 \qquad SD = \sqrt{\sum_{i=1}^{N} \frac{(x_i - \bar{x})^2}{N-1}}$$

where:

%RSD = percent relative standard deviation
x = average of RF's
SD = standard deviation
xi = analytical results of each level in the final reporting units
N = number of results (levels)

11.3 % Difference to evaluate daily calibration standard

Calculate %D by:

$$\%D = \frac{\overline{RF}_i - RF_c}{\overline{RF}_i} \times 100$$

where:

RFi = initial calibration average RF
RFc = continuing calibration RF

11.4 Results of Water Analysis

Calculation as performed in worksheet:

$$\textit{concentration (ng/L)} = \frac{\textit{(Conc) (Vf) (DF) x 1000}}{\textit{(Vi)}}$$

where:

Conc = Raw result concentration obtained from the quantitation report in ng/mL (ppb)
Vf = Final volume of extract (mL)
Vi = Volume of sample extracted (mL)
DF = Dilution factor, for manually prepared dilutions, not instrumental “dilutions”

11.5 Results of Sediment/Soil, Sludge/Waste Tissue, Oil and Air PUF Analysis

Calculation as performed in Excel worksheet:

$$\textit{concentration (}\mu\textit{g/Kg)} = \frac{\textit{(Conc) (Vf) (DF)}}{\textit{(W) (\%S)}}$$

where:

Conc = Raw result concentration obtained from the quantitation report in ng/mL (ppb)
DF = Dilution factor, for manually prepared dilutions, not instrumental “dilutions”
Vf = Extract final volume (mL)
W = Aliquot of sample (wet), g (for Air PUF, sample amount is 1)
%S = Sample % solid

NOTE: Tissue samples are calculated in the same manner, however %S may, or may not be utilized depending upon the client request to report data as a wet or dry weight. Oil and Air PUF sample calculations do not use %S in the final concentration.

11.6 The calculation for percentage breakdown for DDT is:

$$\%\ \text{Breakdown DDT} = \frac{\text{(Area DDD + Area DDE)}}{\text{(Area DDD + Area DDT + Area DDE)}} \times 100$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All results for the organic compounds of interests are reportable without qualification if extraction and analytical holding times are met, preservation (including cooler temperatures) is met, all QC criteria are met, and matrix interference is not suspected during extraction or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

If non-compliant organic compound results are to be reported, the SemiVolatile Organics Department Manager, the Laboratory Director, and/or the QA Officer must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

QC Parameter	Acceptance Criteria
Initial Calibration Curve	Initial Calibration Curve ≤20%RSD or ≥0.99 linear for all targets. If linear regression is used, recalculation of the lowest calibration standard is required to be within 30% of the true value.
Independent Check Verification	+/- 30% recovery of the true values,
Continuing Calibration Verification	Analyzed every 12hr or at the minimum of every 20 samples, < +20% D for all target analytes. .
Method Blank	No analyte detected at or above the reporting limit, "B" qualify analyte if detected concentration is less than 10X the concentration found in the associated sample(s)
Laboratory Control Sample and LCSD	40-140% for all matrices, except for TO-13 - see Table D; 30% RPD.
Matrix Spike / Matrix Spike Duplicate	40-140 recovery; 30% RPD between the duplicates.
Sample / Sample Duplicate	30% RPD between the duplicates.
Surrogates	PAHSIM 30-130% all matrices; PAH/PCB: PAH 30-150% and PCB 50-125% all matrices; USACOE RIM PAH/PCB: 30-150% all matrices; TO-13: see Table A One Surrogate is allowed out from each PAH or PCB portion
Internal Standards	50% - 200% of the daily CCV area for the Internal Standards
Standard Reference Material	40-140% recovery based on values provided by Vendor
Project specific work plans and DQOs supersede the above limits.	

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste and Sample Disposal SOP (1797) for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP 1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)

SOP 1739 Demonstration of Capability (DOC) Generation
SOP 1797 Hazardous Waste and Sample Disposal

SOP 1731 Manual Integration & Compound Rejection

16. Attachments

Table A: Surrogate Recovery Acceptance Criteria

Table B: Target Compounds and Quantitation Ions

Table C: Recommended Minimum Response Factor Initial and Continuing Calibration

Table D: Matrix Spike and LCS Recovery Limits

Table A
Surrogate Recovery Acceptance Criteria

PAH Surrogate (separate analysis)	30-130% all matrices
PCB Congener Surrogate (separate analysis)	50-125% all matrices
PAH/PCB Surrogate (combined analysis)	PAH: 30-150%; PCB: 50-125% all matrices
USACOE RIM PAH/PCB Surrogate	30-150% all matrices

Surrogate Recovery Acceptance Criteria for TO-13

Compound	Recovery Limits (%)
PYRENE-D10	60-120
BENZO(B)FLUORANTHENE-D12	60-120
FLUORANTHENE-D10	60-120
BENZO(A)PYRENE-D12	40-120

Table B
Target Compounds and Quantitation Ions

Compound	Quantitation Ions Primary	Secondary
Acenaphthene	153	154
Acenaphthene-d10 **(IS)**	164	162
Acenaphthylene	152	151
Anthracene	178	176
Benzo(a)Anthracene	228	229
Benzo(b)fluoranthene	252	253
Benzo(k)fluoranthene	252	253
Benzo(g,h,i)perylene	276	138
Benzo(a)pyrene	252	253
Chrysene	228	226
Chrysene-d12 **(IS)**	240	120
Dibenz(a,h)anthracene	278	139
Fluoranthene	202	101
Fluorene	166	165
2-Methylnaphthalene-D10 **(SURR)**	152	150
Indeno(1,2,3-cd)pyrene	276	138
2-Methylnaphthalene	142	141
1-Methylnaphthalene	142	141
Naphthalene	128	129
Naphthalene-d8 **(IS)**	136	68
Fluoranthene-D10 **(SURR)**	178	179
Perylene-d12 **(IS)**	264	260
Phenanthrene	178	179
Phenanthrene-d10 **(IS)**	188	94
Pyrene	202	200
Pyrene-D10 **(SURR)**	212	211
1,4-Dichlorobenzene	146	148
2-Methylnaphthalene	142	141
1-Methylnaphthalene	142	141
Dibenzothiophene	184	139
Benzo(a)pyrene-D12 **(SURR)**	264	132
2-Chloronaphthalene	162	164
Benzo)b)fluoranthene-D12 **(SURR)**	264	260
Biphenyl	154	153
2,6-Dimethylnaphthalene	156	155
2,3,5-Trimethylnaphthalene	170	155
1-Methylphenanthere	192	191
Perylene	252	253
Benzo(e)pyrene	252	253
BZ#15-C13 (IS)	234	236
BZ#180-C13 (IS)	406	408
DBOB (Surr)	456	296
BZ198 (Surr)	428	429
BZ#8	222	224
BZ#18	256	258

Table B (continued)
Target Compounds and Quantitation Ions

Compound	Quantitation Ions	
	Primary	Secondary
BZ#28	256	258
BZ#52	292	290
BZ#49	292	290
BZ#44	292	290
BZ#66	292	290
BZ#101	326	324
BZ#87	326	324
BZ#77	292	290
BZ#110	326	324
BZ#118	326	324
BZ#153	360	362
BZ#184	394	396
BZ#105	326	324
BZ#138	360	362
BZ#126	326	324
BZ#187	394	396
BZ#183	394	396
BZ#128	360	362
BZ#180	394	396
BZ#170	394	396
BZ#195	428	429
BZ#206	464	466
BZ#209	498	500

Table C: Recommended Minimum Response Factor Initial and Continuing Calibration

Semivolatile Compounds	Minimum Response Factor (RF)
Naphthalene	0.7
2-Methylnaphthalene	0.4
1,1'-Biphenyl	0.01
Acenaphthylene	0.9
Acenaphthene	0.9
Fluorene	0.9
Phenanthrene	0.7
Anthracene	0.7
Fluoranthene	0.6
Pyrene	0.6
Benzo(a)anthracene	0.8
Chrysene	0.7
Benzo(b)fluoranthene	0.7
Benzo(k)fluoranthene	0.7
Benzo(a)pyrene	0.7
Indeno(1,2,3-cd)pyrene	0.5
Dibenz(a,h)anthracene	0.4
Benzo(g,h,i)perylene	0.5

Table D. Matrix Spike and LCS Recovery Limits

Compound	All Matrices Recovery Limits (%)
Acenaphthene	40-140
Acenaphthylene	40-140
Anthracene	40-140
Benzo(a)Anthracene	40-140
Benzo(b)fluoranthene	40-140
Benzo(k)fluoranthene	40-140
Benzo(g,h,i)perylene	40-140
Benzo(a)pyrene	40-140
Chrysene	40-140
Dibenz(a,h)anthracene	40-140
Fluoranthene	40-140
Fluorene	40-140
Indeno(1,2,3-cd)pyrene	40-140
Naphthalene	40-140
Phenanthrene	40-140
Pyrene	40-140
1,4-Dichlorobenzene	40-140
2-Methylnaphthalene	40-140
1-Methylnaphthalene	40-140
Dibenzothiophene	40-140
2-Chloronaphthalene	40-140
Biphenyl	40-140
2,6-Dimethylnaphthalene	40-140
2,3,5-Trimethylnaphthalene	40-140
1-Methylphenanthere	40-140
Perylene	40-140
Benzo(e)pyrene	40-140
BZ#8	40-140
BZ#28	40-140
BZ#52	40-140
BZ#49	40-140
BZ#44	40-140
BZ#66	40-140
BZ#101	40-140
BZ#87	40-140
BZ#77	40-140
BZ#110	40-140
BZ#118	40-140
BZ#153	40-140
BZ#184	40-140
BZ#105	40-140
BZ#138	40-140
BZ#126	40-140
BZ#187	40-140
BZ#183	40-140
BZ#128	40-140
BZ#180	40-140
BZ#170	40-140
BZ#195	40-140
BZ#206	40-140
BZ#209	40-140

Matrix spike and LCS recovery Limits for TO-13

Compound	Recovery Limits (%)
Acenaphthene	60-120
Acenaphthylene	30-114
Anthracene	40-98
Benzo(a)Anthracene	60-120
Benzo(b)fluoranthene	60-120
Benzo(k)fluoranthene	60-120
Benzo(g,h,i)perylene	60-120
Benzo(a)pyrene	60-120
Chrysene	60-120
Dibenz(a,h)anthracene	60-120
Fluoranthene	60-120
Fluorene	60-120
Indeno(1,2,3-cd)pyrene	60-120
Naphthalene	60-120
Phenanthrene	60-120
Pyrene	60-120
1-Methylnaphthalene	60-120
2-Methylnaphthalene	60-120

Particle Size Analysis of Soils – With / Without Hydrometer

References: ASTM D6913-04 (Re-approved 2009), *Standard Test Method for Particle-Size Distribution (Gradation) of Soils using Sieve Analysis*.

ASTM D7928-16, *Standard Test Method for Particle-Size Distribution (Gradation) of Fine-Grained Soils using the Sedimentation (Hydrometer) Analysis*.

ASTM D2487-11 (Re-approved 2011), *Standard Practice for Classification of Soils for Engineering Purposes (Unified Soil Classification System),* pp.246-257.

ASTM D2216-90 (Re-approved 1990), *Standard Test Method for Laboratory Determination of Water (Moisture) Content of Soil, Rock, and Soil-Aggregate Mixtures*, pp.278-281.

ASTM D2217-66 (Re-approved 1978), *Standard Method for Wet Preparation of Soil Samples for Particle Size Analysis and Determination of Soil Constants*.

The American Association of State Highway and Transportation Officials, *Standard Specifications for Transportation Materials and Methods of Sampling and Testing*, July 1978, 12th edition, pp.250-276.

Department of the Army, E M 1110-2-1906, *Laboratory Soils Testing*, November 1970, pp. V-1 – V-28.

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1. Scope and Application

Matrices: Soil and sediment samples, as received from the field.

Definitions: Refer to Alpha Analytical Quality Manual.

This analytical method covers the quantitative determination of the distribution of particle size in soils and sediments. The distribution of particle sizes larger than 75μm (retained on the No.200 sieve) is determined by sieving. The distribution of particle sizes smaller than 75μm (not retained on the No.200 sieve) is determined by a sedimentation process, using a hydrometer.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the equipment and in the interpretation of data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability and completing the record of training.

A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the Quality Assurance Officer and/or Laboratory Director on a case-by-case basis.

2. Summary of Method

Particle Size *without* Hydrometer (often referred to as *Wet Sieve* or *Grain Size Analysis*) is determined by drying a homogenized soil sample. The dried homogenate is placed onto a number 200 sieve, washed with tap water, re-dried, and re-homogenized. The dried soil is

mechanically sieved with an array of sieve sizes. The distribution of soil sample remaining in each of the sieves is determined by weight and represents various classes of soil including cobble, gravel, course and fine sand, silt and clay.

Particle Size with Hydrometer is determined by drying a homogenized soil sample and immersing it in a *sodium hexametaphosphate* solution. A hydrometer test is then performed which defines the fraction of sample that is silt and clay. The sample from the hydrometer test is transferred to a number 200 sieve and washed with tap water to remove all particles less than 75 μm. The sample remaining on the number 200 sieve is then re-dried and re-homogenized and is mechanically sieved with an array of sieve sizes that define the medium and fine sands present in the sample.

When receiving limited amount of sample (e.g. 75-100gm), the test can be performed on a wet sample. % solids must be analyzed and the dry weight used for final calculations. Then proceed as in Section 10.3.2.

2.1 Method Modifications from Reference

The hydrometers are left in place between reads to ensure stability and reduce loss of material and testing volume. An internal study demonstrated equivalent data results through real world sample duplicates.

3. Reporting Limits

Not Applicable

4. Interferences

Special consideration should be given to soils from a marine environment or other sources where high soluble salt concentrations may be present. The degree to which the salts present in these soils are diluted or concentrated must be given consideration if meaningful results are to be obtained.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

A minimum of 200-grams of sample should be collected in an 8 oz. plastic jar.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

The samples must be aliquoted and dried in an oven or air dried in a hood. Remaining sample can be stored at room temperature. Hold times are not applicable to particle-size determination.

7. Equipment and Supplies

7.1 Analytical balance: Capable of weighing to the nearest 0.01g

7.2 Aluminum pie tins, plastic 250-mL beakers, *Parafilm*

7.3 Vapor hood, sink with tap water for rinsing/cleaning

7.4 Mechanical stirring apparatus

7.5 Stainless steel spatulas, spoons, and mixing bowls

7.6 Drying ovens: Maintained at 110±5°C , 40°C±5°C

7.7 Hydrometer: 151H, (or 152H for high fines samples) per ASTM specifications E 100

7.8 Sedimentation cylinder: 1000mL glass with large rubber stoppers to fit

7.9 Thermometer: Accurate to 0.5°C (able to read 20°C)

7.10 Timing device: electronic timer

7.11 Constant room-temperature, 22±5°C

7.12 Mechanical sieve shakers: Models RX-29, Type:110V, 60HZ and CI-305A-1, Type: 120V, 60HZ

7.13 Sieves: Square-mesh, woven-wire cloth per ASTM specifications E11: 3in., 2in., 1.5in., 1.0in., 0.75in., 0.375in., #4, #10, #20, #40, #60, #100, #140, #200, Pan

7.14 Bristle brushes: For sieve cleaning

7.15 Mortar and Pestle

8. Reagents and Standards

8.1 Deionized (DI) water or RO water: Lab generated. Note: Tap water cannot be used for the soaking or hydrometer solutions.

8.2 Sodium Hexametaphosphate Solution: Dissolve 240g into approx.1000-mL DI water, dilute to 6-L with DI water. (Solution expires monthly, and must be kept at a pH of 8-9, using sodium carbonate.) Store at room temperature.

8.3 Sodium Carbonate, 0.5M: Thoroughly dissolve 26.49g of Na_2CO_3 in 400-mL DI water, dilute to 500-mL. Store at room temperature; expires in one year stored in a closed vessel.

8.4 Wide-Range pH paper.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Not applicable.

9.2 Laboratory Control Sample (LCS)

Not applicable.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

One Duplicate per 20 or one duplicate per batch.

RPD <25%, narrate non-conformances in the final report

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

Refer to Section 10

10. Procedure

10.1 Equipment Set-up

Not applicable.

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Sample Preparation – Particle Size with/without Hydrometer

Samples are prepared immediately upon arrival.

10.3.1.1 Homogenize the sample using a stainless steel mixing bowl, spatula, and/or spoon. Transfer a sample aliquot into a properly labeled aluminum pie tin and dry in an oven or set to air-dry in a hood. (It is recommended for visually wet samples that the entire sample is set to dry to ensure adequate mass is available, since dry weight is unknown.) A matrix duplicate, if not already specified by the client, is chosen and set to dry in its own tin. *Note: Samples can be dried overnight in an oven set at 110 ℃, with the exception of claylike samples. Claylike samples are partially dried in an oven or in the hood and broken up as they are drying to help prevent solidifying of samples. Note in the case of limited sample, a smaller aliquot may be taken upon approval of the Project Manager.*

10.3.1.2 Carefully grind dried sample using a mortar and pestle. Do not grind any shells, wood, rocks, or large particles. Remove any matter that does not properly represent the sample.

10.3.1.3 If only a limited amount of sample is available, see Section 2.0 regarding Hydrometer analysis.

10.3.2 Particle Size (without Hydrometer) Analysis

10.3.2.1 Weigh approximately 100g to 500g of the sample, as prepared in Sec.10.3.1.1, and record this weight as the *original sample weight*. A sample consisting of heavy materials, such as large rocks, requires a greater *original sample weight* for proper sample representation.

10.3.2.2 Transfer entire sample aliquot to a No. 200 sieve and wash with tap water until the wash water is clear. (Be very careful not to damage the sieve or to lose any soil by splashing the material out of the sieve. Do not force material through sieve with fingers.) Transfer washed material on the No. 200 sieve to aluminum tin. Dry the residue in an oven at 105°C, overnight, or until completely dry.

10.3.2.3 Remove dried sample residue from oven. Gently grind and weigh the residue. Record this weight as the *post-wash weight*.

10.3.2.4 Refer to Section 7.13 for list of sieves. Add sample residue to the stack of sieves. Place lid on stack of sieves. Shake sieves containing sample for four (4) minutes. *Note: Seven sieves and pan will fit on each mechanical shaker at one time, but up to thirteen sieves may be required. Consult the Section Supervisor for proper guidance.*

10.3.2.5 Remove the stack of sieves from the shaker and record the final weight of the contents of each sieve. A brush is used to remove all sample from sieves and collected onto the weighing container.

10.3.2.6 Optionally, if oily material adheres to the sieves after brush cleaning: Soak in soapy hot water and use the wire (coarse) sieve brush to remove particulate, taking care not to damage the wire mesh. Brush the sieve on both sides, paying attention to fine matter. Once there are no particles left on the sieve mesh, and the sieve rinses clean, it is ready for reuse.

10.3.3 Particle Size with Hydrometer Analysis

10.3.3.1 Weigh approximately 50g of the sample, as prepared in Section 10.3.1.1, into a 1000ml glass cylinder. The suspended sediment capacity of the 152H hydrometer is about 55 g of dry soil and the capacity of the 151H is about 45 g. Record this weight as *original sample weight* or *hydrometer weight.* Add 125-mL of sodium hexametaphosphate solution, seal with a rubber stopper, and let sit for at least 16 hours at room temperature.

10.3.3.1.1 If a limited amount of wet sample is available, refer to Section 2.0.

10.3.3.2 Homogenize the contents of the glass cylinder. Bring up to 1000-mL mark with RO water. Cap with rubber stopper. Note: The water used for volumizing should be at room temperature.

10.3.3.3 At a constant room temperature, mix the contents of the capped cylinder for up to one minute by inverting until all contents are suspended (residue on bottom of cylinder has loosened completely). At this point, immediately begin timing analysis. *Note: Small variations of temperature do not introduce differences that are of practical significance however, loss of water does and must be avoided.*

10.3.3.4 Slowly immerse the 151H hydrometer into the sample 30 seconds prior to the time the initial reading should be taken in order to assure that the hydrometer stabilizes before the reading. Take readings at 2, 5, 15, 30, 60, 240, and 1440 minutes (total elapsed time). Cover the cylinder and hydrometer with an inverted plastic funnel to inhibit evaporation beyond the 30 minute reading.

10.3.3.5 Follow steps 10.3.3.1 through 10.3.3.4 for each sample.

10.4 Continuing Calibration

Not applicable.

10.5 Preventive Maintenance

Not applicable.

11. Data Evaluation, Calculations and Reporting

11.1 Particle size with/without hydrometer is calculated by entering raw data, recorded in the laboratory notebook, into the LIMS. Using the particle percent retained, calculated in LIMS, the analyst calculates the particle percent passing for each sample. This is calculated as follows:

Percent Passing (%) = 100 – Percent Retained (%)

The analyst transfers this data, along with sample identification, into*, GEOSYSTEM.* This program performs all necessary calculations. It determines the particle size distribution ratios and illustrates them graphically.

11.2 To report *Particle Size With/Without Hydrometer*, first go to the LIMS and “batch” all samples in this analytical batch. Then enter the results from the laboratory notebook into *GEOSYSTEM.* Finally, use the results to create a graph in *GEOSYSTEM.*

11.2.1 Enter “*GEOSYSTEM*”

11.2.2 To create a New Project

11.5.2.1 Type name of project as LIMS Workgroup number in bottom left hand box that reads "Name"

11.5.2.2 Click "Open." A box will come up that reads "The Project (name of the project just created) can't be found. Would you like to create it?" Click "Create." This will open a new window

11.5.3 To Set up Project Data

11.5.3.1 Click "Project Data" in left hand column

11.5.3.2 Type full client name, project name, and project number (WG number)

11.5.4 To Set up Samples

11.5.4.1 Click first button on left hand side of the screen (green "+") to create a new source. Each new source is a sample - create one for each sample in the ETR.

11.5.4.2 Type Sample ID on the "Source" line in the right hand screen and Lab ID where it says "Sample No." in the first card.

11.5.4.3 Repeat for each sample.

11.5.5 To Enter Data

11.5.5.1 Click link for USCS in sample card

11.5.5.2 Click link for Grain Size at the top of the screen

11.5.5.3 Click tab for Sieve Test

11.5.5.4 Choose a 'sieve nest' from the drop down list, (*i.e.*, Army Corps Sieve). If your group of sieves does not match any of these, create a new nest.

11.5.5.4.1 Type sieve sizes in Sieve opening size column, using enter to move between columns, and click "save current sieve nest" at the top of the screen - it will ask you to name the nest.

11.5.5.5 For samples with hydrometer, enter sample weight into box for "Dry sample and tare weight." Type 0 in box for "Tare weight." (For samples with sieve only, skip to 15.5.5.14)

11.5.5.6 Type sample weight into "Dry Sample and Tare" column in line 1 and 0 in "Tare" column.

11.5.5.7 Enter "Weight retained" (our Sieve minus sample Weight as read from tared scale) and enter 0 for "Sieve weight" for each sieve. Use enter to move from column to column. Do not enter pan. Click disk icon to save.

11.5.5.8 Click hydrometer test tab. (For samples with sieve only, skip to 15.5.5.14)

11.5.5.9 Hydrometer sample box: Enter sample weight as total dry weight. Specific gravity = 2.65

11.5.5.10 Corrections box: Hydrometer correction @ 20°C = hydrometer correction factor, Meniscus correction = 0

11.5.5.11 Hydrometer box: Select 151H

11.5.5.12 Enter hydrometer data starting with 2 minutes; enter reading as whole numbers (i.e. 14 rather than 1.014). Use the enter key to move through columns; do not need to reenter temperature with every hydrometer reading.

11.5.5.13 Click disk icon to save.

11.5.5.14 Click on reports tab.

11.5.5.15 Select "Similar to the Corps of Engineers report form (GSGEOSYS)" from the drop down box.

11.5.5.16 Click printer icon.

11.5.5.17 Click "Test 1" tab from right side of the screen.

11.5.5.18 Click disk for save to LIMS. Add analyst initials to end of file name with a comma and no space to have the analyst initials appear correctly on the LIMS report.

11.6 Forward all hand recorded raw data from the laboratory notebook, Excel spreadsheets, LIMS batch printouts, and *GEOSYSTEM* graphs for secondary review.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9 outlines sample batch QC and acceptance criteria. Corrective action for non-compliant data is discussed in Section 9. If a situation arises (such as loss of sample, or the inability for re-extraction or re-analysis) that requires additional action, the Project Manager and QA Officer will be immediately notified and will contact the client for discussion and/or instruction for any further action. The situation and the agreed upon actions will be discussed in the project narrative that accompanies the data package.

13. Method Performance

13.1 Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

These studies are not applicable to this method.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739IDC/DOC Generation

16. Attachments

None.

PCBs

By Capillary Column Gas Chromatography

Reference Methods: Method 8082A SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, 2007.

Quality Control Requirements and Performance Standards for Analysis of Polychlorinated Biphenyls (PCBs) by Gas Chromatography (GC) in Support of Response Action under the Massachusetts Contingency Plan (MCP), Revision No.2, February 15, 2024.

State of Connecticut, Department of Environmental Protection, RRCP, Version 3.0, May 2024.

1. Scope and Application

Method 8082A is used to determine the concentrations of Polychlorinated Biphenyls (PCBs) as Aroclors in extracts from solid, oil and liquid matrices. This SOP details the analysis for PCBs using fused-silica, open-tubular, capillary columns with electron capture detectors (ECD).

Matrices: Extracts from solid, oil and liquid matrices.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Regulatory Parameter List: The standard compounds listed below are determined by this method.

Parameter	CAS#
Aroclor 1016	12674-11-2
Aroclor 1221	11104-28-2
Aroclor 1232	11141-16-5
Aroclor 1242	53469-21-9
Aroclor 1248	12672-29-6
Aroclor 1254	11097-69-1
Aroclor 1260	11096-82-5
* Aroclor 1262	37324-23-5
*Aroclor 1268	11100-14-4

*Note – not certified by NJ for the oil matrix

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.
This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph (GC) and in the interpretation of gas chromatograms. Each analyst must demonstrate

the ability to generate acceptable results with this method by performing an initial demonstration of capability (see section 13.2).

2. Summary of Method

A measured volume or weight of sample (100mL for liquids, 1g for oil and up to 15g for solids) is extracted using the appropriate matrix-specific sample extraction technique.

Liquid samples are extracted at neutral pH with methylene chloride using Method 3510C (separatory funnel), or other appropriate technique. See extraction SOP for details.

Solid samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet), or other appropriate technique. Solid samples may also be extracted with hexane: acetone (1:1) using Method 3546 (microwave). See extraction SOP for details.

Wipe samples are extracted with methylene chloride: acetone (1:1) using Method 3540C (Soxhlet) or other appropriate technique. See extraction SOP for details.

Oil samples are diluted with hexane following the procedure outlined in the extraction SOP.

Sulfuric acid cleanup (Method 3665A), Copper cleanup (Method 3660B) and Silica Gel cleanup (Method 3630) are utilized for PCB extracts. See extraction SOP for details.

After cleanup, the extract is analyzed by injecting 1µL into a gas chromatograph equipped with narrow- or wide-bore fused silica capillary columns and electron capture (GC/ECD) detectors.

2.1 Method Modifications from Reference

Not applicable.

3. Reporting Limits

The reporting limits for this method as outlined is as follows:

- **Aqueous samples**: 0.04 ug/L / Aroclor based on a 100mL extraction
- **Soil Samples**:
  - 33.3 ug/kg / Aroclor based on 15g extraction
  - 50.0 ug/kg / Aroclor based on 10g extraction
  - 100.0 ug/kg / Aroclor based on 5g extraction
  - 250.0 ug/kg / Aroclor based on 2g extraction
- **Oil Samples**: 5 mg/kg based on 1g extraction
- **Solid of Difficult Matrices** (i.e Caulking, Concrete, etc. are logged using the Alpha Low Level 8082 products): based on a 15g extraction
  - Aroclors 1016, 1221, 1232, 1242, 1254: 20 ug/kg
  - Aroclors 1248, 1260: 13.3 ug/kg
  - Aroclors 1262, 1268: 6.67 ug/kg

4. Interferences

4.1 Instrumental

4.1.1 Only high purity gases are used in the GC system to eliminate this source of possible contamination. Both the Hydrogen (carrier gas) and Nitrogen (detector make-up gas) are certified by the gas supplier.

4.1.2 Preventive instrument maintenance is performed routinely, and whenever highly contaminated extracts are analyzed that could result in chromatographic interferences or result in degradation of system performance. Section 10.6 describes the maintenance steps.

4.1.3 Glassware must be scrupulously cleaned. This procedure is detailed in the Organic Extraction Cleaning and Handling SOP/1953. Store dry glassware in a clean environment.

4.2 Parameters

4.2.1 All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free. All these materials are routinely determined to be free of interferences by analysis of extraction blanks with every extraction batch performed.

4.2.2 Certain compounds (i.e. phthalates) can be extracted from the sample matrix and be detected by the ECD that could possibly result in false positive results or complicate the data interpretation. The use of the cleanup procedures detailed in the extraction SOP minimizes these possible interferences. Analyst experience is also crucial in making compound determinations.

4.2.3 Interferences co-extracted from the samples will vary considerably from waste to waste. While a general cleanup technique is referenced or provided as part of the method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. PCBs have been tentatively classified as known or suspected human or mammalian carcinogens. Additional references to laboratory safety are available in the Chemical Hygiene Plan.
All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammable cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 100mL amber glass jars with Teflon-lined lids. Solid samples are collected in one 250mL wide-mouth glass jar with a Teflon-lined lid. Oil samples are collected in a glass jar with Teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ± 2° C. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ± 2° C. Preservation is not required for oil samples.

6.3 Sample Handling

- TCLP/SPLP tumbled extracts, NJ DKQP aqueous samples must be extracted within 7 days of sample collection.
- NJ DKQP soil/solid and oil samples within 14 days of collection (NJ DKQP allows 365 days for solids if frozen).
- All other samples, both aqueous and solids, have a 365-day hold time.
- Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph, Agilent 6890, 7890: An analytical system completed with gas chromatograph configured for split-splitless injection and all required accessories including syringes, analytical columns, gases, electron capture detectors (ECD), and data system.

7.2 GC Columns: Alpha utilizes dual-column analyses. The dual-column approach involves a single injection that is split between two columns that are mounted in a single gas chromatograph. Typical column pair used is listed below. Other columns may be used as long as method performance criteria can be met.

Column pair:

RTX-CLP: Cat. #11141 from Restek or equivalent; 30m, 0.32mm, 0.32µm

RTX-CLPII Cat. #11324 from Restek or equivalent; 30m, 0.32mm, 0.25µm

7.3 Guard Column: Cat. #10027 from Restek or equivalent; 5m, 0.32mm

7.4 Class "A" Volumetric Flasks: 10mL and 25mL (and other volumes), for standards preparation

7.5 Microsyringes: 10 µL – 1000 µL

7.6 Gooseneck splitless injecton liner, Cat #20799-214.5 from Restek or equivalent

7.7 Universal "Y" Press-tight tee split: Cat. #20406 from Restek or equivalent / **Siltek MXT Connector:** Cat. #21388 from Restek or equivalent

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.
NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4 ± 2° C in Teflon(R)-sealed containers in the dark. When a Lot of standards is prepared, aliquots of that Lot are stored in individual small vials. All stock standard solutions must be replaced after one year or sooner if routine QC tests indicate a problem. All other standard solutions must be replaced after six months or sooner if routine QC indicates a problem.

8.1 n-Hexane: Pesticide quality or equivalent.

8.2 Acetone: Pesticide quality or equivalent.

8.3 Organic-free Reagent Water: All references to water in this method refer to organic-free reagent water from Alpha's RO water treatment system.

8.4 Stock Standard Solutions: All stock standard solutions are purchased from commercial vendors as ampullated certified solutions. When an ampullated stock solution is opened, it is transferred to a labeled amber screw-cap vial. The expiration date of the stock solution is either the vendor specified expiration date, or 1 year from the date the ampule was opened, whichever is sooner.

8.5 Calibration Standards: Calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Calibration standards expire 6 months from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the calibration standard. Calibrations are performed at the 6 concentration levels listed in Table 1. The list of ampullated calibration standards is obtain from **Agilent**:

- Aroclor 1016, Cat. #PP-282, at 100ug/mL
- Aroclor 1260, Cat. #PP-361, at 100ug/mL
- Aroclor 1262, Cat. #PP-371, at 100ug/mL
- Aroclor 1268, Cat. #PP-382, at 100ug/mL
- Aroclor 1221, Cat. #PP-292, at 100ug/mL
- Aroclor 1232, Cat. #PP-302, at 100ug/mL

- Aroclor 1242, Cat. #PP-312, at 100ug/mL
- Aroclor 1248, Cat. #PP-342, at 100ug/mL
- Aroclor 1254, Cat. #PP-351, at 100ug/mL

8.5.1 Preparation of Initial Calibration Standards:

8.5.1.1 SOIL ICAL:

- **1660 L7**: (mix of Aroclors 1016 and 1260) is prepared by diluting
  - 5mL 1016 Standard (Agilent, Cat. #PP-282)
  - 5mL 1260 Standard (Agilent, Cat. #PP-361)
  - 6.4mL of PCB ICAL Surrogate Stock

  to 25ml of Hexane to achieve concentration of
  - 1016 and 1260 at 20ug/mL
  - TCMX at 1.28ug/mL and DCB at 2.56ug/mL.

- **2154 L7:** (mix of Aroclors 1221 and 1254) is prepared by diluting
  - 5mL 1221 Standard (Agilent, Cat. #PP-292)
  - 5mL 1254 Standard (Agilent, Cat. #PP-351)

  to 25ml of Hexane to achieve concentration of
  - 1221 and 1254 at 20ug/mL
- **4268 L7** (mix of Aroclors 1242 and 1268), **3262 L7** (mix of Aroclors 1232 and 1262) and **1248 L7;** all are prepared from their respective Stock Standards according to the formula used for L7 of 2154.
- Levels 1-6 are prepared by serial dilutions and a formula with volumes needed can be found in Table 2.

8.5.1.2 RVT ICAL:

- Prepare 2 Intermediate ICAL Standards by diluting 1016/1260 L7 of Soil ICAL:
  - **Stock#1** (10ug/mL): 500uL of 1660 L7 + 500uL of Hexane
  - **Stock#2** (0.5ug/mL): 25uL of 1660 L7 + 975uL of Hexane
- Use the VYPER programs: **pcb_rvt_L1-L6** and **pcb_rvt_L7-L10** to vial 1660 ICAL according to the formula and to achieve the concentrations provided in Table 3.
- Prepare one-point cal standards for all other Aroclors by diluting respective L7 stock solutions by using the VYPER program:

  12.5uL of L7 + 987.5uL of Hexane
  pcb_rvt_1point_set

8.6 Second Source Standards: (ICV) Initial Calibration Verification standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. Initial Calibration Verification standards expire 6 months from the date of preparation, or on the

earliest expiration date of any of the stock solutions used to prepare the standard. The list of ampullated standards is obtain from **Accustandard**:

- Aroclor 1016, Cat. #C-216S-H-10X, at 1000ug/ml
- Aroclor 1260, Cat. #C-260S-H-10X, at 1000ug/ml
- Aroclor 1262, Cat. #C-262S-H-10X, at 1000ug/ml
- Aroclor 1268, Cat. #C-268S-H-10X, at 1000ug/ml
- Aroclor 1221, Cat. #C-221S-H-10X, at 1000ug/ml
- Aroclor 1232, Cat. #C-232S-H-10X, at 1000ug/ml
- Aroclor 1242, Cat. #C-242S-H-10X, at 1000ug/ml
- Aroclor 1248, Cat. #C-248S-H-10X, at 1000ug/ml
- Aroclor 1254, Cat. #C-254S-H-10X, at 1000ug/ml

8.6.1 Preparation of ICV Standards:

- **1660 ICV:** is prepared by diluting
  - 50uL 1016 Standard (AccuStandard, Cat. #C-216S-H-10X)
  - 50uL 1260 Standard (AccuStandard, Cat. #C-260S-H-10X)
  - 320uL of PCB CCAL Surrogate Stock

  to 20ml of Hexane to achieve concentration of
  - 1016 and 1260 at 2.5ug/mL
  - TCMX at 0.16ug/mL and DCB at 0.32ug/mL

- **2154, 4268, 3262 and 1248 ICVs** are prepared from their respective Stock Standards according to the formula used for 1660 ICV.

- Prepare **RVT ICVs** for all Aroclor standard solutions by diluting respective ICV stocks by using VYPER's pcb_rvt_icv program:
  - 100uL of ICV to 900uL of Hexane, achieving a concentration of
  - 1016 and 1260 at 0.25ug/mL
  - TCMX at 0.016ug/mL and DCB at 0.032ug/mL

8.7 Internal Standard Solution: 1-Bromo-2-nitrobenzene (Ultra, Cat. #PPS-351) is used as the internal standard and is added to all single-component calibration standards and sample extracts to achieve a concentration of 0.25µg/mL. For RVT, this solution is diluted 10X more, achieving a concentration of 0.025µg/mL.

8.8 Surrogate Standards: Tetrachloro-m-xylene (TCMX) and Decachlorobiphenyl (DCB) are used as surrogates for Aroclor analysis. They are added to the calibration standards at the concentrations listed in Table 1, Continuing Calibration Standards and are spiked into all samples and QC samples prior to extraction.

- **ICAL Surrogates Stock:** is prepared by diluting 500uL of Pesticides Surrogates Standard Spiking Solution (Ultra, Cat. #ISM-320-1) and 500uL of Decachlorobiphenyl (Accustandard, Cat. #CLP-032-R-01) to 20mL of Hexane to achieve concentration of TCMX at 5ug/mL and DCB at 10ug/mL.

- **CCAL Surrogates Stock:** is prepared by diluting 1mL of TCMX&DCB (Accustandard, Cat. #CLP-032-R) and 1mL of Decachlorobiphenyl (Accustandard, Cat. #CLP-032-R-01) to 20mL of Hexane to achieve concentration of TCMX at 10ug/mL and DCB at 20ug/mL.
- **Extraction Surrogates Stock:** is prepared by diluting 10mL of TCMX&DCB (Accustandard, Cat. #CLP-032-R) to 1000mL of Acetone to achieve concentration of TCMX and DCB at 2ug/mL.
- **RVT Extraction Surrogates Stock**: is prepared by diluting 1.25mL of TCMX&DCB (Accustandard, Cat. #CLP-032-R) to 500mL of Acetone to achieve concentration of TCMX and DCB at 0.5ug/mL.
- **PCB Caulking Surrogates Stock:** is prepared by diluting 10mL of TCMX&DCB (Accustandard, Cat. #CLP-032-R) to 100mL of Acetone to achieve concentration of TCMX and DCB at 20ug/mL.
- **PCB Oil Surrogate Stock:** is prepared by diluting 5mL of TCMX&DCB (Accustandard, Cat. #CLP-032-R) to 500mL of Hexane to achieve concentration of TCMX and DCB at 2.0ug/mL.

8.9 LCS/MS Spiking Solutions:

- **PCB Matrix Spike:** The LCS/MS spiking solution is prepared by diluting 6.25mL of Arochlor 1016/1260 (Restek, Cat. #32039) to 500mL of Acetone to achieve concentration of Arochlor 1016/1260 at 12.5ug/ml. For **RVT**, 1.25mL of the stock solution is diluted to 500mL of Acetone to achieve a concentration of Aroclor 1016/1260 at 2.5 ug/mL.

- **PCB Caulking Matrix Spike:** The LCS/MS spiking solution is prepared by diluting 6.25mL of Arochlor 1016/1260 (Restek, Cat. #32039) to 50mL of Acetone to achieve concentration of Arochlor 1016/1260 at 125ug/mL.

- **PCB Oil Matrix Spike:** The LCS/MS spiking solution is prepared by diluting 2.5mL of Arochlor 1016/1260 (Restek, Cat. #32039) to 200mL of Hexane to achieve concentration of Arochlor 1016/1260 at 12.5ug/mL.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A Method Blank is an aliquot of a clean reference matrix (reagent water for water samples, Ottawa sand for soil/sediment samples, or PCB free transformer oil for oil samples) that is carried through the entire analytical procedure. Extraction blanks are performed with each extraction batch of 20 or less samples, according to the extraction SOPs. The extraction blank must not contain any of the reportable analytes above the reporting limit. If any reportable analytes are detected in the blank, the entire extraction batch is suspect and re-extraction of all associated samples is required, unless the associated samples are non-detect or concentration of the analyte in the samples is 10 times greater than the concentration of this analyte in the blank. The surrogate recoveries must also be within the acceptance criteria listed in Table 4. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary. Actions associated with Method Blank failures are listed in Table 6.

9.2 Laboratory Control Sample (LCS)

A Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD) pair is extracted with each analytical batch. The LCS/LCSD consist of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. For Aroclor analysis, the LCS/LCSD are spiked with a mixture of Aroclor 1016 and 1260 (1660) at the levels listed in Table 5. The recovery acceptance criteria are listed in Table 4. If any recovery criteria are not met, the extract may be re-analyzed. If the criteria are still not met, the **entire batch is re-extracted**, unless the recoveries are high, and the associated samples are non-detect. If this is not possible, due to insufficient sample or holding time exceedances, the analyst must narrate the failure in the LIMS for inclusion in the client report. Actions associated with LCS/LCSD failures are listed in Table 6.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.7.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike/ Matrix Spike Duplicate (MS/MSD)

Matrix Spike and Matrix Spike Duplicate are field samples spiked with known target analyte(s) at the levels listed in Table 5. Upon client request, an MS/MSD pair is extracted and analyzed with each batch of 20 or less samples. MS samples serve as a measure of extraction accuracy, by allowing the comparison of the found amount(s) of target analyte(s) with the spiked amount(s). An MS/MSD set also allows for the calculation of the extraction precision, by comparing the results of the two samples. The recovery acceptance criteria are listed in Table 4. If the recovery criteria are not met, but are met in the LCS, the failure may be attributed to sample matrix effects. Actions associated with MS/MSD failures are listed in Table 6.

For samples with a state of origin of New Jersey, a MS must be extracted for every twenty samples within a 24hr period.

9.6 Laboratory Duplicate (DUP)

Duplicates are laboratory selected replicate samples, prepared by taking an additional sample aliquot of a sample. Upon client request, a DUP is extracted and analyzed with batch of 20 or

less samples. Duplicates serve as a measure of the extraction precision, by comparing the results of the sample and duplicate. The relative percent difference (RPD) acceptance criteria are listed in Table 4. If the RPD criteria are not met, the failure may be attributed to matrix effect. Actions associated with DUP failures are listed in Table 6.

For samples with a state of origin of New Jersey, a DUP must be extracted for every twenty samples within a 24hr period.

9.7 Surrogates

All extracted samples and associated QC are spiked with surrogates at the levels listed in Table 5. The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate control limits listed in Table 4. If the surrogate limits are not met, the extract may be reanalyzed to determine if the failure was due to an instrument problem. If the criteria are still not met, the affected samples must be re-extracted to confirm that the failure was due to sample matrix, unless the surrogate recovery is high, and the associated sample is non-detect. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

9.8 Method Sequence

Typical Initial calibration (each level and ICV to identified with the standard lot number)

1. Prime
2. Blank
3. Standard Level 1
4. Standard Level 2
5. Standard Level 3
6. Standard Level 4
7. Standard Level 5
8. Standard Level 6
9. Standard Level 7
10. Initial Calibration Verification Standard (ICV)

Repeat steps 3 – 9 as needed for each Aroclor necessary for calibration.

NOTE: If multiple calibration mixtures are analyzed, it is acceptable to analyze appropriate ICVs after all calibration standards have been injected.

Typical Daily Sequence

1. 1660 Continuing Calibration Standard **(identified with the standard lot number)**
2. Extraction Blank

3. Laboratory Control Sample / Laboratory Control Sample Duplicate
4. Matrix Spike / Matrix Spike Duplicate (if requested by Client)
5. Duplicate (if included with batch QC)
6. Samples - **up to 20 field samples**
7. Repeat 1 – 6 as needed.

10. Procedure

10.1 Equipment Set-up

10.1.1 GC Conditions:

The dual-column / dual-detector approach involves the use of the columns listed in section 7.2. The columns are connected to an injection tee or dual injection GC, and separate electron capture detectors. Alpha typical GC conditions are listed below but may be altered as long as method performance criteria are met.

	INSTRUMENT CONDITIONS
Injector temperature	250^{0}C
Injector mode	Split 10:1
Split Flow	60.1 mL/min
Total Flow	78.2 mL/min
Detector temperature	350^{0}C
Carrier gas	Hydrogen
Carrier flow	20mL/min
Carrier mode	Constant flow
Makeup gas	Nitrogen
Total detector flow	60mL/min
Injection Volume	1µL

	OVEN TEMP PROGRAM
Temperature 1	150 ^{0}C
Time 1	0 min
Ramp 1	45^{0}C/minute
Temperature 2	230^{0}C
Time 2	0 min
Ramp 2	30^{0}C/minute
Temperature 3	330^{0}C
Time 3	1 min

10.2 Initial Calibration

10.2.1 Prepare calibration standards using the standards listed in Section 8.5 to achieve the concentrations from Table 1.

For Analysis of Aroclors which use an **Average Response Factor model (AVG)** the minimum requirement is to perform an initial multi-point calibration for a mixture of 1016/1260 and to use a **one-point initial calibration** to determine the calibration factor and pattern recognition for the remaining Aroclors (TNI standard 1.7.1.1.m). The concentrations of the other Aroclors should generally correspond to the mid-point of the linear range of the detector, but lower concentrations may be employed.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in section 10.1.1. The same operating conditions are used for calibrations and sample analyses. Create the analytical sequence using the Agilent Chemstation data acquisition software. Record the

calibration standard, unique lot number (PP#) and analyst's initials in the analytical sequence list.

10.2.3 A 1µL injection volume of each calibration standard is typically used. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume must be used for all standards and samples.

10.2.4 Column adsorption may be a problem when the GC has not been used for a day or more or after system maintenance. The GC column may be primed (or deactivated) by injecting a PCB standard mixture approximately 20 times more concentrated than the mid-concentration standard. Inject this standard mixture prior to beginning the initial calibration or calibration verification.

Alternately, the system may be primed by baking at the final analytical temperature for approximately 30 minutes.

Several analytes may be observed in the injection just following system priming. Always run an instrument blank after system priming.

10.2.5 **Calibration Factor:** Internal Standard Calibration techniques are employed in this method.

10.2.7.1 Internal Standard Procedure. In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formula listed in Quality Manual.

Alternatively, standards of the other seven Aroclors are necessary for pattern recognition. When employing the traditional model of a linear calibration through the origin, these standards are also used to determine a single-point calibration factor for each Aroclor, assuming that the Aroclor 1660 mixture has been used to describe the detector response.

The standards for these seven Aroclors should be analyzed before the analysis of any samples with hits above the RL. For example, an Aroclor 1254 standard should be analyzed before a sample with a hit of Aroclor 1254.

10.2.6 Initial Calibration Criteria

- If the **RSD for an analyte is < 20%**, then the response of the instrument for this compound is considered linear over the range and the mean calibration factor can be used to quantitate sample results.
- If the **RSD for any analyte is > 20%**, then linearity through the origin cannot be assumed. The mean response factor cannot be used for quantitation. An alternative calculation may be done using **linear regression** or **quadratic regression** (minimum of six ICAL points are needed and regression must be weighted inversely proportional to concentration) if the correlation coefficient is **>0.990**. If both quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated, and a new calibration curve must be analyzed. If quadratic regression is used for calibration, this must be noted in the laboratory narrative.

- **A minimum of 3 peaks must be chosen** for each Aroclor and preferably 5 peaks (11.4.6.1 8082A).

- **The minimum number of calibration standards** based on the type of calibration curve is listed below:

Type of Calibration Curve	Minimum Number of Calibration Standards
Linear Fit/Avg Response	5
Quadratic Fit	6

- **Removal and replacement of the calibration standards:**
  - The Analyst may remove individual analyte from the lowest (cannot remove RL level) and or the highest calibration levels of the curve. Removal of the individual analyte of interior levels is not permitted.
  - The Analyst may remove an entire single standard calibration level from the interior of the calibration curve when the instrument response demonstrates that the standard was not properly introduced to the instrument, or an incorrect standard was analyzed. The Analyst who chooses to remove a calibration standard from the interior of the calibration shall remove that particular standard calibration level for all analytes.
  - The Analyst may replace a calibration standard provided that:
    - the replacement standard will be analyzed within twenty-four (24) hours of the original calibration standard analysis for that particular calibration level;
    - all analytes of the replacement calibration standard must be replaced if a standard within the interior of the calibration is replaced;
    - the laboratory limits the replacement of calibration standards to one calibration standard concentration.

- **Percent Relative Error (%RE)**: For linear and quadratic fit compounds, the %RE must be calculated for the standard at or near the midpoint and at the lowest level of the initial calibration. The %RE between the calculated and expected concentration of an analyte must be **≤ 30%**, otherwise recalibration is necessary.

10.2.7 Initial Calibration Verification

An initial calibration verification standard must be run immediately after each initial calibration, near the midpoint of the curve. The standard must be prepared using a second source that is different than the source used for the initial calibration. (Standards listed in Section 8.6). The **%D** has to be within **20%** when compared to the mean response factor from the initial calibration.

If recovery is **outside the 80-120% criteria**, the following steps should be taken:

- Analyze the replacement standard. The cause of the failure of the first calibration verification result should be documented.
- If the reanalysis still didn't achieve the criteria, the source of the problem must be investigated, and instrument must be recalibrated.

10.2.8 Retention Time Window

10.2.8.1 The retention time window used for the identification of target analytes is ± 0.07 minutes. These criteria have been adopted from the EPA CLP Statement of Work (OLM04.2). It has been found that these limits work well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positives. Windows that are calculated using the procedure recommended in Method 8000 tend to be very narrow, creating the risk of false negative results.

10.2.8.2 The windows listed above are used as guidance; however, the experience of the analyst weighs heavily in the interpretation of the chromatograms. For example, it has been observed that certain oil matrices can cause the retention times to shift more dramatically.

10.3 Sample Processing

The determination of PCB Aroclors is accomplished by comparing the sample chromatogram to that of the most similar Aroclor standard. The use of PCB overlays is extremely helpful, either by using hardcopies of chromatograms or by utilizing the Enviroquant software. A choice must be made as to which Aroclor is most similar and whether that standard is truly representative of the PCB in the sample. Both retention time and pattern are important when determining PCBs in a sample.

Samples that contained weathered PCB present special analytical challenges. Weathering could alter the Aroclor pattern to the extent that different peaks have to be selected for quantitation. Samples that contained more than one Aroclor present similar problems. For these samples, the Analyst may have to consider selecting the earlier eluting peaks for the lower boiling Aroclor and selecting the later eluting peaks for the higher boiling Aroclors to minimize overlapping peaks.

Minimum of 3 peaks must be chosen for each Aroclor. In these instances, the Analyst may need request the assistance of someone with more expertise in determining the presence of PCB Aroclor.

If compound identification or quantitation is precluded due to interference (e.g., broad, rounded peaks or ill-defined baselines are present) cleanup of the extract may be needed. If instrument problems are suspected, rerun the extract on another instrument to determine if the problem results from analytical hardware or the sample matrix. Refer to the extraction SOPs for the procedures to be followed in sample cleanup.

- The laboratory must report the ***HIGHER*** of the two results unless obvious interference is present on of the columns.

- The **Relative Percent Difference (RPD)** should be **≤ 40%.** If the RPD exceeds 40, this will be denoted with a P flag. If the RPD exceedance is due to interference, the lower of the dual column values can be reported with I and P flags.

If any peaks are above the calibration, a dilution must be done. The dilution reported is the least dilute level at which the peak area is within the calibration range.

10.4 Continuing Calibration

10.4.1 Verify calibration each **12-hours** shift by injecting calibration verification standards prior to conducting any sample analyses. A calibration standard must also be injected at intervals of not less than **once every twenty filed samples**.

A bracketing CCV is not required with the use of internal standard calibration (Method 8082A 11.6.8)

For Aroclor analysis, the calibration verification standard should be a mixture of Aroclor 1016 and 1260 (1660). The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition (Method 8082A 11.6.2). However, if the one-point calibration is used for the seven other Aroclor, a calibration standard must be analyzed before the sample for any hits.

10.4.2 The **Response Factor** (for internal standard compounds) for each analyte to be quantitated must not exceed a **± 20% difference** when compared to the initial calibration curve The Enviroquant data processing software automatically calculates the **%D** for all analytes.

10.5 Internal Standard

The use of internal standard calibration does not require that all sample results be bracketed with CCV standard. However, when internal standard calibration is used, the retention times of internal standards and the area response of internal standards should be checked for each analysis (10.4.3).

10.5.1 **IS in CCAL** – The measured area of the internal standard must be no more than ± 50% different from the average area calculated during initial calibration **(-50 to 150%).**

10.5.2 **IS in samples** - The measured area of the internal standard must be no more than -50% to +100% different from the area calculated from opening CCV **(-50 to 200%)**

Retention Time shift >30 seconds from the retention time of the most recent calibration standard are cause for concern and must be investigated.

10.6 Preventive Maintenance

10.6.1 **Preventive Maintenance:** Routine preventive maintenance is performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and replacement of injector seals.

10.6.2 Other Maintenance: ECD detectors may become contaminated, requiring bake out at elevated temperatures, (no greater than 375°C) or repair by the manufacturer.

11. Data Evaluation, Calculations and Reporting

11.1 Quantitation of Aroclors

Per Method 8082A, quantitation is based on the use of a minimum of 3 of the major peaks present in the analyte, although the use of 5 of the major peaks is recommended. Each of these peaks is individually calibrated with a **minimum of five calibration points** based on average response factors. The %RSD must meet the criteria of ≤20% for the ICAL. The five major peaks are calculated as described below. After individual calculation meets criteria, the average of the peaks selected for quantitation is used to determine the final concentration.

11.1.1 Aqueous samples

$$\text{Concentration } (\mu g/L) = \frac{C \times DF \times Vf \times 1000}{Vo}$$

where:
C = Extract concentration (µg/mL), **NOTE**: ng on column = ng/injection volume = ng/uL = ug/mL
DF = Dilution factor
Vf = Final extract volume (mL)
Vo = Sample volume (mL)

11.1.2 Soil/sediment samples

$$\text{Concentration } (\mu g/Kg, \text{ dry weight}) = \frac{C \times DF \times Vf \times 1000}{W\ (gm)} \div \%S$$

where:
C = Extract concentration (µg/mL), **NOTE**: ng on column = ng/injection volume = ng/uL = ug/mL
DF = Dilution factor
Vf = Final extract volume (mL)
W = Weight of the sample extracted (10g for high, 30g for low)
%S = Percent solids, as a decimal value

11.1.3 Reporting Results

11.1.3.1 After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

11.1.3.2 Reporting Results for PCBs in Caulking Samples

If in the screen sample Aroclor concentration as calculated above is **≥20000ppm**, the Client is contacted by a Customer Service Representative and these results are sent to the LIMS and reported to the Client.

If the sample concentration as calculated above for any Aroclor is **<20000ppm**, the sample is sent for re-extraction by Method 3540C (Alpha SOP/1954).

11.1.3.3 Summation Rules

"TOTAL" concentrations are calculated for **ALL samples and Quality Control Samples** (i.e. LCS, MS, DUP, BLK).

TOTAL = sum of "reportable" Aroclors

Reportable- all Aroclors reported for associated project.

For dual-column analysis, Total is reported as part of column "A" data, unless all individuals are reported from "B" column. "Total" is calculated based on the associated "Report List". See Work Instruction #14335 for details.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and/or improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written into the LIMS by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Pace SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Pace SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Pace's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

Table 1: STANDARD SOLUTIONS

Table 2: PCB ICAL PREPARATION

Table 3: PCB RVT ICAL PREPARATION

Table 4: QC ACCEPTANCE CRITERIA

Table 5: SPIKING LEVEL

Table 6: QC FAILURE ACTION PLAN

TABLE 1

STANDARD SOLUTIONS – Suggested Concentrations

STANDARD SOLUTIONS	Stock Solution (ug/mL)	Level 1 (ug/mL)	Level 2 (ug/mL)	Level 3 (ug/mL)	Level 4 (ug/mL)	Level 5 (ug/mL)	Level 6 (ug/mL)	Level 7 (ug/mL)
AROCLORS								
1016,1221,1232,1242, 1248,1254, 1260,1262,1268	100	0.1	0.5	1	2.5	5	10	20
Aroclors 1221, 1232, 1242, 1254, 1262, 1268	100	0.1	0.5	1	2.5	5	10	20
Internal Standard								
1-Bromo-2-Nitrobenzene	5000	0.25	0.25	0.25	0.25	0.25	0.25	0.25
Surrogates:								
Tetrachloro-m-xylene	2.0	0.0064	0.032	0.064	0.16	0.32	0.64	1.28
Decachlorobiphenyl	2.0	0.0126	0.064	0.128	0.32	0.64	1.28	2.56

TABLE 2

PCB SOIL ICAL PREPARATION:

Aroclors: 1660, 2154, 4268, 3262, 1248				
Level	**Conc [ug/L]**	**Spike**	**Spike [uL]**	**Hexane [uL]**
6	10000	L7	5000	10000
5	5000	L6	500	500
4	2500	L6	250	750
3	1000	L6	100	900
2	500	L6	50	950
1	100	L6	10	990

TABLE 3

PCB RVT ICAL PREPARATION:

Level	Aroclor Conc	TCMX/DCB Conc	Stock#	VYPER Program	Stock Vol used	FV
	ug/L	ug/L			uL	mL
L1	4	0.256/0.512	2	**pcb_rvt_L1-L6**	8	1
L2	10	0.64/1.28			20	
L3	20	1.28/2.56			40	
L4	30	1.92/3.84			60	
L5	40	2.56/5.12			80	
L6	50	3.20/6.40			100	
L7	100	6.40/12.8	1	**pcb_rvt_L7-L10**	10	
L8	250	16/32			25	
L9	500	32/64			50	
L10	1000	64/128			100	

TABLE 4

QC ACCEPTANCE CRITERIA

Aqueous, Soils, Oils		
Surrogate	Lower Control Limit	Upper Control Limit
2,4,5,6-Tetrachloro-m-xylene	30%	150%
Decachlorobiphenyl		

	Aqueous, Soils % Recovery	**RPD%**	
		Waters	**Soils**
MS/MSD LCS/LCSD	40-140%	50%	50%
		20% (NJ/CT/MCP)	30% (NJ/CT/MCP)

TABLE 5

SPIKING LEVEL

	LCS\MS\Surr Spiking Level			
	Spike Solution [ug/mL]	**Surr. Solution [ug/mL]**	**Spike Conc [ug/L]**	**Surr Conc [ug/L]**
RVT	2.5	0.5	250	50
Soil/Water	12.5	2	3125	500
Oil	12.5	2	1250	200
Caulking	125	20	3125	200

TABLE 6

QC FAILURE ACTION PLAN

Blank Failures	Failure Situation	Action(s)
Surrogate Failure	1 of 4 high or low	Report as is
	2 or more high	Report as is
	2 or more low, samples are non-detect with passing surrogates	Report as is
	2 or more low, samples have hits and/or failing surrogates	Revial/rerun Re-extract if still failing
IS Failure	IS not present (less than 10% recovery)	Add IS Rerun
	IS out high or low	Revial/rerun Re-extract if still failing
Contamination	Below RL; sample(s) non-detect, below RL, or concentration 10 times or more above the blank	Report as is
	Below RL, sample(s) concentration above RL	See team leader/manager
	Above RL, sample(s) concentration 10 times or more above the blank	Report as is
	Above RL with non-detect sample(s)	Report as is
	Above RL, sample(s) concentration less than 10 times above the blank and volume available	Revial/rerun Re-extract if contamination still present
	Above RL, sample(s) concentration less than 10 times above the blank and no volume available	Revial/rerun B-flag affected data

LCS/LCSD Failures	Failure Situation	Action(s)
Surrogate Failure	1 or more high or low	Report as is
IS Failure	IS not present (less than 10% recovery)	Add IS

		Rerun
	IS out high or low	Revial/rerun Re-extract if still failing
Spike Failure	Spike compounds out high and sample(s) are non-detect with passing surrogates	Report as is
	Spike compounds out high and sample(s) have hits	Revial/rerun Re-extract if still high
	Spike compounds out low	Revial/rerun Re-extract if still low
	RPD failure	Report as is

MS/MSD/DUP Failures	**Failure Situation**	**Action(s)**
Surrogate Failure	(MS/MSD) 1 or more high or low	Report as is
	(MS/MSD) 2 or more high or low	Report as is
	(DUP) 1 or more high, parent sample non-detect	Report as is
	(DUP) 1 or more low, parent sample matches	Report as is
	(DUP) 1 or more low, parent sample doesn't match	Revial/rerun Re-extract if still failing
IS Failure	IS not present (less than 10% recovery)	Add IS Rerun
	IS out high or low	Revial/rerun Re-extract if still failing
Spike Failure (MS/MSD only)	Compounds out high or low due to sample matrix or hits	Report as is
	Compounds out high or low due to extraction error	Revial/rerun Re-extract if still failing
	RDP failure	Report as is

PFAS by EPA 537 in Drinking Water by LC/MS/MS

EPA Method 537.1, Version 2, March 2020,
Document #: EPA/600/R-20/006

Department of Defense, Quality Systems Manual for
Environmental Laboratories, Version 5.4, 2021

1. Scope and Application

Matrices: Drinking Water

Definitions: Refer to Alpha Analytical Quality Manual.

1.1 This is a liquid chromatography/tandem mass spectrometry (LC/MS/MS) method for the determination of selected perfluorinated alkyl substances (PFASs) in drinking water. Accuracy and precision data have been generated for the compounds listed in Table 1.

1.2 The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

1.3 This method is restricted to use by or under the supervision of analysts experienced in the operation of the LC/MS/MS and in the interpretation of LC/MS/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Table 1

Parameter	Acronym	CAS
Hexafluoropropylene oxide dimer acid [1]	HFPO-DA	13252-13-6
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluorodecanoic acid	PFDA	335-76-2
Perfluorododecanoic acid	PFDoA	307-55-1
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluorohexanoic acid	PFHxA	307-24-4

Table 1 (cont.)

Perfluorononanoic acid	PFNA	375-95-1
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorotetradecanoic acid	PFTA	376-06-7
Perfluorotridecanoic acid	PFTrDA	72629-94-8
Perfluoroundecanoic acid	PFUnA	2058-94-8
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid [1]	11Cl-PF3OUdS	763051-92-9
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid [1]	9Cl-PF3ONS	756426-58-1
4,8-dioxa-3H-perfluorononanoic acid [1]	ADONA	919005-14-4

2. Summary of Method

2.1 A 250-mL water sample is fortified with surrogates and passed through a solid phase extraction (SPE) cartridge containing polystyrenedivinylbenzene (SDVB) to extract the method analytes and surrogates. The compounds are eluted from the solid phase with a small amount of methanol. The extract is concentrated to dryness with nitrogen in a heated water bath, and then adjusted to a 1-mL volume with 96:4% (vol/vol) methanol: water after adding the IS(s). A 2 µL injection is made into an LC equipped with a C18 column that is interfaced to an MS/MS. The analytes are separated and identified by comparing the acquired mass spectra and retention times to reference spectra and retention times for calibration standards acquired under identical LC/MS/MS conditions. The concentration of each analyte is determined by using the internal standard technique. Surrogate analytes are added to all Field and QC Samples to monitor the extraction efficiency of the method analytes.

2.2 Method Modifications from Reference

2.2.1 None.

3. Reporting Limits

3.1 The reporting limit for PFAS's is 2 ng/L (4ng/L for HFPO-DA).

4. Interferences

4.1 PFAS standards, extracts and samples should not come in contact with any glass containers or pipettes as these analytes can potentially adsorb to glass surfaces. PFAS analyte, IS and SUR standards commercially purchased in glass ampoules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene containers.

4.2 Method interferences may be caused by contaminants in solvents, reagents (including reagent water), sample bottles and caps, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the chromatograms. The method analytes in this method can also be found in many common laboratory supplies and equipment, such as PTFE (polytetrafluoroethylene) products, LC solvent lines, methanol, aluminum foil, SPE sample transfer lines, etc. All items such as these must be routinely demonstrated to be

free from interferences (less than 1/3 the RL for each method analyte) under the conditions of the analysis by analyzing laboratory reagent blanks as described in Section 9.2. **Subtracting blank values from sample results is not permitted.**

4.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature of the water. Humic and/or fulvic material can be co-extracted during SPE and high levels can cause enhancement and/or suppression in the electrospray ionization source or low recoveries on the SPE sorbent. Total organic carbon (TOC) is a good indicator of humic content of the sample. Under the LC conditions used during method development, matrix effects due to total organic carbon (TOC) were not observed.

4.4 Relatively large quantities of the preservative (Sect. 6.2.1) are added to sample bottles. The potential exists for trace-level organic contaminants in these reagents. Interferences from these sources should be monitored by analysis of laboratory reagent blanks (Sect. 9.2.1), particularly when new lots of reagents are acquired.

4.5 SPE cartridges can be a source of interferences. The analysis of field and laboratory reagent blanks can provide important information regarding the presence or absence of such interferences. Brands and lots of SPE devices should be tested to ensure that contamination does not preclude analyte identification and quantitation.

5. Health and Safety

5.1 The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

5.2 All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.3 PFOA has been described as “likely to be carcinogenic to humans.” Pure standard materials and stock standard solutions of these method analytes should be handled with suitable protection to skin and eyes, and care should be taken not to breathe the vapors or ingest the materials.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

6.1.1 Samples must be collected in two (2) 250-mL high density polyethylene (HDPE) container with an unlined plastic screw cap.

6.1.2 The sample handler must wash their hands before sampling and wear nitrile gloves while filling and sealing the sample bottles. PFAS contamination during sampling can occur from a number of common sources, such as food packaging and certain foods and beverages. Proper hand washing and wearing nitrile gloves will aid in minimizing this type of accidental contamination of the samples.

6.1.3 Open the tap and allow the system to flush until the water temperature has stabilized (approximately 3 to 5 min). Collect samples from the flowing system.

6.1.4 Fill sample bottles, taking care not to flush out the sample preservation reagent. Samples do not need to be collected headspace free.

6.1.5 After collecting the sample, cap the bottle and agitate by hand until preservative is dissolved. Keep the sample sealed from time of collection until extraction.

6.1.6 Field Reagent Blank (FRB)

6.1.6.1 A FRB must be submitted with each sample set. At the laboratory, fill an unpreserved 250 mL container with reagent water. For each FRB, an empty sample bottle (containing only the Trizma preservative) must also be shipped. At the sampling site, the sampler must open the shipped FRB and pour the reagent water into the empty shipped sample bottle, seal and label this bottle as the FRB. The FRB is shipped back to the laboratory along with the samples and analyzed to ensure that PFASs were not introduced into the sample during the sample collection/handling.

6.1.6.2 The same batch of preservative must be used for the FRBs as for the field samples.

6.1.6.3 The reagent water used for the FRBs must be initially analyzed for method analytes as a MB and must meet the MB criteria in Section 9.2.1 prior to use. This requirement will ensure samples are not being discarded due to contaminated reagent water rather than contamination during sampling.

6.2 Sample Preservation

6.2.1 The preservation reagent, listed in the table below, is added to each sample bottle as a solid prior to shipment to the field (or prior to sample collection).

Table 2

Compound	Amount	Purpose
Trizma	5.0 g/l	Buffering reagent and removes free chlorine

6.3 Sample Shipping

6.3.1 Samples must be chilled during shipment and must not exceed 10 °C during the first 48 hours after collection. Sample temperature must be confirmed to be at or below 10 °C when the samples are received at the laboratory. Samples stored in the lab must be held at or below 6 °C until extraction but should not be frozen.

NOTE: Samples that are significantly above 10° C, at the time of collection, may need to be iced or refrigerated for a period of time, in order to chill them prior to shipping. This will allow them to be shipped with sufficient ice to meet the above requirements.

6.4 Sample Handling

6.4.1 Holding Times

6.4.1.1 Water samples should be extracted as soon as possible but must be extracted within 14 days. Extracts must be stored at room temperature and analyzed within 28 days after extraction.

7. Equipment and Supplies

7.1 SAMPLE CONTAINERS – 250-mL high density polyethylene (HDPE) bottles fitted with unlined screw caps. Sample bottles must be discarded after use.

7.2 POLYPROPYLENE BOTTLES – 4-mL narrow-mouth polypropylene bottles.

7.3 CENTRIFUGE TUBES – 15-mL conical polypropylene tubes with polypropylene screw caps for storing standard solutions and for collection of the extracts.

7.4 AUTOSAMPLER VIALS – Polypropylene 0.7-mL autosampler vials with polypropylene caps.

7.4.1 NOTE: Polypropylene vials and caps are necessary to prevent contamination of the sample from PTFE coated septa. However, polypropylene caps do not reseal, so evaporation occurs after injection. Thus, multiple injections from the same vial are not possible.

7.5 POLYPROPYLENE GRADUATED CYLINDERS – Suggested sizes include 25, 50, 100 and 1000-mL cylinders.

7.6 MICRO SYRINGES – Suggested sizes include 5, 10, 25, 50, 100, 250, 500 and 1000-µL syringes.

7.7 PLASTIC PIPETS – Polypropylene or polyethylene disposable pipets.

7.8 pH PAPER – Capable of increments of .5

7.9 ANALYTICAL BALANCE – Capable of weighing to the nearest 0.0001 g.

7.10 SOLID PHASE EXTRACTION (SPE) APPARATUS FOR USING CARTRIDGES

7.10.1 SPE CARTRIDGES – 0.5 g, 6-mL SPE cartridges containing styrenedivinylbenzene (SDVB) sorbent phase.

7.10.2 VACUUM EXTRACTION MANIFOLD – A manual vacuum manifold with large volume sampler for cartridge extractions, or an automatic/robotic sample preparation system designed for use with SPE cartridges, may be used if all QC requirements discussed in Section 9 are met. Extraction and/or elution steps may not be changed or omitted to accommodate the use of an automated system. Care must be taken with automated SPE systems to ensure the PTFE commonly used in these systems does not contribute to unacceptable analyte concentrations in the MB (Sect. 9.2.1).

7.10.3 SAMPLE DELIVERY SYSTEM – Use of a polypropylene transfer tube system, which transfers the sample directly from the sample container to the SPE cartridge, is recommended, but not mandatory. Standard extraction manifolds come equipped with PTFE transfer tube systems. These can be replaced with 1/8” O.D. x 1/16” I.D. polypropylene or polyethylene tubing cut to an appropriate length to ensure no sample contamination from the sample transfer lines. Other types of non-PTFE tubing may be used provided it meets the MB (Sect. 9.2.1) and LCS (Sect. 9.3) QC requirements. The PTFE transfer tubes may be used, but an MB must be run on each PFTE transfer tube and the QC requirements in

Section 13.2.2 must be met. In the case of automated SPE, the removal of PTFE lines may not be feasible; therefore, MBs will need to be rotated among the ports and must meet the QC requirements of Sections 13.2.2 and 9.2.1.

7.11 EXTRACT CONCENTRATION SYSTEM – Extracts are concentrated by evaporation with nitrogen using a water bath set no higher than 60-65 °C.

7.12 LABORATORY OR ASPIRATOR VACUUM SYSTEM – Sufficient capacity to maintain a vacuum of approximately 10 to 15 inches of mercury for extraction cartridges.

7.13 LIQUID CHROMATOGRAPHY (LC)/TANDEM MASS SPECTROMETER (MS/MS) WITH DATA SYSTEM

7.13.1 LC SYSTEM – Instrument capable of reproducibly injecting up to 10-µL aliquots and performing binary linear gradients at a constant flow rate near the flow rate used for development of this method (0.3-1 mL/min). The LC must be capable of pumping the water/methanol mobile phase without the use of a degasser which pulls vacuum on the mobile phase bottle (other types of degassers are acceptable). Degassers which pull vacuum on the mobile phase bottle will volatilize the ammonium acetate mobile phase causing the analyte peaks to shift to earlier retention times over the course of the analysis batch. The usage of a column heater is optional.

7.13.2 LC/TANDEM MASS SPECTROMETER – The LC/MS/MS must be capable of negative ion electrospray ionization (ESI) near the suggested LC flow rate of 0.3-1 mL/min. The system must be capable of performing MS/MS to produce unique product ions for the method analytes within specified retention time segments. A minimum of 10 scans across the chromatographic peak is required to ensure adequate precision.

7.13.3 DATA SYSTEM – An interfaced data system is required to acquire, store, reduce, and output mass spectral data. The computer software should have the capability of processing stored LC/MS/MS data by recognizing an LC peak within any given retention time window. The software must allow integration of the ion abundance of any specific ion within specified time or scan number limits. The software must be able to calculate relative response factors, construct linear regressions or quadratic calibration curves, and calculate analyte concentrations.

7.13.4 ANALYTICAL COLUMN – An LC C_{18} column (2.1 x 50 mm) packed with 1.7 µm d_p C_{18} solid phase particles was used. Any column that provides adequate resolution, peak shape, capacity, accuracy, and precision (Sect. 9) may be used.

8. Reagents and Standards

8.1 GASES, REAGENTS, AND SOLVENTS – Reagent grade or better chemicals should be used.

8.1.1 REAGENT WATER – Purified water which does not contain any measurable quantities of any method analytes or interfering compounds greater than 1/3 the RL for each method analyte of interest. Prior to daily use, at least 3 L of reagent water should be flushed from the purification system to rinse out any build-up of analytes in the system's tubing.

8.1.2 METHANOL (CH_3OH, CAS#: 67-56-1) – High purity, demonstrated to be free of analytes and interferences.

8.1.3 AMMONIUM ACETATE ($NH_4C_2H_3O_2$, CAS#: 631-61-8) – High purity, demonstrated to be free of analytes and interferences.

8.1.4 2 mM AMMONIUM ACETATE/REAGENT WATER – To prepare 1 L, add .154 g ammonium acetate to 1 L of reagent water. This solution is prone to volatility losses and should be replaced at least every 48 hours.

8.1.5 TRIZMA PRESET CRYSTALS, pH 7.0 – Reagent grade. A premixed blend of Tris [Tris(hydroxymethyl)aminomethane] and Tris HCL [Tris(hydroxymethyl)aminomethane hydrochloride]. Alternatively, a mix of the two components with a weight ratio of 15.5/1 Tris HCL/Tris may be used. These blends are targeted to produce a pH near 7.0 at 25 °C in reagent water. Trizma functions as a buffer, and removes free chlorine in chlorinated finished waters (Sect. 6.2.1).

8.1.6 NITROGEN – Used for the following purposes: Nitrogen aids in aerosol generation of the ESI liquid spray and is used as collision gas in some MS/MS instruments. The nitrogen used should meet or exceed instrument manufacturer's specifications. In addition, Nitrogen is used to concentrate sample extracts (Ultra High Purity or equivalent).

8.1.7 ARGON – Used as collision gas in MS/MS instruments. Argon should meet or exceed instrument manufacturer's specifications. Nitrogen gas may be used as the collision gas provided sufficient sensitivity (product ion formation) is achieved.

8.2 STANDARD SOLUTIONS – When a compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. PFAS analyte, IS and SUR standards commercially purchased in glass ampoules are acceptable; however, all subsequent transfers or dilutions performed by the analyst must be prepared and stored in polypropylene containers. Standards for sample fortification generally should be prepared in the smallest volume that can be accurately measured to minimize the addition of excess organic solvent to aqueous samples.

NOTE: Stock standards (Sect. 8.2.1, 8.2.3 and 8.2.5) are stored at ≤4 °C. Primary dilution standards (Sect. 8.2.2 and 8.2.4) are stored at room temperature to prevent adsorption of the method analytes onto the container surfaces that may occur when refrigerated. Storing the standards at room temperature will also minimize daily imprecision due to the potential of inadequate room temperature stabilization.

8.2.1 IS STOCK STANDARD SOLUTIONS - IS stock standard solutions are stable for at least 6 months when stored at 4 °C. The stock solution is purchased at a concentration range of 1-4 ng/µl.

8.2.2 INTERNAL STANDARD PRIMARY DILUTION (IS PDS) STANDARD (0.5-2 ng/µL) – Prepare the IS PDS at a concentration of 0.5-2 ng/µL. The IS PDS is prepared in 96:4% (vol/vol) methanol:water. The IS PDS is stable for at least two months when stored in polypropylene centrifuge tubes at room temperature.

Table 3

Internal Standard	Conc. of IS Stock (ng/uL)	Vol. of IS Stock (mL)	Final Vol. of IS PDS (mL)	Final Conc. of IS PDS (ng/µL)
^{13}C-PFOA	1	1.0	2.0	0.5

^{13}C-PFOS	3	1.0	2.0	1.5
d_3-NMeFOSAA	4	1.0	2.0	2.0

8.2.3 SUR STOCK STANDARD SOLUTIONS – SUR stock standard solutions are stable for at least 6 months when stored at 4 °C.

8.2.4 SURROGATE PRIMARY DILUTION STANDARD (SUR PDS) (0.5-2 ng/µL) – Prepare the SUR PDS at a concentration of 0.5-2 ng/µL. The SUR PDS is prepared in 96:4% (vol/vol) methanol:water. This solution is used to fortify all QC and Field Samples. The PDS is stable for one year when stored in polypropylene centrifuge tubes at room temperature.

Table 4

Surrogate	Conc. of SUR Stock (ng/µL)	Vol. of SUR Stock (mL)	Final Vol. of SUR PDS (,L)	Final Conc. of SUR PDS (ng/µL)
^{13}C-PFHxA	1.0	1.0	2.0	0.5
^{13}C-PFDA	1.0	1.0	2.0	0.5
d_5-NEtFOSAA	4.0	1.0	2.0	2.0
Tetrafluoro-2-heptafluoropropoxy-13C3-propanoic acid	50	1.0	2.0	0.5

8.2.5 ANALYTE STOCK STANDARD SOLUTION – Analyte stock standards are stable for at least 6 months when stored at -15 °C. When using these stock standards to prepare a PDS, care must be taken to ensure that these standards are at room temperature and adequately vortexed.

Table 5

Analyte	Analyte Stock Solvent	Concentration (ug/mL)
PFHxA	100% methanol	1.0
PFHpA	100% methanol	1.0
PFOA	100% methanol	1.0
PFNA	100% methanol	1.0
PFDA	100% methanol	1.0
PFUnA	100% methanol	1.0
PFDoA	100% methanol	1.0
PFTrDA	100% methanol	1.0
PFTA	100% methanol	1.0
PFBS	100% methanol	1.0
PFHxS	100% methanol	1.0
PFOS	100% methanol	1.0
NEtFOSAA	100% methanol	1.0
NMeFOSAA	100% methanol	1.0
HFPO-DA	100% methanol	50.0
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	100% methanol	50.0
9-chlorohexadecafluoro-3-oxanone-1-sulfonic acid	100% methanol	50.0

Analyte	Analyte Stock Solvent	Concentration (ug/mL)
4,8-dioxa-3H-perfluorononanoic acid	100% methanol	50.0

8.2.6 LOW, MEDIUM AND HIGH LEVEL LCS – The LCS's will be prepared at the following concentrations and rotated per batch; 2 ng/L, 40 ng/L, 160 ng/l. The analyte PDS contains all the method analytes of interest at various concentrations in methanol containing 4% water. The analyte PDS has been shown to be stable for 6 months when stored at room temperature.

8.2.7 CALIBRATION STANDARDS (CAL) –

Current Concentrations (ng/mL): 0.25, 0.5, 5.0, 10.0, 50.0 and 100 (optional)

Prepare the CAL standards over the concentration range of interest from dilutions of the analyte PDS in methanol containing 4% reagent water. The IS and SUR are added to the CAL standards at a constant concentration (10-40 ng/L). The lowest concentration CAL standard must be at or below the RL (2 ng/L), which may depend on system sensitivity. The CAL standards may also be used as CCVs (Sect. 9.9). The CAL standards are stable for at least two weeks when stored at room temperature. Longer storage times are acceptable provided appropriate QC measures are documented demonstrating the CAL standard stability.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 REPORTING LIMIT (RL) CONFIRMATION

9.1.1 Fortify, extract, and analyze seven replicate LCSs at 2 ng/l. These LCSs must contain all method preservatives described in Section 6.2.1. Calculate the mean measured concentration (*Mean*) and standard deviation for these replicates. Determine the Half Range for the prediction interval of results (HR_{PIR}) using the equation below

$$HR_{PIR} = 3.963s$$

Where:

s = the standard deviation
3.963 = a constant value for seven replicates.

9.1.2 Confirm that the upper and lower limits for the Prediction Interval of Result ($PIR = Mean \pm HR_{PIR}$) meet the upper and lower recovery limits as shown below

The Upper PIR Limit must be ≤150% recovery.

$$\frac{Mean + HR_{PIR}}{Fortified\ Concentration} \times 100\% \leq 150\%$$

The Lower PIR Limit must be ≥ 50% recovery.

$$\frac{Mean - HR_{PIR}}{Fortified\ Concentration} \times 100\% \geq 50\%$$

9.1.3 The RL is validated if both the Upper and Lower PIR Limits meet the criteria described above. If these criteria are not met, the RL has been set too low and must be determined again at a higher concentration.

9.2 Blank(s)

9.2.1 **METHOD BLANK (MB) -** A Method Blank (MB) is required with each extraction batch to confirm that potential background contaminants are not interfering with the identification or quantitation of method analytes. If more than 20 Field Samples are included in a batch, analyze an MB for every 20 samples. If the MB produces a peak within the retention time window of any analyte that would prevent the determination of that analyte, determine the source of contamination and eliminate the interference before processing samples. Background contamination must be reduced to an acceptable level before proceeding. Background from method analytes or other contaminants that interfere with the measurement of method analytes must be below 1/3 of the RL. Blank contamination is estimated by extrapolation, if the concentration is below the lowest CAL standard. This extrapolation procedure is not allowed for sample results as it may not meet data quality objectives. If the method analytes are detected in the MB at concentrations equal to or greater than 1/3 of the RL, then re-extraction must be performed for all samples in the batch that have those target analytes above the reporting limit.

9.2.2 **FIELD REAGENT BLANK (FRB)** - The purpose of the FRB is to ensure that PFASs measured in the Field Samples were not inadvertently introduced into the sample during sample collection/handling. Analysis of the FRB is required only if a Field Sample contains a method analyte or analytes at or above the RL. The FRB is processed, extracted and analyzed in exactly the same manner as a Field Sample. If the method analyte(s) found in the Field Sample is present in the FRB at a concentration greater than 1/3 the RL, then all samples collected with that FRB are invalid and must be recollected and reanalyzed.

9.3 Laboratory Control Sample (LCS)

9.3.1 An LCS is required with each extraction batch. The fortified concentration of the LCS must be rotated between low, medium, and high concentrations from batch to batch.

9.3.2 The low concentration LCS must be as near as practical to, but no more than two times, the RL. Similarly, the high concentration LCS should be near the high end of the calibration range established during the initial calibration (Sect. 10.6).

9.3.3 Results of the low-level LCS analyses must be 50-150% of the true value. Results of the medium and high-level LCS analyses must be 70-130% of the true value. If the LCS results do not meet these criteria for method analytes, then all data for the problem analyte(s) must be considered invalid for all samples in the extraction batch.

9.3.4 It is the responsibility of the extraction chemist to view the previous extraction batch to determine the next spiking concentration. (Low → Medium → High)

9.4 Internal Standards (IS)

The analyst must monitor the peak areas of the IS(s) in all injections during each analysis day. The IS responses (peak areas) in any chromatographic run must be within 70-140% of the response in the most recent CCV and must not deviate by more than 50% from the average area measured during initial analyte calibration. If the IS areas in a chromatographic run do not meet these criteria, inject a second aliquot of that extract aliquoted into a new capped autosampler vial. Random evaporation losses have been observed with the polypropylene caps causing high IS(s) areas.

9.4.1 If the reinjected aliquot produces an acceptable IS response, report results for that aliquot.

9.4.2 If the reinjected extract fails again, the analyst should check the calibration by reanalyzing the most recently acceptable CAL standard. If the CAL standard fails the criteria of Section 9.9, recalibration is in order per Section 10.6. If the CAL standard is acceptable, extraction of the sample may need to be repeated provided the sample is still within the holding time. Otherwise, report results obtained from the reinjected extract, but annotate as suspect. Alternatively, collect a new sample and re-analyze.

9.5 Surrogate Recovery

The SUR standard is fortified into all samples, CCVs, MBs, LCSs, MSs, MSDs, FD, and FRB prior to extraction. It is also added to the CAL standards. The SUR is a means of assessing method performance from extraction to final chromatographic measurement. Calculate the recovery (%R) for the SUR using the following equation

$$\%R = (A / B) \times 100$$

Where:

A = calculated SUR concentration for the QC or Field Sample
B = fortified concentration of the SUR.

9.5.1.1 SUR recovery must be in the range of 70-130%. When SUR recovery from a sample, blank, or CCV is less than 70% or greater than 130%, check 1) calculations to locate possible errors, 2) standard solutions for degradation, 3) contamination, and 4) instrument performance. Correct the problem and reanalyze the extract.

9.5.1.2 If the extract reanalysis meets the SUR recovery criterion, report only data for the reanalyzed extract.

9.5.1.3 If the extract reanalysis fails the 70-130% recovery criterion, the analyst should check the calibration by injecting the last CAL standard that passed. If the CAL standard fails the criteria of Section 10.7, recalibration is in order per Section 10.6. If the CAL standard is acceptable, extraction of the sample should be repeated provided the sample is still within the holding time. If the re-extracted sample also fails the recovery criterion, report all data for that sample as suspect/SUR recovery to inform the

data user that the results are suspect due to SUR recovery. Alternatively, collect a new sample and re-analyze.

9.6 Matrix Spike (MS)

9.6.1 Analysis of an MS is required in each extraction batch and is used to determine that the sample matrix does not adversely affect method accuracy. Assessment of method precision is accomplished by analysis of a Field Duplicate (FD) (Sect. 9.7); however, infrequent occurrence of method analytes would hinder this assessment. If the occurrence of method analytes in the samples is infrequent, or if historical trends are unavailable, a second MS, or MSD, must be prepared, extracted, and analyzed from a duplicate of the Field Sample. Extraction batches that contain MSDs will not require the extraction of a field sample duplicate.

9.6.2 Within each extraction batch, a minimum of one Field Sample is fortified as an MS for every 20 Field Samples analyzed. The MS is prepared by spiking a sample with an appropriate amount of the Analyte Stock Standard (Sect. 8.2.5). Use historical data and rotate through the low, mid and high concentrations when selecting a fortifying concentration. Calculate the percent recovery (%*R*) for each analyte using the equation

$$\%R = \frac{(A - B)}{C} \times 100$$

Where:

A = measured concentration in the fortified sample
B = measured concentration in the unfortified sample
C = fortification concentration.

9.6.3 Analyte recoveries may exhibit matrix bias. For samples fortified at or above their native concentration, recoveries should range between 70-130%, except for low-level fortification near or at the RL (within a factor of 2-times the RL concentration) where 50-150% recoveries are acceptable. If the accuracy of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the CCVs, the recovery is judged to be matrix biased. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.7 Laboratory Duplicate

9.7.1 FIELD DUPLICATE OR LABORATORY FORTIFIED SAMPLE MATRIX DUPLICATE (FD or MSD) – Within each extraction batch (not to exceed 20 Field Samples), a minimum of one FD or MSD must be analyzed. Duplicates check the precision associated with sample collection, preservation, storage, and laboratory procedures. If method analytes are not routinely observed in Field Samples, an MSD should be analyzed rather than an FD.

9.7.2 Calculate the relative percent difference (*RPD*) for duplicate measurements (*FD1* and *FD2*) using the equation

$$RPD = \frac{|FD1 - FD2|}{(FD1 + FD2) / 2} \times 100$$

9.7.3 RPDs for FDs should be ≤30%. Greater variability may be observed when FDs have analyte concentrations that are within a factor of 2 of the RL. At these concentrations, FDs should have RPDs that are ≤50%. If the RPD of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the CCV, the recovery is judged to be matrix biased. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.7.4 If an MSD is analyzed instead of a FD, calculate the relative percent difference (RPD) for duplicate MSs (MS and MSD) using the equation

$$RPD = \frac{|MS - MSD|}{(MS + MSD) / 2} \times 100$$

9.7.5 RPDs for duplicate MSs should be ≤30% for samples fortified at or above their native concentration. Greater variability may be observed when MSs are fortified at analyte concentrations that are within a factor of 2 of the RL. MSs fortified at these concentrations should have RPDs that are ≤50% for samples fortified at or above their native concentration. If the RPD of any analyte falls outside the designated range, and the laboratory performance for that analyte is shown to be in control in the CCV, the recovery is judged to be matrix biased. The result for that analyte in the unfortified sample is labeled suspect/matrix to inform the data user that the results are suspect due to matrix effects.

9.8 Initial Calibration Verification (ICV)

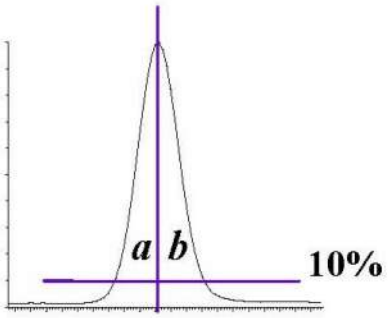
9.8.1 As part of the IDC (Sect. 13.2), each time a new Analyte Stock Standard solution (Sect. 8.2.5) is used, and at least quarterly, analyze a QCS sample from a source different from the source of the CAL standards. If a second vendor is not available, then a different lot of the standard should be used. The QCS should be prepared and analyzed just like a CCV. Acceptance criteria for the QCS are identical to the CCVs; the calculated amount for each analyte must be ± 30% of the expected value. If measured analyte concentrations are not of acceptable accuracy, check the entire analytical procedure to locate and correct the problem.

9.9 Continuing Calibration Verification (CCV)

9.9.1 CCV Standards are analyzed at the beginning of each analysis batch, after every 10 Field Samples, and at the end of the analysis batch. See Section 10.7 for concentration requirements and acceptance criteria.

9.10 Method-specific Quality Control Samples

9.10.1 PEAK ASYMMETRY FACTOR – A peak asymmetry factor must be calculated using the equation below during the IDL and every time a calibration curve is generated. The peak asymmetry factor for the first two eluting peaks in a midlevel CAL standard (if only two analytes are being analyzed, both must be evaluated) must fall in the range of



0.8 to 1.5. Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted. See guidance in Section 10.6.4.1 if the calculated peak asymmetry factors do not meet the criteria.

$$A_s = b / a$$

Where:

A_s = peak asymmetry factor
b = width of the back half of the peak measured (at 10% peak height) from the trailing edge of the peak to a line dropped perpendicularly from the peak apex
a = the width of the front half of the peak measured (at 10% peak height) from the leading edge of the peak to a line dropped perpendicularly from the apex.

9.11 Method Sequence

ICV

CCV-LOW

MB

LCS

LCSD

MS

Duplicate or MSD

Field Samples (1-10)

CCV-MID

Field Samples (11-20)

CCV-HIGH

10. Procedure

10.1 Equipment Set-up

10.1.1 This procedure may be performed manually or in an automated mode using a robotic or automatic sample preparation device. If an automated system is used to prepare samples, follow the manufacturer's operating instructions, but all extraction and elution steps must be the same as in the manual procedure. Extraction and/or elution steps may not be changed or omitted to accommodate the use of an automated system. If an automated system is used, the MBs should be rotated among the ports to ensure that all the valves and tubing meet the MB requirements (Sect. 9.2).

10.1.2 Some of the PFASs adsorb to surfaces, including polypropylene. Therefore, the aqueous sample bottles must be rinsed with the elution solvent (Sect 10.3.4) whether extractions are performed manually or by automation. The bottle rinse is passed through the cartridge to elute the method analytes and is then collected (Sect. 10.3.4).

10.1.3 **NOTE:** The SPE cartridges and sample bottles described in this section are designed as single use items and should be discarded after use. They may not be refurbished for reuse in subsequent analyses.

10.2 Sample Preparation

10.2.1 Samples are preserved, collected and stored as presented in Section 6. All Field and QC Samples, including the MB, LCS and FRB, must contain the dechlorinating agent listed in Section 6.2.1. Determine sample volume. An indirect measurement may be done in one of two ways: by marking the level of the sample on the bottle or by weighing the sample and bottle to the nearest 1 g. After extraction, proceed to Section 10.5 for final volume determination. Some of the PFASs adsorb to surfaces, thus the sample volume may **NOT** be transferred to a graduated cylinder for volume measurement. The MB, LCS and FRB may be prepared by measuring 250 mL of reagent water with a polypropylene graduated cylinder or filling a 250-mL sample bottle to near the top.

Before extraction, verify the pH of the sample is 7 ± 0.5 by taking a drop of sample using a plastic (polypropylene or polyethylene) disposable pipet and transfer to a strip of pH paper with 0.5 unit increments. The sample pH is to be recorded in the laboratory extraction logbook.

The entire sample that is received must be sent through the SPE cartridge. In addition, the bottle must be solvent rinsed and this rinse must be sent through the SPE cartridge as well. The method blank (MB) and laboratory control sample (LCS) must be extracted in exactly the same manner (i.e., must include the bottle solvent rinse). It should be noted that a water rinse alone is not sufficient. This does not apply to samples with high concentrations of PFAS that are prepared using serial dilution and not SPE.

10.2.2 Add 20 µL of the SUR PDS (Sect. 8.2.4) to each sample, cap and invert to mix for a final concentration of 10 ng/L for ^{13}C-PFHxA and ^{13}C-PFDA and 40 ng/L for d_5-NEtFOSAA.

10.2.3 In addition to the SUR(s) and dechlorination agent, if the sample is an LCS, MS, or MSD, add the necessary amount of analyte PDS (Sect. 8.2.5). Cap and invert each sample to mix.

10.3 Cartridge SPE Procedure

10.3.1 CARTRIDGE CLEAN-UP AND CONDITIONING – DO NOT allow cartridge packing material to go dry during any of the conditioning steps. Rinse each cartridge with 15 mL of methanol. Next, rinse each cartridge with 18 mL of reagent water, without allowing the water to drop below the top edge of the packing. If the cartridge goes dry during the conditioning phase, the conditioning must be started over. Add 4-5 mL of reagent water to each cartridge, attach the sample transfer tubes (Sect. 7.2.3), turn on the vacuum, and begin adding sample to the cartridge.

10.3.2 SAMPLE EXTRACTON – Adjust the vacuum so that the approximate flow rate is 10-15 mL/min. Do not allow the cartridge to go dry before all the sample has passed through.

10.3.3 SAMPLE BOTTLE AND CARTRIDGE RINSE – After the entire sample has passed through the cartridge, rinse the sample bottles with two 7.5-mL aliquots of reagent water and draw each aliquot through the sample transfer tubes and the cartridges. Draw air or nitrogen through the cartridge for 5 min at high vacuum (10-15 in. Hg).

NOTE: If empty plastic reservoirs are used in place of the sample transfer tubes to pass the samples through the cartridges, these reservoirs must be treated like the transfer tubes. After the entire sample has passed through the cartridge, the reservoirs must be rinsed to waste with reagent water.

10.3.4 SAMPLE BOTTLE AND CARTRIDGE ELUTION – Turn off and release the vacuum. Lift the extraction manifold top and insert a rack with collection tubes into the extraction tank to collect the extracts as they are eluted from the cartridges. Rinse the sample bottles with 4 mL of methanol and elute the analytes from the cartridges by pulling the 4 mL of methanol through the sample transfer tubes and the cartridges. Use a low vacuum such that the solvent exits the cartridge in a dropwise fashion. Repeat sample bottle rinse and cartridge elution with a second 4-mL aliquot of methanol.

NOTE: If empty plastic reservoirs are used in place of the sample transfer tubes to pass the samples through the cartridges, these reservoirs must be treated like the transfer tubes. After the reservoirs have been rinsed in Section 10.3.3, the elution solvent used to rinse the sample bottles must be swirled down the sides of the reservoirs while eluting the cartridge to ensure that any method analytes on the surface of the reservoirs are transferred to the extract.

10.4 Extract Concentration

10.4.1 Concentrate the extract to dryness under a gentle stream of nitrogen in a heated water bath (60-65 °C) to remove all the water/methanol mix. Add the appropriate amount of 96:4% (vol/vol) methanol:water solution and the IS PDS (Sect. 8.2.2) to the collection vial to bring the volume to 1 mL and vortex. Transfer a small aliquot with a plastic pipet (Sect. 7.6) to a polypropylene autosampler vial.

NOTE: It is recommend that the entire 1-mL aliquot not be transferred to the autosampler vial because the polypropylene autosampler caps do not reseal after injection. Therefore, do not store the extracts in the autosampler vials as evaporation losses can occur occasionally in these autosampler vials. Extracts can be stored in 15-mL centrifuge tubes (Sect. 7.3).

10.5 Sample Volume Determination

10.5.1 If the level of the sample was marked on the sample bottle, use a graduated cylinder to measure the volume of water required to fill the original sample bottle to the mark made prior to extraction. Determine to the nearest 10 mL. If using weight to determine volume, weigh the empty bottle to the nearest 10 g and determine the sample weight by subtraction of the empty bottle weight from the original sample weight (Sect. 10.2.1). Assume a sample density of 1.0 g/mL. In either case, the sample volume will be used in the final calculations of the analyte concentration (Sect. 11.2).

10.6 Initial Calibration - Demonstration and documentation of acceptable initial calibration is required before any samples are analyzed. After the initial calibration is successful, a

CCV is required at the beginning and end of each period in which analyses are performed, and after every tenth Field Sample.

10.6.1 ESI-MS/MS TUNE

10.6.1.1 Calibrate the mass scale of the MS with the calibration compounds and procedures prescribed by the manufacturer.

10.6.1.2 Optimize the [M-H]- for each method analyte by infusing approximately 0.5-1.0 µg/mL of each analyte (prepared in the initial mobile phase conditions) directly into the MS at the chosen LC mobile phase flow rate (approximately 0.6 mL/min). This tune can be done on a mix of the method analytes. The MS parameters (voltages, temperatures, gas flows, etc.) are varied until optimal analyte responses are determined. The method analytes may have different optima requiring some compromise between the optima.

10.6.1.3 Optimize the product ion for each analyte by infusing approximately 0.5-1.0 µg/mL of each analyte (prepared in the initial mobile phase conditions) directly into the MS at the chosen LC mobile phase flow rate (approximately 0.6 mL/min). This tune can be done on a mix of the method analytes. The MS/MS parameters (collision gas pressure, collision energy, etc.) are varied until optimal analyte responses are determined. Typically, the carboxylic acids have very similar MS/MS conditions and the sulfonic acids have similar MS/MS conditions.

10.6.2 Establish LC operating parameters that optimize resolution and peak shape. Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted.

10.6.3 Inject a mid-level CAL standard under LC/MS conditions to obtain the retention times of each method analyte. If analyzing for PFTA, ensure that the LC conditions are adequate to prevent co-elution of PFTA and the mobile phase interferants. These interferants have the same precursor and products ions as PFTA, and under faster LC conditions may co-elute with PFTA. Divide the chromatogram into retention time windows each of which contains one or more chromatographic peaks. During MS/MS analysis, fragment a small number of selected precursor ions ([M-H]-) for the analytes in each window and choose the most abundant product ion. For maximum sensitivity, small mass windows of ±0.5 daltons around the product ion mass were used for quantitation. If sufficient sensitivity exists to meet the RL, wider mass ranges may be used to obtain more confirmation ions.

10.6.4 Inject a mid-level CAL standard under optimized LC/MS/MS conditions to ensure that each method analyte is observed in its MS/MS window and that there are at least 10 scans across the peak for optimum precision.

10.6.4.1 If broad, split or fronting peaks are observed for the first two eluting chromatographic peaks (if only two analytes are being analyzed, both must be evaluated), change the initial mobile phase conditions to higher aqueous content until the peak asymmetry ratio for each peak is 0.8 – 1.5. The peak asymmetry factor is calculated as described in Section 9.10.1 on a mid-level CAL standard. The peak asymmetry factor must meet the above criteria for the first two eluting peaks during the IDL and every time a new calibration curve is generated. Modifying the standard or extract composition to more aqueous content to prevent poor shape is not permitted.

10.6.5 Prepare a set of CAL standards as described in Section 8.2.7. The lowest concentration CAL standard must be at or below the RL (2 ng/L), which may depend on system sensitivity. It is recommended that at least four of the CAL standards are at a concentration greater than or equal to the RL.

10.6.6 The LC/MS/MS system is calibrated using the IS technique. Use the LC/MS/MS data system software to generate a linear regression or quadratic calibration curve for each of the analytes. This curve **must always** be forced through zero and may be concentration weighted, if necessary. Forcing zero allows for a better estimate of the background levels of method analytes.

10.6.6.1 The isotopically labeled IS(s) in this method may undergo suppression in the ESI source if the concentration of the co-eluting unlabeled method analyte(s) is too high. The analyte concentration at which suppression may occur can vary depending on the instrument, LC conditions, ESI conditions, IS concentration, etc. To evaluate whether suppression is occurring during calibration, calculate the relative percent difference (*RPD*) for each IS using the equation

$$RPD = \frac{(H - L)}{(H + L) / 2} \times 100$$

10.6.6.2 The RPD calculated above must be <20% for each IS during calibration. If the calculated RPD is >20% for any IS, the analyst must recalibrate at lower analyte concentrations until the IS RPDs are <20%.

10.6.7 CALIBRATION ACCEPTANCE CRITERIA – When quantitated using the initial calibration curve, each calibration point, except the lowest point, for each analyte should calculate to be within 70-130% of its true value. The lowest CAL point should calculate to be within 50-150% of its true value. If these criteria cannot be met, the analyst will have difficulty meeting ongoing QC criteria. It is recommended that corrective action is taken to reanalyze the CAL standards, restrict the range of calibration, or select an alternate method of calibration (forcing the curve through zero is still required).

10.7 CONTINUING CALIBRATION CHECK (CCV) – Minimum daily calibration verification is as follows. Verify the initial calibration at the beginning and end of each group of analyses, and after every tenth sample during analyses. In this context, a “sample” is considered to be a Field Sample. MBs, CCVs, LCSs, MSs, FDs FRBs and MSDs are not counted as samples. The beginning CCV of each analysis batch must be at or below the RL in order to verify instrument sensitivity prior to any analyses. If standards have been prepared such that all low CAL points are not in the same CAL solution, it may be necessary to analyze two CAL standards to meet this requirement. Alternatively, the analyte concentrations in the analyte PDS may be customized to meet this criterion. Subsequent CCVs should alternate between a medium and high concentration CAL standard.

10.7.1 Inject an aliquot of the appropriate concentration CAL standard and analyze with the same conditions used during the initial calibration.

10.7.2 Determine that the absolute areas of the quantitation ions of the IS(s) are within 70-140% of the areas measured in the most recent continuing calibration check, and within 50-150% from the average areas measured during initial calibration. If any of the IS areas has changed by more than these amounts, adjustments must

be made to restore system sensitivity. These adjustments may include cleaning of the MS ion source, or other maintenance as indicated in Section 10.7.4. Major instrument maintenance requires recalibration (Sect 10.6) and verification of sensitivity by analyzing a CCV at or below the RL (Sect 10.7). Control charts are useful aids in documenting system sensitivity changes.

10.7.3 Calculate the concentration of each analyte and SUR in the CCV. The calculated amount for each analyte and SUR for medium and high level CCVs must be within ± 30% of the true value. The calculated amount for the lowest calibration point for each analyte must be within ± 50% and the SUR must be within ± 30% of the true value. If these conditions do not exist, then all data for the problem analyte must be considered invalid, and remedial action should be taken (Sect. 10.7.4) which may require recalibration. Any Field or QC Samples that have been analyzed since the last acceptable calibration verification should be reanalyzed after adequate calibration has been restored, with the following exception. **If the CCV fails because the calculated concentration is greater than 130% (150% for the low-level CCV) for a particular method analyte, and Field Sample extracts show no detection for that method analyte, non-detects may be reported without re-analysis.**

10.7.4 REMEDIAL ACTION – Failure to meet CCV QC performance criteria may require remedial action. Major maintenance, such as cleaning the electrospray probe, atmospheric pressure ionization source, cleaning the mass analyzer, replacing the LC column, etc., requires recalibration (Sect 10.6) and verification of sensitivity by analyzing a CCV at or below the RL (Sect 10.7).

10.8 EXTRACT ANALYSIS

10.8.1 Establish operating conditions equivalent to those summarized in Tables 5-8 of Section 16. Instrument conditions and columns should be optimized prior to the initiation of the IDC.

10.8.2 Establish an appropriate retention time window for each analyte. This should be based on measurements of actual retention time variation for each method analyte in CAL standard solutions analyzed on the LC over the course of time. A value of plus or minus three times the standard deviation of the retention time obtained for each method analyte while establishing the initial calibration and completing the IDC can be used to calculate a suggested window size. However, the experience of the analyst should weigh heavily on the determination of the appropriate retention window size.

10.8.3 Calibrate the system by either the analysis of a calibration curve (Sect. 10.6) or by confirming the initial calibration is still valid by analyzing a CCV as described in Section 10.7. If establishing an initial calibration, complete the IDC as described in Section 13.2.

10.8.4 Begin analyzing Field Samples, including QC samples, at their appropriate frequency by injecting the same size aliquots, under the same conditions used to analyze the CAL standards.

10.8.5 At the conclusion of data acquisition, use the same software that was used in the calibration procedure to identify peaks of interest in predetermined retention time windows. Use the data system software to examine the ion abundances of the peaks in the chromatogram. Identify an analyte by comparison of its retention time with that of the corresponding method analyte peak in a reference standard.

10.8.6 Comparison of the MS/MS mass spectra is not particularly useful given the limited ±0.5 dalton mass range around a single product ion for each method analyte.

10.8.7 The analyst must not extrapolate beyond the established calibration range. If an analyte peak area exceeds the range of the initial calibration curve, the extract may be diluted with 96%:4% vol/vol) methanol:water solution and the appropriate amount of IS added to match the original concentration. Re-inject the diluted extract. Incorporate the dilution factor into the final concentration calculations. Acceptable SUR performance (Sect. 9.5.1.1) should be determined from the undiluted sample extract. The resulting data should be documented as a dilution, with an increased RL.

11. Data Evaluation, Calculations and Reporting

11.1 Complete chromatographic resolution is not necessary for accurate and precise measurements of analyte concentrations using MS/MS. In validating this method, concentrations were calculated by measuring the product ions listed in Table 8. Other ions may be selected at the discretion of the analyst.

11.2 Calculate analyte and SUR concentrations using the multipoint calibration established in Section 10.6. Do not use daily calibration verification data to quantitate analytes in samples. Adjust final analyte concentrations to reflect the actual sample volume determined in Section 10.5.

11.3 Prior to reporting the data, the chromatogram should be reviewed for any incorrect peak identification or poor integration.

11.4 PFHxS, PFOS, NMeFOSAA, and NEtFOSAA have multiple chromatographic peaks using the LC conditions in Table 5 due to the linear and branch isomers of these compounds (Sect. 10.6.4.1). The areas of all the linear and branched isomer peaks observed in the CAL standards for each of these analytes must be summed and the concentrations reported as a total for each of these analytes.

11.5 Calculations must utilize all available digits of precision, but final reported concentrations should be rounded to an appropriate number of significant figures (one digit of uncertainty), typically two, and not more than three significant figures.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 Section 9.0 outlines sample batch QC acceptance criteria. If non-compliant organic compound results are to be reported, the Organic Section Head and/or the Laboratory Director, and the Operations Manager must approve the reporting of these results. The laboratory Project Manager shall be notified and may choose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst, Data Reviewer, or Department Supervisor performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

12.2 All results for the organic compounds of interest are reportable without qualification if extraction and analytical holding times are met, preservation requirements (including cooler temperatures) are met, all QC criteria as defined in Section 9 are met, and matrix interference is not suspected during extraction or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-extraction and/or re-analysis.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

13.1.1 The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

13.2.1 The IDC must be successfully performed prior to analyzing any Field Samples. Prior to conducting the IDC, the analyst must first generate an acceptable Initial Calibration following the procedure outlined in Section 10.6.

13.2.2 INITIAL DEMONSTRATION OF LOW SYSTEM BACKGROUND – Any time a new lot of SPE cartridges, solvents, centrifuge tubes, disposable pipets, and autosampler vials are used, it must be demonstrated that an MB is reasonably free of contamination and that the criteria in Section 9.2.1 are met. If an automated extraction system is used, an MB should be extracted on each port to ensure that all the valves and tubing are free from potential PFAS contamination.

13.2.3 INITIAL DEMONSTRATION OF PRECISION (IDP) – Prepare, extract, and analyze four to seven replicate LCSs fortified near the midrange of the initial calibration curve according to the procedure described in Section 10. Sample preservatives as described in Section 6.2.1 must be added to these samples. The relative standard deviation (RSD) of the results of the replicate analyses must be less than 20%.

13.2.4 INITIAL DEMONSTRATION OF ACCURACY (IDA) – Using the same set of replicate data generated for Section 13.2.3, calculate average recovery. The average recovery of the replicate values must be within ± 30% of the true value.

13.2.5 INITIAL DEMONSTRATION OF PEAK ASYMMETRY FACTOR – Peak asymmetry factors must be calculated using the equation in Section 9.10.1 for the first two eluting peaks (if only two analytes are being analyzed, both must be evaluated) in a mid-level CAL standard. The peak asymmetry factors must fall in the range of 0.8 to 1.5. See guidance in Section 10.6.4.1 if the calculated peak asymmetry factors do not meet the criteria.

13.2.6 Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.7 The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

14.1 Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

14.2 This method utilizes SPE to extract analytes from water. It requires the use of very small volumes of organic solvent and very small quantities of pure analytes, thereby minimizing the potential hazards to both the analyst and the environment as compared to the use of large volumes of organic solvents in conventional liquid-liquid extractions.

14.3 The analytical procedures described in this method generate relatively small amounts of waste since only small amounts of reagents and solvents are used. The matrices of concern are finished drinking water or source water. However, laboratory waste management practices must be conducted consistent with all applicable rules and regulations, and that laboratories protect the air, water, and land by minimizing and controlling all releases from fume hoods and bench operations. Also, compliance is required with any sewage discharge permits and regulations, particularly the hazardous waste identification rules and land disposal restrictions.

15. Referenced Documents

15.1 Chemical Hygiene Plan – ID 2124

15.2 SOP ID 1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

15.3 SOP ID 1739 Demonstration of Capability (DOC) Generation SOP

15.4 SOP ID 1728 Hazardous Waste Management and Disposal SOP

16. Attachments

Table 6: LC Method Conditions

Time (min)	2 mM Ammonium Acetate (5:95 MeOH/H_2O)	2 mM Ammonium Acetate (100% Methanol)
Initial	100.0	0.0
1.0	100.0	0.0
1.1	85.0	15.0
5.8	20.0	80.0
5.9	0.0	100.0
6.9	100.0	0.0
7	100.0	0.0
Waters Aquity UPLC ® $BEHC_{18}$ 2.1 x 50 mm packed with 1.7 µm BEH C_{18} stationary phase Flow rate of 0.6 mL/min 2 µL injection		

Table 7: ESI-MS Method Conditions

ESI Conditions	
Polarity	Negative ion
Ion Spray voltage	-4500 V
Curtain Gas	30.0 PSI
Collision Gas	9.0 PSI
Ion Source Gas 1	30.0 PSI
Ion Source Gas 2	50.0 PSI
Ion Source Temperature	500 ºC

Table 8: Method Analyte Source, Retention Times (RTs), and IS References

Analyte	Peak #	IS# Ref
PFBS	1	2
PFHxA	3	1
HFPO-DA	5	1
PFHpA	6	1
PFHxS	7	2
ADONA	8	1
PFOA	10	1
PFNA	11	1
PFOS	12	2
PFDA	14	1
9CL-PF3ONS	15	2
NMeFOSAA	17	3
PFUnA	18	1
NEtFOSAA	20	3
PFDoA	21	1
11CL-PFOUdS	22	2
PFTrDA	23	1
PFTA	24	1
^{13}C-PFHxA	2	1
^{13}C-HFPO-DA	4	1
^{13}C-PFDA	13	1
d_5-NEtFOSAA	19	3
^{13}C-PFOA-IS#1	9	-
^{13}C-PFOS-IS#2	10	-
d_3-NMeFOSAA-IS#3	16	-

Table 9: MS/MS Method Conditions

Segment[a]	Analyte	Precursor Ion [b] (m/z)	Product Ion [b,c] (m/z)
1	PFBS	299	80
2	PFHxA	313	269
4	HFPO-DA	285	169
5	PFHpA	363	319
6	PFHxS [e]	399	80
7	ADONA	377	251
9	PFOA	413	369
10	PFNA	463	419
11	9CL-PF3ONS	531	351
13	PFOS [e]	499	80
15	PFDA	513	469
17	NMeFOSAA [e]	570	419
19	NEtFOSAA [e]	584	419
20	11CL-PFOUdS	631	451
21	PFUnA	563	519
22	PFDoA	613	569
23	PFTrDA	663	619
24	PFTA	713	669
2	^{13}C-PFHxA	315	270
3	^{13}C-HFPO-DA	287	169
14	^{13}C-PFDA	515	470
16	d_5-NEtFOSAA	589	419
8	^{13}C-PFOA	415	370
12	^{13}C-PFOS	503	80
18	d_3-NMeFOSAA	573	419

[a] Segments are time durations in which single scan events occur; segments overlap where R.T. dictate.
[b] Precursor and product ions listed in this table are nominal masses. During MS and MS/MS optimization, the analyst should determine the precursor and product ion masses to one decimal place by locating the apex of the mass spectral peak place. These precursor and product ion masses (with one decimal place) should be used in the MS/MS method for all analyses.
[c] Ions used for quantitation purposes.
[d] Nitrogen used as collision gas at a flow rate of 9 PSI
[e] Analyte has multiple resolved chromatographic peaks due to linear and branched isomers. All peaks summed for quantitation purposes.

pH, Liquid Samples

Reference Methods: **Method 9040C,** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IIIB, 2004.

SM 4500H$^+$-B, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

1. Scope and Application

Matrices: This method is used to measure the pH of aqueous wastes and those multiphase wastes where the aqueous phase constitutes at least 20% of the total volume of the waste.

The corrosivity of concentrated acids and bases, or of concentrated acids and bases mixed with inert substances, cannot be measured. The pH measurement requires some water content.

Definitions: See Alpha Laboratories Quality Manual Appendix A

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the pH Meter and in the interpretation of pH data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The pH of the sample is determined electrometrically using a temperature compensating combination electrode.

2.1 Method Modifications from Reference

The pH meter that is utilized will compensate for the temperature of the sample. Therefore, the temperature is not reported with the data. However, the sample is analyzed at room temperature.

3. Reporting Limits

None.

4. Interferences

4.1 Errors will occur when the electrode becomes coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can (1) be cleaned with an ultrasonic bath, or (2) be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water, or (3) be cleaned per the manufacturer's instructions.

4.2 Samples with a very low or very high pH may give incorrect readings on the meter.

For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode.

Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

4.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by rinsing with distilled water. An additional treatment with hydrochloric acid (1:10) may be necessary to remove any remaining film.

4.4 Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference is controlled with instruments having temperature compensation. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This error is sample-dependent and cannot be controlled. However, prior to analysis the samples are brought to room temperature (20 – 25 °C).

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in plastic or glass containers. The minimum volume required is 100mL.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are to be analyzed as soon as possible, within 24 hours of collection. Samples are stored at 4 ±2 °C. However, prior to analysis, samples are allowed to come to room temperature.

7. Equipment and Supplies

7.1 pH Meter: Laboratory benchtop model with automatic temperature compensation.

7.1.1 Combination electrode: Incorporates measuring and referenced functions; solid, gel-type filling material. For use with benchtop pH Meter (Section 7.1).

7.2 Titrator: T70 Mettler Toledo, with Inmontion Flex Autosampler and Computer with LabX software package.

7.2.1 Combination electrode: Mettler Toledo, Type DG111-SC, Part #00089596. For use with Titrator (Section 7.2).

7.3 Plastic Cups: Mettler Toledo brand, 100mL volume.

7.4 Magnetic stirrer

7.5 Teflon-coated stirring bar

7.6 Glass Beakers

7.7 Kimwipes

7.8 Plastic cups

8. Reagents and Standards

8.1 pH Buffers: Commercially available pH 4 (or 4.01), pH 7 (two sources), pH 10 (or 10.01). Buffers must have been validated by comparison to NIST standards. Certificate of analysis is kept on file.

9. Quality Control

9.1 Blank(s)

Not applicable.

9.2 Laboratory Control Sample (LCS)

One LCS is analyzed with each batch 20 of samples or less. It is a pH 7 buffer of a different source than the calibration buffer. Results must be within ±0.05.

When the LCS fails the acceptance criteria, the analyst must locate and correct the source of the problem and repeat the calibration. Repeated failure confirms a general problem with the measurement system or analytical technique of the analyst.

9.3 Initial Calibration Verification (ICV)

Refer to LCS Section 9.2.

9.4 Continuing Calibration Verification (CCV)

The calibration is also verified at the end of the sample sequence and after every 10 samples. The results of the continuing calibration verification must be within ± 2% (6.86-7.14). If this criterion is not met, the titrator or meter must be re-calibrated and any samples analyzed since the last acceptable CCV are reanalyzed

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

One sample is analyzed in duplicate per batch of 20 samples or less. %RPD for Duplicates must be within in-house acceptance criteria determined from Control Charts. If the result is outside of the criteria, the duplicate result may be narrated.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Calibration
- Initial Calibration Verification (LCS)
- Sample analysis, 1-10 samples
- Duplicate analysis
- Continuing Calibration Verification (CCV), analyzed every 10 samples and at the end of analytical run.

10. Procedure

10.1 Equipment Set-Up

Thoroughly rinse and gently wipe the electrode prior to measuring the pH of buffers for calibration.

10.2 Initial Calibration

10.2.1 Benchtop Meter

The benchtop pH meter is calibrated on a daily basis using three pH buffers (Section 8.1). Follow manufacturer's instructions for a 3-point calibration of the pH meter. The results of the calibration must be recorded in the pH Calibration Log including correlation coefficient. Slope of the meter must fall within 95% to 105%.

Initial Calibration Verification: Prior to sample analysis, the calibration is initially verified by using a pH 7 buffer from a source other than the source used for calibration. The results of the initial verification must be within ± 0.05. If this criterion is not met, the meter must be re-calibrated.

10.2.2 Titrator

The pH titrator is calibrated on a daily basis using three pH buffers (Section 8.1).

- Pour 20-25mL of pH 4, 7 and 10 buffers (Section 8.1) into separate plastic cups (Section 7.3). Record the Lot numbers of the buffers into the Laboratory Notebook.
- Place the cups onto the Inmontion Flex Autosampler in that order.
- Ensure the Titrator is in "Remote Control" mode.
- Open LabX software package. Select "Open View" on the Titrator. Choose the appropriate method: Method 1 – PH Calibration.
- With a right-click on the mouse, select "Create Task". Type in the series ID (Cal-MM/DD/YYYY), and the sample sequence: PH 4, PH 7, PH 10. Select the "Save" button.
- The Series ID will appear in the "Samples" column. Click on that Series ID and using a right mouse click, select "Run" from the drop down menu.

After the run is complete, check for printout results. . Slope of the meter must fall within 95% to 105%.

Initial Calibration Verification: Prior to sample analysis, the calibration is initially verified by using a pH 7 buffer from a source other than the source used for calibration. The results of the initial calibration verification (LCS) must be within ±2%. If this criterion is not met, the Titrator must be recalibrated before any samples are analyzed.

10.3 Equipment Operation and Sample Processing

10.3.1 Benchtop Meter

Place the sample in a clean glass beaker or plastic cup using a sufficient volume to cover the sensing elements of the electrode and to give adequate clearance for the magnetic stirring bar.

Thoroughly rinse and gently wipe the electrode prior to measuring the pH of samples. Immerse the electrode into the sample beaker and gently stir at a constant rate to provide homogeneity and suspension of solids. Note and record the sample pH when meter has stabilized. Record temperature at which pH is recorded.

10.3.2 Titrator

- Pour 20-25mL of each sample into a separate, labeled plastic cup (Section 7.3).
- Place the cups onto the Inmontion Flex Autosampler.
- Choose the appropriate method: Method 2 – PH Measurement.
- With a right-click on the mouse, select “Create Task”. Type in the series ID (LCS-MM/DD/YYYY), and the sample sequence. Make sure the order of the samples on the autosampler matches the sample sequence. Select the “Save” button.
- The Series ID will appear in the “Samples” column. Click on that Series ID and using a right mouse click, select “Run” from the drop down menu.
- After the run is complete, check for printout results.

10.4 Continuing Calibration

10.4.1 Benchtop Meter

The meter calibration is also verified at the end of the sample sequence and after every 10 samples. The results of the continuing calibration verification must be within ± 2%. If this criterion is not met, the meter must be re-calibrated and any samples analyzed since the last acceptable CCV are reanalyzed.

10.4.2 Titrator

The titrator calibration is also verified at the end of the sample sequence and after every 10 samples. The results of the continuing calibration verification must be within ± 2%. If this criterion is not met, the titrator must be re-calibrated and any samples analyzed since the last acceptable CCV are reanalyzed.

10.5 Preventative Maintenance

The combination electrode on the benchtop meter is rinsed with DI and gently dried with a KimWipe between sample readings. Both electrodes are stored in buffer 7 when not in use.

11. Data Evaluation, Calculations and Reporting

pH is read directly from the pH benchtop meter or Titrator. No calculations are necessary.

For pH readings of less than 1, the reported result is: pH <1.

For pH readings greater than 10, report result in 3 significant figures.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

The combination electrode is replaced as necessary.

Review of standards for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the LCS recovery falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. Re-calibration of the meter or titrator is necessary prior to sample analysis.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

Not applicable to this method.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734, 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1734, 1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

pH, Soil and Waste

References: **Method 9045D, Soil and Waste pH,** Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 4, November 2004.

Method 9040C, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 3, November 2004.

NJDEP Site Remediation Program, Data of Known Quality Protocol, Version 1, April 2014

1. Scope and Application

Matrices: Soils and wastes, including solids, sludges, or non-aqueous liquids.

Definitions: See Alpha Laboratories Quality Manual Appendix A

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the pH meter and in the interpretation of pH data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The sample is mixed with reagent water, and the pH of the resulting aqueous solution is measured.

2.1 Method Modifications from Reference

The pH meter that is utilized will compensate for the temperature of the sample. Therefore, the temperature is not reported with the data. However, the sample is analyzed at room temperature. Only one aliquot is used for measurement.

3. Reporting Limits

None.

4. Interferences

4.1 Errors will occur when the electrode becomes coated. If an electrode becomes coated with an oily material that will not rinse free, the electrode can (1) be cleaned with an ultrasonic bath, or (2) be washed with detergent, rinsed several times with water, placed in 1:10 HCl so that the lower third of the electrode is submerged, and then thoroughly rinsed with water, or (3) be cleaned per the manufacturer's instructions.

4.2 Samples with a very low or very high pH may give incorrect readings on the meter.

For samples with a true pH of >10, the measured pH may be incorrectly low. This error can be minimized by using a low-sodium-error electrode.
Strong acid solutions, with a true pH of <1, may give incorrectly high pH measurements.

4.3 Coatings of oily material or particulate matter can impair electrode response. These coatings can usually be removed by gentle wiping or detergent washing, followed by rinsing with distilled water. An additional treatment with hydrochloric acid (1:10) may be necessary to remove any remaining film.

4.4 Temperature effects on the electrometric determination of pH arise from two sources. The first is caused by the change in electrode output at various temperatures. This interference should be controlled with instruments having temperature compensation. The second source of temperature effects is the change of pH due to changes in the sample as the temperature changes. This error is sample-dependent and cannot be controlled. However, prior to analysis the samples are brought to room temperature (20 – 25 °C).

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in 4-ounce glass jars.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are to be analyzed as soon as possible after sampling. Samples are stored at 4 ± 2°C. Samples that are to be run in conjunction with NJ-Hex DAK acceptance criteria must be analyzed within 24 hours of Hex sample preparation.

7. Equipment and Supplies

7.1 pH Meter: Laboratory benchtop model with automatic temperature compensation.

7.1.1 Combination electrode: Incorporates measuring and referenced functions; solid, gel-type filling material. For use with benchtop pH Meter (Section 7.1).

7.3 Beakers: 50mL glass or plastic

7.4 Magnetic Stirrer

7.5 Teflon-coated stirring bar

7.6 Analytical Balance: Capable of weighing 0.1g

7.7 Kimwipes

8. Reagents and Standards

8.1 Reagent Water: All references to water in this method refer to reagent water.

8.2 pH Buffers: Commercially available pH 4 (or 4.01), pH 7, pH 10 (or 10.01). In addition, an alternate source of pH 7 Buffer is necessary. All Buffers must have been validated by comparison to NIST standards. Certificate of analysis is required. Buffers are stored at room temperature and expire upon manufacturer's specified date.

9. Quality Control

9.1 Blank(s)

Not applicable.

9.2 Laboratory Control Sample (LCS)

One LCS is analyzed with each batch of 20 samples or less. It is a pH 7 buffer of a different source than the calibration buffer. Results must be within ± 0.05.

9.3 Initial Calibration Verification (ICV)

Refer to LCS Section 9.2.

9.4 Continuing Calibration Verification (CCV)

A CCV of pH 7 is analyzed at the end of the analytical run. Results must be within ± 0.2 units.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

One duplicate sample is analyzed per batch of 20 samples or less

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Calibration
- Calibration Verification (LCS)
- Sample analysis
- Duplicate Analysis
- Calibration Verification (CCV)

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation

In a 50mL beaker, weigh 20 grams of soil; record the weight. Add 20mL of reagent water, cover and continuously stir the suspension for 30 minutes, using a teflon stir bar on a magnetic stirrer. Let samples stand for 1 hour before measurement.

Additional dilutions are permissible if working with hydroscopic soils, salts or other problematic matrices.

NOTE: If the sample is hygroscopic and absorbs all the reagent water, begin again using 20 grams of sample and 40mL of reagent water.

NOTE: If the supernatant is multiphasic, decant the oily phase and measure the pH of the aqueous base. The electrode may need to be cleaned (Section 4.1) if it becomes coated with an oily material.

10.2 Initial Calibration

The pH meter is calibrated on a daily basis using three pH buffers (Section 8.1). Follow manufacturer's instructions for a 3-point calibration of the pH meter. The results of the calibration must be recorded in the pH Calibration Log.

10.3 Equipment Operation and Sample Processing

10.3.1 Allow the sample suspension to stand for about 30 minutes to allow suspended material to settle out from the suspension.

10.3.2 Immerse the pH electrode just below the suspension and allow pH meter to stabilize. Note and record the sample pH in the Laboratory notebook. Report sampletemperature at the time of measurement.

10.3.3 Rinse the electrode thoroughly between samples, using reagent water.

10.4 Continuing Calibration

Prior to sample analysis, the calibration is initially verified by using a pH 7 buffer from a source other than the source used for calibration. The results must be within ± 0.2. If this criterion is not met, the meter must be re-calibrated before sample analysis can begin.

10.5 Preventative Maintenance

The pH probe is rinsed with DI and gently dried with a KimWipe between sample readings.

11. Data Evaluation, Calculations and Reporting

pH is read directly from the pH meter. No calculations are necessary.

For pH readings of less than 1, the reported result is: pH <1.

For pH readings greater than 10, report result in 3 significant figures.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the maintenance logbook.

The pH electrode is replaced as necessary.

Review of standards for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the LCS or CCV recovery falls outside the designated acceptance range, the laboratory performance for the parameter is judged to be out of control, and the problem must be immediately identified and corrected. Re-calibration of the meter is necessary prior to sample analysis. All samples analyzed since the last acceptable QC standard must be reanalyzed following re-calibration of the meter.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

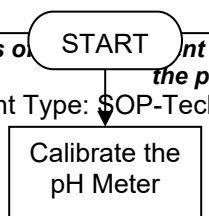
SOP/1728 Waste Management and Disposal SOP

16. Attachments

Flow Chart: Soil and Waste pH

Flow Chart:

Soil and Waste pH



YES

NO

NO

YES

Phenol (Total)

Reference Methods: **EPA 9065**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1986.

EPA 420.1, Methods for the Chemical Analysis of Water and Wastes, EPA 600/ 4-79-020, 1983.

1. Scope and Application

Matrices: This method is applicable to the analysis of ground water, drinking, surface, and saline waters, domestic and industrial wastes, and soils.

Definitions: See Alpha Analytical Quality Manual Appendix A

Phenols, defined as hydroxy derivatives of benzene and its condensed nuclei, may occur in domestic and industrial wastewaters and in drinking water supplies. Chlorination of such waters may produce odoriferous and objectionable-tasting chlorophenols, which may include o-chlorophenol, p-chlorophenol, 2,6-dichlorophenol, and 2,4-dichlorophenol. Phenol removal processes in water treatment include superchlorination, chlorine dioxide or chlorine ammonia treatment, ozonation, and activated carbon adsorption.

This method uses the 4-aminoantipyrine colorimetric method that determines phenol, the ortho- and metasubstituted phenols, and, under proper pH conditions, those parasubstituted phenols in which the substitution is a carboxyl, halogen, methoxyl, or sulfonic acid group.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the distillation units and the spectrophotometer, and in the interpretation of spectrophotometer data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The phenols are distilled at a more or less constant rate from the nonvolatile impurities. The rate of volatilization of the phenols is gradual, so that the volume of the distillate must equal that of the sample being distilled. The use of $CuSO_4$ during distillation of an acidic sample permits the formation of cupric sulfide without subsequent decomposition to H_2S. The acidic solution also prevents the precipitation of cupric solution hydroxide, which acts as an oxidizing agent toward phenols.

The steam-distilled phenols react with 4-aminoantipyrine at a pH of 10.0 in the presence of potassium ferricyanide to form a colored antipyrine dye. This dye is kept in an aqueous solution and the absorbance is measured at 510nm

2.1 Method Modifications from Reference

The aqueous sample size has been reduced from 500mL to 250mL. Consequently, the volume of reagents added for distillation has been reduced by half.

Method 420.1 specify 24h holding time; lab is using 28 days holding time per method 9065; all samples are acidified.

3. Reporting Limits

The Reported Detection Limit for this analysis is 0.03mg phenol/L for liquids and 0.75mg/Kg for soils.

4. Interferences

4.1 Instrumental

Distillate turbidity will interfere with Spectrophotometer reading. Refer to Section 10.3.1.3.

4.2 Parameters

4.2.1 Domestic and industrial wastewaters may contain such interferences as phenol-decomposing bacteria, oxidizing and reducing substances, and alkaline pH values. Biological degradation is inhibited by the addition of $CuSO_4$ to the sample. Acidification with H_2SO_4 assures the presence of the copper ion and eliminates any chemical changes resulting from the presence of strong alkaline conditions.

4.2.2 Some of the treatment procedures used for the removal of interferences before analysis may result in an unavoidable loss of certain types of phenols. Consequently, some highly contaminated wastewaters may require specialized screening techniques for elimination of interferences and for quantitative recovery of the phenolic compounds.

4.2.3 Color response of phenolic materials with 4-aminoantipyrine is not the same for all compounds. Because phenolic-type wastes usually contain a variety of phenols, it is not possible to duplicate a mixture of phenols to be used as a standard. For this reason phenol has been selected as a standard and any color produced by the reaction of other phenolic compounds is reported as phenol. This value will represent the minimum concentration of phenolic compounds present in the sample.

4.2.4 Interferences from sulfur compounds are eliminated by acidifying the sample to a pH of < 4 with H_2SO_4 and aerating briefly by stirring.

4.2.5 Oxidizing agents such as chlorine, detected by the liberation of iodine upon acidification in the presence of potassium iodide, are removed immediately after sampling by the addition of an excess of ferrous ammonium sulfate. If chlorine is not removed, the phenolic compounds may be partially oxidized and the results may be low.

4.2.6 Oils and tars contain phenol, and if present in the sample would cause interference. Refer to the original method for guidance in the extraction of oils and tars from the sample.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Collect aqueous samples in a 1L Amber Glass bottle and soil samples in an 8-ounce glass jar.

6.2 Sample Preservation

Phenols in concentrations usually encountered in wastewaters are subject to biological and chemical oxidation. If analysis cannot be done within 4 hours after collection, acidify to a pH <4 with H_2SO_4. If H_2S or SO_2 is known to be present, briefly aerate or stir the sample with caution.

Soil samples are not preserved.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Keep the sample refrigerated at 4±2ºC. Analyze the preserved aqueous samples and soil samples within 28 days after collection.

Soil distillates must be analyzed within 24 hours of distillation. Soil distillates are kept refrigerated at 4±2ºC if not immediately analyzed.

7. Equipment and Supplies

7.1 Distillation apparatus: All glass, consisting of 500mL Pyrex distilling flasks with condenser and heating apparatus. Steam cleaned, per Section 10.1.1.

7.2 pH meter, calibrated.

7.3 Spectrophotometer: for use at 510nm.

7.4 Volumetric Pipettes: 1mL, 2mL, 3mL

7.5 Volumetric Flasks: 100mL, 200mL

7.6 Glass Beakers: 250mL or greater

7.7 Chiller.

7.8 Analytical Balance.

7.9 Syringes 10-20 ml

7.10 Acrodisks.

7.11 pH Paper

7.12 Boiling Chips

7.13 Timer

8. Standards and Reagents

8.1 DI Water.

8.2 1000ppm LCS Stock Phenol Solution: Commercially available with a certificate of analysis. Store at room temperature. Expires upon manufacturer's specified date.

8.2.1 200ppm LCS Working Solution: Dilute 20mL of 1000ppm LCS Stock Phenol Solution (Section 8.2) to 100mL with DI. Store at room temperature. Expires 3 months from date of preparation.

8.3 1000ppm Stock Calibration Standard: Commercially available with a certificate of analysis; must be purchased from a different source than the 1000ppm LCS Stock Phenol Solution (Section 8.2). Store at room temperature. Expires upon manufacturer's specified date.

8.3.1 10ppm Working Calibration Standard: Pipet 10mL of the 1000ppm Stock Calibration Standard (Section 8.3) into a 1L volumetric flask and bring to volume with DI. Prepare fresh on each day of use.

8.3.2 200ppm Spiking Solution: Pipet 20mL of the 1000ppm Stock Calibration Standard (Section 8.3) into a 100mL volumetric flask and bring to volume with DI. Store at room temperature. Expires 3 months from date of preparation.

8.4 Copper sulfate solution: Dissolve 100g $CuSO_4 \cdot 5H_2O$ in 1L of DI. Store at room temperature. Expires one year from date of preparation.

8.5 Sulfuric acid, H_2SO_4: Concentrated

8.6 Buffer Solution: Dissolve 16.9g of NH_4Cl in 143mL concentrated NH_4OH and dilute to 250mL with DI water. Typically, 2mL of Buffer adjusts 100mL of distillate to pH 10. Store at room temperature. Expires 3 months from date of preparation.

8.7 Aminoantipyrine solution: Dissolve 2g of 4-aminoantipyrine (4-AAP) in DI and dilute to 100mL. Prepare a fresh solution on each day of use.

8.8 Potassium ferricyanide solution: Dissolve 8.0g K3Fe $(CN)_6$ in DI and dilute to 100mL. Filter if necessary. Store at room temperature. Prepare fresh each week of use.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

One distillation blank per batch of 10 samples or less. A Distillation Blank will determine if any contamination has occurred. (Section 10.1.2.2)

9.2 Laboratory Control Sample (LCS)

Analysis of a LCS/ICV and all samples in the batch takes place after the spectrophotometer is set to zero using an undistilled Blank. One LCS is run per batch of 10 samples or less. (Section 10.1.2.3)

The LCS recovery must be within ± 30% for aqueous samples and ± 35% for solid samples. If LCS recovery is outside acceptance criteria, the entire batch is re-distilled and reanalyzed.

The ICV must be within +/- 10% of the true value before reading any sample on the spectrophotometer. If the ICV recovery is not within the limits, then a new calibration curve is generated.

9.3 Initial Calibration Verification (ICV)

Refer to Section 9.2.

9.4 Continuing Calibration Verification (CCV)

After every 10 or less samples, or for every hour of continuous analysis, verify the calibration curve by coloring and reading the absorbance of an undistilled phenol standard (CCV). The CCV must recover within +/- 10% of the true value. If the CCV recovery is not within the limits, CCV is re-poured and re-analyzed. If the CCV failure continues, then a new calibration curve is generated (Section 10.2.1).

9.5 Matrix Spike

Prep and analyze one matrix spike per batch of 10 samples or less. (Section 10.1.2.4) The spike recovery for aqueous samples must be within ± 30% and within ± 35% for solid samples. If recovery is outside of the acceptance criteria, the matrix spike is re-distilled and reanalyzed to confirm matrix interference. If interference is confirmed, a narrative is submitted to be included on the Client report.

9.6 Laboratory Duplicate

Prep and analyze one duplicate per batch of 10 samples or less. (Section 10.1.2.5) The %RPD for aqueous samples must be within ± 20% and within ± 30% for solid samples. If %RPD is outside of acceptance criteria, the sample and its duplicate are re-distilled and reanalyzed. If failure continues, a narrative is submitted to be included on the Client report.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Glassware preparation
- Calibration curve generation
- Distillation
- Coloring
  - Blank
  - ICV/LCS
  - Samples and QC Samples
  - CCV
- Reading of absorbances
- Calculations

10. Procedure

10.1 Equipment Set-up

10.1.1 Preparation of Distillation Glassware

Prior to use, steam clean the distillation apparatus using 150mL of DI water plus 1mL concentrated H_2SO_4. Add boiling chips and assemble the stills. Turn on the chiller and heating units. Distill over about 100mL distillate. After stills cool, discard the distillate appropriately, then rinse the flask, the arm, and the column with DI.

10.1.2 Distillation Set-up

10.1.2.1 Samples

10.1.2.1.1 Liquid Samples

Measure 250mL of well-homogenized sample and 50mL of DI into a 500mL distilling flask. Check the pH using pH paper. Adjust the pH to <4 using H_2SO_4 if necessary.

10.1.2.1.2 Solid Samples

Weigh approximately 10g of well-homogenized soil sample using an analytical balance. Record the exact weight in a logbook.

Measure 300mL of DI into a 500mL distilling flask and add the 10g of soil sample. Check the pH using pH paper. Adjust the pH to <4 using H_2SO_4 if necessary.

10.1.2.2 Blank

Measure 300mL of DI into a 500mL distilling flask. Adjust the pH to <4 using H_2SO_4 if necessary.

For a batch of solid samples, also weigh approximately 10g of boiling chips(Section 8.9). Record the exact weight in the logbook and add to the distilling flask.

Prepare one Blank per batch of 10 samples or less.

10.1.2.3 Laboratory Control Sample (LCS) / Initial Calibration Verification (ICV)

Measure 300mL of DI into a 500mL distilling flask and add 1mL of 200ppm LCS Working Solution (Section 8.2.1). Adjust the pH to <4 using H_2SO_4 if necessary. Final concentration is 0.8ppm.

For a batch of solid samples, also weigh approximately 10g of boiling chips (Section 8.9). Record the exact weight in the logbook and add to the distilling flask 300mL of DI and add 1mL of 200ppm LCS Working Solution (Section 8.2.1). Adjust the pH to <4 using H_2SO_4 if necessary. Final concentration is 0.8ppm

Prepare one LCS/ICV per batch of 10 samples or less.

10.1.2.4 Matrix Spike

Measure 250mL of sample and 50mL of DI water into a 500mL distilling flask and add 1mL of 200ppm Spiking Solution (Section 8.3.2). If the pH is not <4, adjust using H_2SO_4. Final concentration is 0.8ppm. Prepare one matrix spike per batch of 10 samples or less.
For a batch of solid samples, also weigh approximately 10g of sample Record the exact weight in the logbook and add to the distilling flask. Measure 300mL of DI and add 1mL of 200ppm Spiking Solution (Section 8.3.2).

10.1.2.5 Sample Duplicate

Prepare one sample in duplicate per batch of 10 samples or less, as per Section 10.1.2.1.

10.2 Initial Calibration

10.2.1 Calibration Curve Generation: A calibration curve is prepared fresh on each day of analysis, with four calibration standards and a blank

10.2.1.1 Preparing the Standards

Using the 10ppm working calibration standard (Section 8.3.1), prepare the following standards in volumetric flasks using DI water:

10ppm Working Calibration Standard (mL)	Final Volume (Volumetric Flask – mL)	Concentration (mg/L)
0.0	100	0.0
0.5	200	0.025

1.0	100	0.10
10.0	100	1.0
25.0	100	2.5

10.2.1.2 Coloring and Reading the Standards

10.2.1.2.1 To 100mL of standard, add 2mL of buffer solution (Section 8.6) and mix. The pH of the standards must be 10. Additional buffer solution may be added if necessary.

10.2.1.2.2 Add 2.0mL aminoantipyrine solution (Section 8.7) and mix.

10.2.1.2.3 Add 2.0mL of potassium ferricyanide solution (Section 8.8) and mix.

10.2.1.2.4 After 15 minutes, zero the spectrophotometer at 510nm with the 0.0ppm standard (Blank) sample in a 2.5 cm cell.

10.2.1.2.5 Read the standard absorbances on the Spectrophotometer at 510nm, in a 2.5cm cell and record in the laboratory notebook.

10.2.1.3 Preparing a Standard Curve

Plot a curve of the absorbance values of the standards versus the corresponding phenol concentrations. Correlation coefficient will be calculated. All calibration points are back calculated on excel and should be within 10% from true concentration, except lowest point of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 50% and correlation coefficient must be > or = 0.995. If calibration curve criteria failed, then new calibration standards have to be re-analyzed and new calibration curve has to be generated and all calibration points have to be recalculated to meet criteria. If second calibration curve fails acceptance criteria, then analysis have to stopped, problem has to be identified; new calibration standards made and analyzed.

10.3 Equipment Operation and Sample Processing

10.3.1 Distillation

10.3.1.1 To each distilling flask add one scoop of boiling chips and 3mL of $CuSO_4$ Solution (Section 8.4). Swirl vigorously.

10.3.1.2 Assemble the stills; turn on the chiller and heating units and collect 250mL of distillate into each glass beaker. Record the distillate volume in the laboratory notebook.

NOTE: At this point the distillate can be covered and refrigerated until the next day for the coloring procedure, if desired.

Check pH of distillate; it should be 10+/- 0.2. If pH is outsed of the range, it should be adjusted using Buffer solution (8.6)

10.3.1.3 Turbid Distillate

Initial distillation should purify the sample adequately. Occasionally, however, the distillate is turbid.

NOTE: *In this case, notify the Department Supervisor before proceeding.*

Refer to the original method for guidance in removing turbidity from the distillate; distillate may be filtered prior to colorimetric procedure to reduce turbidity and avoid false positive reading, using syringe and acrodisk

10.3.2 Coloring Procedure

Pour 100mL of distillate into a clean glass beaker. Add the following in order, and mix following each addition:

- 2mL of Buffer Solution (Section 8.6)
- 2mL of 4-AAP (Section 8.7)
- 2mL of $K_3Fe(CN)_6$ (Section 8.8).

10.3.3 Spectrophotometer Reading

10.3.3.1 Color DI water as in 10.3.2 above. After 15 minutes, use this to zero the Spectrophotometer at 510nm using a 2.5cm cell.

10.3.3.2 Read the colored distilled blank. If the Spectrophotometer reading is less than 0.010 absorbance, re-zero the instrument using the colored distilled blank. If the absorbance of the colored distilled blank is 0.010 or greater, then a contamination has occurred and the batch must be re-distilled.

10.3.3.3 Read the colored sample and QC sample distillates on the Spectrophotometer at 510nm in a 2.5cm cell. Record absorbances in the laboratory notebook.

10.3.3.4 If a sample distillate has an absorbance that is greater than that of the highest standard on the calibration curve (Section 10.2.1), dilute a fresh aliquot of distillate with DI water and re-color (Section 10.3.2) the sample distillate. Read again on the spectrophotometer as outlined above.

10.4 Continuing Calibration

After every 10 or less samples, or for every hour of continuous analysis, verify the calibration curve by coloring and reading the absorbance of an undistilled phenol standard (as in Section 10.2.1.2). Samples analyzed for NJ- use mid-point calibration standard for CCV

10.5 Preventive Maintenance

The Spectrophotometer is calibrated on a semi-annual basis by an instrument service company. These records are kept on file.

11. Data Evaluation, Calculations and Reporting

11.1 Calculate the concentration value of the sample directly from the standard curve. (Section 10.2.1.3).

$$\text{Phenol (mg/L)} = \frac{\text{absorbance} - \text{y-intercept}}{\text{slope}} \times \text{Dilution factor}$$

11.2 For liquid samples, calculate mg/L phenol as follows:

$$\text{mg/L phenol} = \mathbf{A} \times \frac{\mathbf{250}}{\mathbf{B}}$$

where:

A = mg/L phenol, from calibration curve (Section 11.1)
B = mL original sample.

11.3 For solid samples, calculate mg/kg phenol as follows:

$$\text{mg/kg phenol} = \mathbf{A} \times \frac{\mathbf{250}}{\mathbf{B}}$$

where:

A = mg/L phenol, from calibration curve (Section 11.1)
B = g original sample.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the LCS recovery falls outside the designated acceptance range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

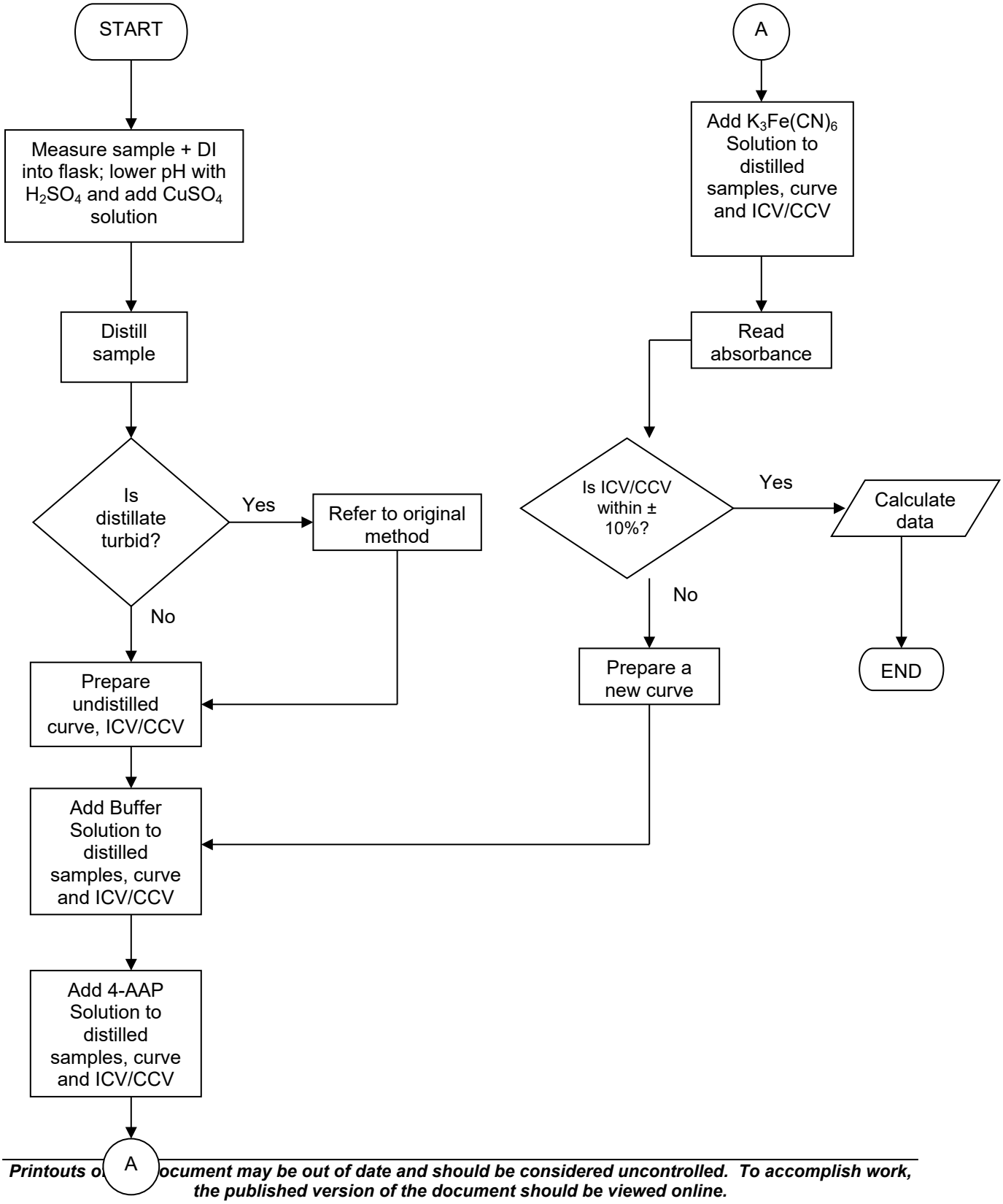
SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

Flow Chart.

Flow Chart
Phenolics (Spectrophotometric, Manual 4-AAP with Distillation)



Polynuclear Aromatic Hydrocarbons (PAHs) by SIM EPA 8270E (M)

Reference: EPA 8270E (Modified). SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update VI (Phase II), June, 2018.

1. Scope and Application

Matrices: This method is used to determine the concentration of Semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, groundwater and wastewater samples

Definitions: Refer to Alpha Analytical Quality Manual.

This method is used to quantitate the neutral, acidic, and basic organic compounds listed in Table 6 that are soluble in methylene chloride and capable of being eluted, without derivatization, from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone.

The following compounds may require special treatment when being determined by this method:

- Pentachlorophenol, is subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of a gas chromatograph/mass spectrometer operating in the selected ion monitoring (SIM) mode. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The samples are introduced into the GC/MS by injecting 1µL of the sample extract (or 2µL for Large Volume Injection, or LVI, method) into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.
The mass spectrometer is operated in the selected ion monitoring (SIM) mode. In this mode of operation, only the characteristic ions of the compounds listed in Table 5 are detected. It is not possible to identify any other compounds that may be present in the sample.

Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing the retention times of the characteristic ions with the retention times of these compounds in the daily check standard. Quantitation is accomplished using the internal standard method, using the average response factors from the initial calibration curve

2.1 Method Modifications from Reference

Operating the mass spectrometer in the SIM mode is a modification from method 8270, which requires operating in the scan mode. This SIM method is used when a project requires lower reporting limits than can be achieved running method 8270 or 625.

3. Reporting Limits

The laboratory follows the procedure found in 40CFR Part 136 to determine the MDL. The method detection limits determined by the laboratory are on file for review.

Table 6 lists our routine reporting limits. Whenever MDL studies are performed, the MDL results are compared with our reporting limits to ensure that the calculated MDLs are equal to or below our reporting limits

4. Interferences

4.1 Only high purity helium is used in the GC system to eliminate this source of possible contamination. The helium (carrier gas) is certified by the gas supplier.

4.2 Preventive instrument maintenance is performed routinely. Section 10.5 details the maintenance steps.

4.3 Contaminated solvents or reagents are also possible sources or contamination. All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free. All of these materials are routinely determined to be free of interferences by analysis of extraction blanks with every extraction batch performed.

4.4 Glassware must be scrupulously cleaned. This procedure is detailed in the Organic Extraction Glassware Cleaning & Handling SOP 1953. Store dry glassware in a clean environment

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves should be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers should be handled in the vented bench area in the GC/MS laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab for future disposal by the Health and Safety Officer. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Flammable solvent bottles must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 1L amber glass jars with Teflon-lined lids. Solid samples are collected in 250mL wide-mouth glass jars with Teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of ≤ 4ºC. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of ≤ 4ºC

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Aqueous samples must be extracted within 7 days of sample collection, solid samples within 14 days of collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph/Mass Spectrometer System:

7.1.1 **Gas Chromatograph, Hewlett Packard 6890 or equivalent:** An analytical system complete with a temperature-programmable gas chromatograph configured for split/splitless-injection and all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source.

7.1.2 **Column:** 30m x 0.25mm ID, 0.25 µm film thickness silicone-coated, fused-silica capillary column (RTX-5MS, Restek), or equivalent.

7.1.3 **Mass Spectrometer, Hewlett Packard 5973 or equivalent:** Scanning in SIM mode using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for Decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 1 when 1 µL of the GC/MS tuning standard is injected through the GC (50ng of DFTPP).

7.1.4 **Data System:** A computer system is interfaced to the Mass Spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows the analyst to search for any GC/MS data file for ions of specific mass and plot such ion abundances versus time or scan number. *HP ChemServer* software is used for data acquisition and *Enviroquant MSD Chemstation version E.02.02* is used for data reduction.

7.2 Syringes: 10, 25, 50, 100, 250, 500, and 1000µL.

7.3 Volumetric Flasks, Class A: Appropriate sizes with ground-glass stoppers.

7.4 Vials: Glass autosampler vials with Polytetrafluoroethylene (PTFE)-lined crimp top caps.

8. Reagents and Standards

8.1 Stock Standard Solutions

All stock standard solutions are purchased from commercial vendors. They can be replaced with different standards as long as they contain all target analytes.

8.1.1 Internal Standard Stock Solution

The stock internal standard solution: (Restek, 2000µg/mL in Methylene Chloride, catalog# 31206).

1,4-dichlorobenzene-d_4
naphthalene-d_8
acenaphthene-d_{10}
phenanthrene-d_{10}
chrysene-d_{12}
perylene-d_{12}

8.1.2 Calibration Stock Solutions

Stock Standard	Number	Concentration
Restek Acid Surrogate Mix	31025	2000µg/mL
Restek B/N Surrogate Mix	31024	1000µg/mL
Ultra Scientific o-Terphenyl Solution	IST-480	2000µg/mL
Restek 8270 MegaMix	31850	500-1000µg/mL

8.2 Intermediate/Working Standard Solutions

8.2.1 Internal Standard SIM Solution

The SIM internal standard solution is prepared by diluting the internal standard stock solution with Methylene Chloride. Bring 1mL of the internal standard stock solution up to 10mL final volume with Methylene Chloride in a volumetric flask, resulting in a final concentration of 200µg/mL.

Standards, blanks, QC and sample extracts are spiked in the ratio of 10µL of working SIM internal standard to 500µL of extract, resulting in a concentration of 4µg/mL.

8.2.2 Surrogate Spiking Solution

The surrogate spiking solution is prepared from vendor supplied solutions, as follows:

To a 1L volumetric flask add 5mL each of:

Stock Standard	Catalog Number	Concentration
Restek Acid Surrogate Mix	31087	10000µg/mL
Restek B/N Surrogate Mix	31086	5000µg/mL

Bring to volume with Acetone. The final concentration is 50µg/mL for the acid surrogates and 25µg/mL for the B/N surrogates.

1mL of this surrogate spiking solution is added to each sample, blank and QC samples prior to extraction. See the extraction SOPs for details.

The **LVI surrogate** is a 10 fold dilution of the surrogate solution prepared above. For example, to make 200mL of LVI surrogate, add 20mL of the 50/25µg/mL surrogate solution to a 200mL volumetric flask and fill to volume with Acetone. The resulting surrogate concentration is 5µg/mL for the acid surrogates and 2.5µg/mL for the B/N surrogates

8.2.3 Calibration Standards

Calibration standards are prepared by diluting the stock standard solutions (Section 8.1.2) with Methylene Chloride. Two sources of standards are prepared, one is used for the initial calibration curve, and the other is used for the initial calibration verification standard. These intermediate solutions are then used to prepare the working initial calibration as listed in the table in Section 8.2.4.

8.2.3.1 Target Compound Calibration Standard

Using a calibrated syringe, combine the following to a 50mL volumetric flask:

2mL of 8270 MegaMix

Bring up to volume with Methylene Chloride. The final target compound concentration is 40µg/mL.

8.2.3.2 Surrogate Compound Calibration Standard

Using a calibrated syringe, add the following to a 10mL volumetric flask.

2mL of Acid Surrogate 31025

4mL of B/N Surrogate 31024

2mL of o-Terphenyl Solution IST-480-1

Bring up to volume with Methylene Chloride. The final target compound concentration is 400 µg/mL

8.2.3.2.1 Additional Surrogate Compound Calibration Standard for LVI

Using a calibrated syringe, add 1mL of the standard prepared in 8.2.3.2 to a 10mL volumetric flask and bring to volume with Methylene Chloride. The final target compound concentration is 40ug/mL

8.2.4 Working Calibration Standards

A minimum of a five point calibration is used for each target compound.

The daily continuing calibration check standard is from the same source, at 5 µg/mL for Sim curve and 1.0 µg/mL for LVI Sim curve.

SIM CURVE	
Level	**Concentration (µg/mL) (Target/Surrogate)**
L1	**0.05/0.5**
L2	**0.1/1.0**
L3	**0.2/2.0**
L4	**0.5/5.0**
L5	**1.0/10**
L6	**2.0/20**
L7	**5.0/40**
L8	**10/60**
L9	**15/0**
L10	**20/0**

SIM CURVE for LVI method	
Level	**Concentration (µg/mL) (Target/Surrogate)**
L1	**0.005**
L2	**0.010**
L3	**0.025**
L4	**0.05**
L5	**0.1**
L6	**0.25**
L7	**1.000**
L8	**5.000**
L9	**10.000**
L10	**20.000**

8.2.5 Calibration Standards 8270E NJSIMTECH and PA Compounds

Calibration standards are prepared by diluting the stock standard solutions (8.1.2) with Methylene Chloride. Two sources of standards are prepared, one is used for the initial calibration curve and the other is used for the initial calibration verification standard. These intermediate solutions are then used to prepare the working initial calibration and working initial calibration verification.

8.2.5.1 Target Compound Calibration Standard

Using a calibrated syringe, combine the following to a 25mL volumetric flask:

5mL of 8270 MegaMix
2.5mL of Acid Surrogate 31025
5mL of B/N Surrogate 31024

Bring up to volume with Methylene Chloride. The final target compound concentration is 200µg/mL

8.2.5.2 Working Calibration Standards

A minimum of a five point calibration is used for each target compound. The daily continuing calibration check standard is from the same source, at 5µg/mL.

NJSIMTECH AND PA SIM CURVE	
Level	**Concentration (µg/mL) (Target/Surrogate)**
L1	**0.02/0.2**
L2	**0.05/.5**
L3	**0.1/1.0**
L4	**0.2/2.0**
L5	**0.5/5.0**
L6	**1.0/20**
L7	**2.0/30**
L8	**5.0/60**
L9	**10.0**
L10	**20.0**

8.2.6 LCS/MS/MSD Spiking Standards

SPIKE 1

Restek:

8270 Mega Mix	500-1000µg/ml	cat#31850
Benzoic Acid Mix	2000µg/ml	cat#31879
Benzaldehyde Std	2000µg/ml	cat#33017
Custom AP9 ICAL Std	2000µg/ml	cat#571813-FL

Spike 1: In a 500mL volumetric flask add 20mL of 8270 Mega Mix; 10mL of each Benzoic Acid Mix, Custom AP9 ICAL Std, and Benzaldehyde Std. Bring up to volume with Acetone. The final concentration is 40µg/mL for all compounds except Acetophenone and 3,3'-Dichlorobenzidine which are at 80µg/mL

8.2.6.1 SIM/625.1SIM LCS/MS/MSD Spiking Solution

PAH Low Spk 1:

Take 25mL of the current lot of LCS/MS/MSD Spike 1 add it to a 100mL volumetric flask. Bring to volume with acetone. Final concentration is 10µg/mL for all SIM reportable compounds. If SIM spiking solution is not available, 250uL of the LCS/MS/MSD SPK 1 may be substituted.

The **LVI PAH Low Spk1** is a 10 fold dilution of the above preparation. For example, to make 100mL of LVI Spk 1, add 10mL of the 10µg/mL PAH Low Spk 1 solution to a 100mL volumetric flask and fill to volume with Acetone. The resulting analyte concentration is 1µg/mL

8.2.7 GC/MS Tuning Standard

The stock standard solution contains Decafluorotriphenylphosphine (DFTPP), 4,4'-DDT, Pentachlorophenol, and Benzidine at 1000 µg/mL (Ultra Scientific, Catalog# GCM-150, or equivalent).

The intermediate standard is prepared by taking 5uL of the stock solution and bringing up to 1mL in Methylene Chloride. The resulting concentration is

5µg/mL. The 4,4'-DDT, Pentachlorophenol, and Benzidine are used to verify injection port inertness and GC column performance

8.3 Secondary Source Standards 8270E SIM Compounds

Stock Standard	Catalog Number	Concentration
Restek – 8270 Mega Mix	31850	500-1000 µg/mL
Restek – Acid Surrogate Mix	31025	2000 µg/mL
Restek – B/N Surrogate Mix	31024	1000 µg/mL
Accustandard – o-Terphenyl	DRH-006-SS	1000 µg/mL

8.3.1 Second Source Std

Targets: add 1mL of 31850 into 25mL of Methylene Chloride. Final concentration is 40 µg/mL

Surrogates: add 2mL of 31024, 1mL of 31025 and 2mL of DRH-006-SS to a 10mL volumetric flask. Bring to volume with Methylene Chloride. Final concentration is 200 µg/mL.

8.3.2 ICV Standard

Add 1250µL of the Targets and 2000µL of Surrogate Std Sol (8.3.1) to 10mL of Methylene Chloride. Final concentration of target/surrogates analytes will be 5/40 µg/mL. This standard is used for Initial Calibration Verification.

For the LVI method ICV, add 1250µL of the Targets and 250µL of Surrogate Std Sol (8.3.1) to 10mL of Methylene Chloride. Final concentration of target and surrogates analytes will be 5 µg/mL

8.4 Secondary Source Standards 8270E NJSIMTECH and PA Compounds

Stock Standard	Catalog Number	Concentration
Restek – 8270 Mega Mix	31850	500-1000 µg/mL
Restek – Acid Surrogate Mix	31025	2000 µg/mL
Restek – B/N Surrogate Mix	31024	1000 µg/mL

8.4.1 Second Source Std

Add 2mL of 31850, 2mL of 31024 and 1mL of 31025 to a 10mL volumetric flask. Bring to volume with Methylene Chloride. Final concentration is 200µg/Ml.

8.4.2 ICV Standard

Add 2.5mL SIMTECH ICV Standard Stock to a 50mL volumetric flask. Bring to volume with Methylene Chloride. Final concentration is 10µg/mL

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

Extraction blanks are performed with each extraction batch of 20 or less samples. The extraction blank must not contain any of the reportable analytes above the reporting limit. Corrective actions:

- No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample

- If the blank have reportable hits and re-extraction could not be performed due to lack of additional sample volume, the sample results are reported and qualified with “B” flag for any associated samples that concentration is less than 10x the blank concentration

For NJ regulatory work the method blank must have all the target analytes less than RL except for Phthalates which must be less than 5x of the RL. Sample results are qualified with “B” flag for analytes observed in the blank greater than RL and the Phthalates observed in the blank greater than 5x RL

The surrogate recoveries must also be within the acceptance criteria listed in Table 2. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary

9.2 Laboratory Control Sample (LCS)

A Laboratory Control Sample/ Laboratory Control Sample Duplicate pair (LCS/LCSD) are extracted with each analytical batch and analyzed for each batch of 20 or fewer samples.

The recovery acceptance criteria are listed in Table 5-7. If any recovery criteria are not met, the extract should be reanalyzed. If less than 10% of spike compounds does not meet the criteria this should be noted on a narrative inclusion. If more than 10% of spike compounds is out the entire batch should be re-extracted. If this is not possible, due to insufficient sample or holding time exceedance, the analyst must write up the failure on a narrative sheet for inclusion in the client report. If >10% of analytes are recovered above control limits, this is deemed acceptable as long as the analytes in question are not detected in associated samples

9.3 Initial Calibration Verification (ICV)

See Section 10.2.2

9.4 Continuing Calibration Verification (CCV)

See Section 10.4

9.5 Matrix Spike

A matrix spike/matrix spike duplicate pair is extracted and analyzed for each batch of 20 or fewer samples per client request. The recovery acceptance criteria are listed in Table 5-7. If the recovery criteria are not met, but are met in the LCS, this should be noted on a narrative sheet for inclusion in the client report.

9.6 Laboratory Duplicate

The LCSD is typically used as the laboratory duplicate sample for the batch.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

All extracted samples and associated QC are spiked with surrogates as described in Section 8.2.2. The laboratory must evaluate surrogate recovery data from individual samples and QC samples versus the surrogate control limits listed in Table 2.

Corrective action: Up to one surrogate can be out in each fraction (Acid and Base/Neutral) but not less than 10% recovery, before any corrective action is necessary. Otherwise, analysis must be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

Re-extraction is not required if surrogate recoveries are high and target analytes are not detected in the sample

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is:

- DFTPP
- Continuing Calibration Std
- Method Blank/Instrument Blank
- Samples
- QC (as required)

10. Procedure

10.1 Equipment Set-up

10.1.1 GC/MS Operating Conditions for SIM and 625.1SIM:

Typical GC/MS operating conditions are listed below but may be altered as long as method performance criteria are met.

Mass range:	SIM Mode
Initial temperature:	40°C, hold for 2 minutes
Temperature program 1:	25°C / minute
Final temperature 1:	245°C, no hold.
Temperature program 2:	6°C / minute
Final temperature 2:	288°C, hold for 1 minute
Temperature program 3:	15°C / minute
Final temperature 3:	355°C, no hold
Injector temperature:	300°C
Transfer line temperature:	280°C
Source temperature:	230ºC
Injector:	Split-injection, ratio 5:1
Injection volume:	1µL
Carrier gas:	Helium at 1.1 cm / second, constant flow

GC/MS Operating Conditions for SIM and SIM LVI fast method:

Mass range:	SIM Mode
Initial temperature:	60°C, hold for 0.65 minutes
Temperature program 1:	42.7°C / minute
Final temperature 1:	285°C, no hold.
Temperature program 2:	4.7°C / minute
Final temperature 2:	305°C, no hold
Injector temperature:	270°C
Transfer line temperature:	280°C
Source temperature:	330ºC
Injector:	Split- injection, Ratio 20:1
Injection volume:	2µL, 5µl for LVI
Carrier gas:	Helium at 1.2mL/min, constant flow

10.1.2 GC/MS Tune Check:

At the beginning of every 12 hour sequence, analyze the 5µg/L GC/MS tuning standard (Section 8.2.7).

The resultant mass spectrum for DFTPP must meet the criteria given in Table 1 before standard or sample analysis may begin. The mass spectrum of DFTPP should be acquired in the following manner:

(1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.

(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of DFTPP.

The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Benzidine and pentachlorophenol should be present at their normal responses and no peak tailing should be visible.

The tailing factor for benzidine and pentachlorophenol should be calculated in every DFTPP run. (See Table 8)

10.2 Initial Calibration

10.2.1 Initial Calibration

10.2.1.1 Prepare calibration standards for the target analytes at the ten concentration levels specified in Section 8.2.4 or in 8.2.5.1.

10.2.1.2 Initial Calibration Minimum RRF Criteria:

It is recommended that a minimum response factor for the most common target analytes, as noted in Table 11, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity.

10.2.1.3 Initial Calibration RSD Criteria:

For all analytes, the RSD must be < 20% for the mean response factor to be used for sample quantitation. If the RSD for any target analyte exceeds 20%, the fit must be ≥0.99 quadratic or > 0.995 linear fit for all target analytes. A minimum of 5 levels are required for a linear calibration model and a minimum of 6 levels are required for a quadratic calibration model. When calculating calibration using

the linear regression model, a quantitation check must be performed on the lowest calibration point, or the point that corresponds to that compound's established reporting limit. The recalculated concentration of the low calibration point (especially where the linear regression fits are used) should be within ± 50% of the standard's true concentration, and the recalculated concentration of any calibration standards above the LLOQ should be ± 30%. If this criterion is not meet then the corrective action must take place and a new initial calibration must be performed prior to sample analysis

For RCP, the RSD must be < 30% for the mean response factor to be used for sample quantitation.

Percent Relative Error (%RE): For linear and quadratic fit compounds, the %RE must be calculated for the standard at or near the midpoint and at the lowest level of the initial calibration. The %RE between the calculated and expected concentration of an analyte must be ≤ 30%, otherwise recalibration is necessary.

10.2.1.4 Evaluation of Retention Times:

The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.05 RRT units.

10.2.2 Initial Calibration Verification (Second Source Verification)

10.2.2.1 The initial calibration (Section 10.2) for each compound of interest must be verified prior to sample analysis. This is accomplished by analyzing second source calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.2.2.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

If the % Difference for each analyte is ± 30%, then the calibration is assumed to be valid. If this criterion is not met, then corrective action must be taken prior to the analysis.

10.2.2.3 In cases where compounds fail (greater than 30% difference), they may still be reported as non-detects.

10.2.2.4 Once the calibration curve is accepted and prior to processing any samples, the analyst must demonstrate through the analysis of a MB or instrument blank that equipment and reagents are free from contaminants and interferences. If a peak is found in the blank that would prevent the identification or bias the measurement of an analyte, the analyst should determine the source of the contaminant peak and eliminate it, if possible. Blanks are generally considered to be acceptable if target analyte concentrations are less than one half the LLOQ or are less than project specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected.

10.3 Equipment Operation and Sample Processing

10.3.1 Preparation/Analysis of samples

All extracted samples in K-D tubes need to be brought up to 1mL volume with Methylene Chloride. Liquid extracts are adjusted to get as close to 1:1 as possible as long as the

initial volume is ≥ 800mL; 500uL is transferred to autosampler vial, then spiked with 10µL of Internal Standard and capped/crimped.

10.3.2 GC/MS Analysis of Samples

If the response of any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed.

10.4 Continuing Calibration

10.4.1 The initial calibration (Section 10.2) for each target compound must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing a calibration standard at the midpoint level. An acceptable GC/MS tuning standard must be run prior to analyzing the continuing calibration standard.

10.4.2 If the % Difference for each analyte is ≤ 20%, then the calibration is assumed to be valid. If the criterion are not met for more than 20% of the compounds then corrective action must be taken prior to the analysis of samples.

- Note that for NJ and CT methods, % Difference ≤ 30% is acceptable.

Allowances may be made for an RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples

10.4.3 If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.4.4 If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration must be performed. Due to the large number of analytes present, allowances may be made for a RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples. Any QC failures must be written up by the analyst on narrative sheets for inclusion with the sample data.

10.4.5 Internal Standard Response

If the area for any of the internal standards in the calibration verification standard changes by a factor of two (50% to 200%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.5 Preventive Maintenance

When poor sensitivity is observed, replacement of the injector liner and seal may solve the problem. If not, clip approximately 3 – 6 inches from the injector end of the GC column. If the sensitivity does not improve it may be necessary to replace the split line or the injector weldment assembly. If the problem persists, it may be necessary to replace the GC column.

Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

11. Data Evaluation, Calculations and Reporting

11.1 When a parameter is identified, the quantitation of that parameter should be based on the integrated abundance of the quantitation characteristic m/z given in Table 9.

11.2 Calculate the concentration in the sample using the average response factor (RF) from the initial calibration curve according to the formulae in Alpha's Quality Manual.

11.2.1 Aqueous samples

$$\text{Concentration } (\mu g/L) = \frac{C \times DF \times Vf \times 1000}{Vo}$$

where:

C = Extract concentration (µg/mL)
DF = Dilution factor
Vf = Final extract volume (mL)
Vo = Sample volume (mL)

11.2.2 Soil/sediment samples

$$\text{Concentration } (\mu g/Kg, \text{ dry weight}) = \frac{(C \times DF \times Vf \times 1000)}{W \text{ (gm)}} * \%S$$

where:

C = Extract concentration (µg/mL)
DF = Dilution factor
Vf = Final extract volume (mL)
W = Weight of the sample extracted (10g for high, 30g for low)
%S = Percent solids, as a decimal value

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

See Corrective Actions in Section 9.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

1953 Organic Extraction Glassware Cleaning and Handling

16. Attachments

Table 1: DFTPP Key Ions and Ion Abundance Criteria

Table 2: Acceptable Surrogate Spike Recovery Limits

Table 3: Acceptable Surrogate Spike Recovery Limits for NJ Products

Table 4: Acceptable Surrogate Spike Recovery Limits for PA Products

Table 5: QC Acceptance Criteria: LCS, MS, MSD

Table 6: QC Acceptance Criteria: LCS, MS, MSD for NJSIMTECH Products

Table 7: QC Acceptance Criteria: LCS, MS, MSD for PA Products

Table 8: Tailing Factor Calculation

Table 9: Characteristic Ions for Semivolatile Compounds

Table 10: Reported Detection Limits for Semivolatile Organic Compounds

Table 11: Recommended Minimum Response Factor Criteria

Table 12: Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

TABLE 1

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
68	< 2% of mass 69
69	Present
70	< 2% of mass 69
197	< 2% of mass 198
198	Base peak, 100% relative abundance
199	5-9% of mass 198
365	> 1% of mass 198
441	Present but less than 24% of mass 442
442	> 50% of mass 198
443	15-24% of mass 442

TABLE 2

ACCEPTABLE SURROGATE SPIKE RECOVERY LIMITS

Analytical Fraction	Surrogate Compound	Water	Soil/Sediment
BNA	Nitrobenzene-d_5	23-120%	23-120%
BNA	Phenol-d_6	10-120%	10-120%
BNA	2-Fluorophenol	21-120%	25-120%
BNA	2-Fluorobiphenyl	15-120%	30-120%
BNA	p-Terphenyl-d_{14}	41-149%	18-120%
BNA	2,4,6-Tribromophenol	10-120%	09-136%

TABLE 3

ACCEPTABLE SURROGATE SPIKE RECOVERY LIMITS New Jersey Products

Analytical Fraction	Surrogate Compound	Water	Soil/Sediment
BNA	Nitrobenzene-d_5	30-130%	NA
BNA	Phenol-d_6	15-110%	NA
BNA	2-Fluorophenol	15-110%	NA
BNA	2-Fluorobiphenyl	30-130%	NA
BNA	p-Terphenyl-d_{14}	30-130%	NA
BNA	2,4,6-Tribromophenol	15-110%	NA

TABLE 4

ACCEPTABLE SURROGATE SPIKE RECOVERY LIMITS PA Products

Analytical Fraction	Surrogate Compound	Water	Soil/Sediment
BNA	Nitrobenzene-d_5	23-120%	23-120%
BNA	Phenol-d_6	10-120%	10-120%
BNA	2-Fluorophenol	21-120%	25-120%
BNA	2-Fluorobiphenyl	15-120%	30-120%
BNA	p-Terphenyl-d_{14}	41-149%	18-120%
BNA	2,4,6-Tribromophenol	10-120%	10-136%

TABLE 5
QC ACCEPTANCE CRITERIA:
LCS, MS, MSD

Analytical Fraction	Spike Compound	LCS/MS % Recovery Water	LCS/MS % Recovery Soil/Sediment	LCSD/MS MSD %RPD Water	LCSD/MS MSD %RPD Soil/Sediment
ALL TARGET ANALYTES (EXCEPT BELOW)		40-140%	40-140%	40%	50%
BN	Acenaphthene	37-111%	40-140%	40%	50%
BN	Pyrene	26-127%	35-142%	40%	50%
BN	Hexachlorobutadiene	40-140%	34-107%	40%	50%

BN	**Hexachloroethane**	**40-140%**	**29-106%**	**40%**	**50%**
Acid	**Pentachlorophenol**	**9-103%**	**17-109%**	**40%**	**50%**

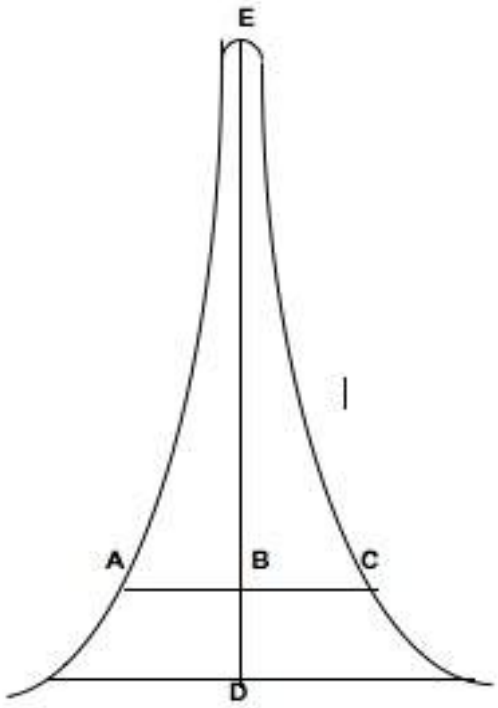
TABLE 6
QC ACCEPTANCE CRITERIA: NJSIMTECH Products
LCS, MS, MSD

Analytical Fraction	Spike Compound	LCS/MS % Recovery Water	MS/MSD %RPD Water
ALL TARGET ANALYTES		**70-130%**	**20%**

TABLE 7
QC ACCEPTANCE CRITERIA: PA Products
LCS, MS, MSD

Analytical Fraction	Spike Compound	LCS/MS % Recovery Water	LCS/MS % Recovery Soil/Sediment	MS/MSD %RPD Water	MS/MSD %RPD Soil/Sediment
ALL TARGET ANALYTES (EXCEPT BELOW)		**40-140%**	**40-140%**	**40%**	**50%**
BN	**Acenaphthene**	**37-111%**	**31-137%**	**40%**	**50%**
BN	**Pyrene**	**26-127%**	**35-142%**	**40%**	**50%**
Acid	**Pentachlorophenol**	**9-103%**	**17-109%**	**40%**	**50%**

TABLE 8



Tailing Factor = $\frac{BC}{AB}$

$\overline{AB}$

Example calculation:

Peak Height = DE = 100mm
10% Peak Height = BD = 10mm
Peak Width at 10% Peak Height = AC = 23mm

AB = 11mm
BC = 12mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Tailing factor for benzidine < 2.0

Tailing factor for pentachlorophenol < 2.0

TABLE 9

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

The ions and intervals are determined by an experienced analyst and are subject to changes.

Compound	Primary Ion	Secondary Ion
1,4-Dichlorobenzene-d_4 (IS)	152	150
Naphthalene-d_8 (IS)	136	68
Naphthalene	128	129
Hexachlorobutadiene	225	223
2-Methylnaphthalene	142	141
Hexachloroethene	117	201
1-Methylnaphthalene	142	141
2-Chloronaphthalene	162	127
Acenaphthylene	152	151
Acenaphthene-d_{10} (IS)	164	162
Acenaphthene	153	154
Fluorene	166	165
Hexachlorobenzene	284	142
Pentachlorophenol	266	264
Phenanthrene-d_{10} (IS)	188	80
Phenanthrene	178	179
Anthracene	178	179
Fluoranthene	202	101
Pyrene	202	200
Benz(a)anthracene	228	229
Chrysene-d_{12} (IS)	240	236
Chrysene	228	226
Benzo(b)fluoranthene	252	253
Benzo(k)fluoranthene	252	253
Benzo(a)pyrene	252	253
Perylene-d_{12} (IS)	264	260
Indeno(1,2,3-cd)pyrene	276	138
Dibenzo(a,h)anthracene	278	139
Benzo(g,h,i)perylene	276	138
Bis(2-chloroethyl)ether	93	63
n-Nitrosodi-n-propylamine	70	130
4,6-Dinitro-o-cresol	198	105
Bis(2-ethylhexyl)phthalate	149	167
2-Fluorobiphenyl (Surrogate)	172	171
Nitrobenzene-d_5 (Surrogate)	82	54
Terphenyl-d_{14} (Surrogate)	244	122
2-Fluorophenol (surrogate)	112	64
Phenol-d_6 (surrogate)	99	71

2,4,6 Tribromophenol (surrogate)	**330**	**62**
o-Terphenyl-MS (surrogate)	**230**	**229**

TABLE 10

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS

Analyte	RDL (µg/L)	RDL (µg/Kg)
Acenaphthene	**0.1**	**6.67**
Acenaphthylene	**0.1**	**6.67**
Anthracene	**0.1**	**6.67**
Benzo(a)anthracene	**0.05**	**6.67**
Benzo(b)fluoranthene	**0.05**	**6.67**
Benzo(k)fluoranthene	**0.1**	**6.67**
Benzo(ghi)perylene	**0.1**	**6.67**
Benzo(a)pyrene	**0.1**	**6.67**
2-Chloronaphthalene	**0.2**	**6.67**
Chrysene	**0.1**	**6.67**
Dibenzo(a,h)anthracene	**0.1**	**6.67**
Fluoranthene	**0.1**	**6.67**
Fluorene	**0.1**	**6.67**
Indeno(1,2,3-cd)pyrene	**0.1**	**6.67**
1-Methylnaphthalene	**0.1**	**6.67**
2-Methylnaphthalene	**0.1**	**6.67**
Naphthalene	**0.1**	**6.67**
Phenanthrene	**0.05**	**6.67**
Pyrene	**0.1**	**6.67**

Additional non-PAH Analytes Available	RDL (µg/L)	RDL (µg/Kg)
Hexachlorobenzene	**0.02**	**6.67**
Hexachlorobutadiene	**0.2**	**6.67**
Hexachloroethane	**0.2**	**6.67**
Pentachlorophenol	**0.3**	**26**
Hexachloroethane	**0.8**	**6.67**
Bis(2-ethylhexyl)phthalate	**1.0**	**NA**
4,6-Dinitro-o-cresol	**1.0**	**NA**
Bis (2-chloroethyl)ether	**0.1**	**33.3**
n-Nitrosodi-n-propylamine	**0.1**	**33.3**

Table 11

Recommended Minimum Response Factor Criteria from Initial and Continuing Calibration Verification Using the Suggested Ions in Table 9

Analyte	MRF
Bis(2-chloroethyl)ether	0.700
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Isophorone	0.400
Naphthalene	0.700
Hexachlorobutadiene	0.010
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2-Chloronaphthalene	0.800
Acenaphthylene	0.900
Acenaphthene	0.900
Fluorene	0.900
4,6-Dinitro-o-cresol	0.010
Hexachlorobenzene	0.100
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Fluoranthene	0.600
Pyrene	0.600
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500

Table 12

Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10
2-Fluorophenol, surr	1-Methylnaphthalene	2,4,6-Tribromophenol, surr
Bis(2-chloroethyl)ether	2 -Chloronaphthalene	Acenaphthene
Hexachloroethane	2-Fluorobiphenyl, surr	Fluorene
Nitrobenzene-d5, surr	2-Methylnaphthalene	
N-nitrosodi-n-propylamine	Acenaphthylene	
Phenol-d6, surr	Hexachlorobutadiene	
	Isophorone	
	Naphthalene	
Phenanthrene-d10	**Chrysene-d12**	**Perylene-d12**
4,6-Dinitro-o-cresol	Benzo(a)anthracene	Benzo(a)pyrene
Anthracene	Bis(2-ethylhexyl) phthalate	Benzo(b)fluoranthene
Fluoranthene	Chrysene	Benzo(g,h,i)perylene
Hexachlorobenzene		Benzo(k)fluoranthene
O-Terphenyl-MS		Dibenzo(a,h)anthracene
Pentachlorophenol		Indeno(1,2,3-cd)pyrene
Phenanthrene		
Pyrene		
Terphenyl-d14, surr		

Reactive Sulfide and Reactive Cyanide

Reference Method: This standard operating procedure (SOP) is a performance-based method. This SOP describes the procedure as developed by Alpha Analytical.

1. Scope and Application

Matrices: This method is applicable to water, wastewater, seawater, soil and solid samples with the condition that the samples combined with acids do not form explosive mixtures.

Definitions: See Alpha Analytical Quality Manual

The regulation in 40CFR 261.23 defines reactive wastes to include wastes that have any of the following properties:

(1) readily undergo violent chemical change;

(2) react violently or form potentially explosive mixtures with water;

(3) generate toxic fumes when mixed with water or, in the case of cyanide- or sulfide-bearing wastes, when exposed to mild acidic or basic conditions;

(4) explode when subjected to a strong initiating force;

(5) explode at normal temperatures and pressures; or

(6) fit within the Department of Transportation's forbidden explosives, Class A explosives, or Class B explosives classifications.

This definition is intended to identify wastes that, because of their extreme instability and tendency to react violently or explode, pose a problem at all stages of the waste management process. The definition is to a large extent a paraphrase of the narrative definition employed by the National Fire Protection Association. The Agency chose to rely almost entirely on a descriptive, prose definition of reactivity because most of the available tests for measuring the variegated class of effects embraced by the reactivity definition suffer from a number of deficiencies.

This procedure releases only the hydrogen sulfide evolved at the test conditions. It is not intended to measure forms of sulfide other than those that are evolvable under the test conditions.

This test measures only the hydrocyanic acid evolved at the test conditions. It is not intended to measure forms of cyanide other than those that are evolvable under the test conditions.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

An aliquot of acid is added to a fixed volume or weight of sample in a closed system. The generated gas is swept into a scrubber. The analyte is quantitated.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

The reported reactive sulfide detection limit is 1.0 mg/L (aqueous samples) and 10mg/kg (solid samples).

The reported reactive cyanide detection limit is 1.0 mg/L (aqueous samples) and 10mg/kg (solid samples).

4. Interferences

4.1 Reactive Sulfide Interferences

4.1.1 Strong reducing agents interfere in the methylene blue method by preventing formation of the blue color.

4.1.2 Thiosulfate at concentrations about 10mg/L may retard color formation or completely prevent it.

4.1.3 High Concentration of sulfide itself may inhibit blue color development. To avoid the possibility of reporting false negative results, test a 1000x dilution of the scrubber solution to see if blue color develops. (Refer to Section 10.3.1).

4.1.4 Ferrocyanide is a positive interference since it produces a blue color; distillation process will minimize interference.

4.2 Reactive Cyanide Interferences

Reactive Cyanide interferences are undetermined.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. Therefore, analysts should take every precaution by utilizing latex/nitrile gloves, safety glasses and a lab coat to minimize or eliminate exposure. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected with minimum aeration in either a glass or plastic bottle or jar. Fill the container completely and cover, ensuring no head space is present.

6.2 Sample Preservation

Samples are stored at 4 ± 2° C.

6.3 Sample Shipping
No special shipping requirements.

6.4 Sample Handling
Aqueous samples are analyzed with 7 days of collection. Soil samples are analyzed within 14 days of collection.

7. Equipment and Supplies

7.1 Spectrophotometer: Genesys 10vis (or equivalent); for use at a wavelength of 664nm and 578nm with cells providing light paths of 1cm.

7.2 Centrifuge Tubes: 50mL capacity

7.3 Pipets: Class A, glass, volumetric, various volumes

7.4 Volumetric Flasks: Class A, various volumes

7.5 Non-rubber Flexible Tubing: For connection from pump supply to apparatus.

7.6 Analytical Balance: Capable of weighing to 0.001g.

7.7 Midi Distillation System: Lab-Crest (or equivalent); 10-position distillation unit equipped with a full set of glassware including 10 each of the following:

7.7.1 Reaction Flask: Flat-bottom flask with sufficient volume (over 100mL) to contain violent reactions; has permanent 50mL indicator.

7.7.2 Absorption Flask: Identical to Reaction Flask (Section 7.7.1).

7.7.3 Reflux Impinger: Fits the reaction flask; has air and reagent intake, full-length impinger, and hose barb.

7.7.4 Cold Finger: Fits the Reflux Impinger; has two hose barbs for water circulation, which is not utilized for this test. It can therefore be substituted with a glass or plastic stopper of the same size.

7.7.5 Absorption Impinger: Full-length impinger with 13mm, medium porosity glass filter, and two hose barbs.

8. Reagents and Standards

8.1 Reagent water: Deionized water (DI) produced by Alpha's water treatment system.

8.2 Sulfide Calibration Stock Standard: Weigh 1.5779 gram of $Na_2S \cdot 9H_2O$ in a 200mL volumetric flask and fill to top with DI water. Mix by swirling gently to prevent oxygen entrapment. Store in the refrigerator in the amber glass bottle. Solution expires one month from the date of preparation. Final concentration is 1097 mg/L

8.3 Sulfide 1 Reagent: Available commercially from HACH Company. Store at 4 ± 2°C. Reagent expires on manufacturer's specified expiration date.

8.4 Sulfide 2 Reagent: Available commercially from HACH Company. Store at 4 ± 2°C. Reagent expires on manufacturer's specified expiration date.

8.5 Sulfide Calibration Standards: These standards are used to generate the calibration curve in Section 9.1.1. Prepare a series of sulfide solutions by diluting the Sulfide Calibration Stock Standard (Section 8.2) with 0.1N NaOH (Section 8.6.2).

Into seven 100mL volumetric flasks, pipet 1mL, 2mL, 5mL, 10mL, 15mL, 30mL and 0mL of the sulfide stock standard (Section 8.2) Bring each flask to volume using 0.1N NaOH solution (Section 8.6.2).

The concentration of the calibration standards is determined by the results of the standardization of the stock sulfide standard, as described in Section 8.2.1. The lowest calibration standard must be 0.1mg/L or less. If necessary, add an additional point to the curve by pipetting 0.5mL of Stock Sulfide Standard (Section 8.2) into an eighth 100mL volumetric flask.

Note: This calibration curve is identical to calibration curve, used for Total Sulfide.

8.6 Sodium Hydroxide, NaOH: In pellet form.

8.6.1 Sodium Hydroxide Solution, NaOH, 1N: Dissolve 40g NaOH (Section 8.11) in 1L of DI water. Store at room temperature. Expires three months from the date of preparation.

8.6.2 Sodium Hydroxide Solution, NaOH, 0.1N: In a 100mL volumetric flask, pipet 10mL of 1N NaOH (Section 8.11.1). Bring to volume with DI water. Store at room temperature. Expires three months from the date of preparation.

8.7 Sulfide LCS Stock Standard: Using a different source than was used for the Calibration Stock Standard. (8.2)

8.8 Sulfide LCS Standard: Pipet 0.5mL of LCS Sulfide Stock Standard (Section 8.7) into a 25mL volumetric flask. Bring to volume with DI water. True value: 22 mg/l.

8.8.1 Sulfide ICV / CCV Standard: Pipet 1.0 mL of LCS Sulfide Standard (Section 8.7) into a 50mL volumetric flask, add 5 ml of 1N NAOH. Bring to volume with DI water. Acceptance criteria +/- 25%; if failed outside of acceptance criteria, data is invalid.

8.9 Cyanide LCS/ICV/CCV Stock Standard, 1000ppm: Commercially available with a certificate of analysis. This must be from a different source than the Calibration Standard Stock. Store at 4 ± 2 °C. Expires on manufacturer's specified expiration date.

8.9.1 Cyanide LCS/ICV/CCV Working Standard, 40ppm: To a 25mL volumetric flask, add 1mL of 1000ppm cyanide LCS Stock Standard (Section 8.9). Bring to volume with DI water. Prepare fresh on each day of use.

8.9.1.1 Cyanide ICV/CCV 0.8ppm Standard: Into a 25mL volumetric flask, pipet 0.5mL of the 40ppm cyanide working calibration standard (Section 8.9.1). Bring to volume using 0.1N NaOH (Section 8.6.2) (5 ml of 1N NaOH can be used and volume brought up to 50 ml with DI. This standard is prepared fresh at the time of use.

8.10 Cyanide 1000ppm Stock Calibration Standard Stock Standard: Commercially purchased with a certificate of analysis. The ICV stock must be a different lot than the calibration standards.

8.10.1 Cyanide 10ppm Working Calibration Standard: Into a 100mL volumetric flask, pipet 1mL of 1000ppm cyanide stock calibration standard (Section 8.10) and bring to volume with 0.1N NaOH (Section 8.6.2). This standard expires after one week. Store in a brown bottle at room temperature, out of direct light.

8.10.1.1 0.0ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 0.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.2 0.05ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 0.5mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.3 0.1ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 1.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.4 0.2ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 2.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.5 0.4ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 4.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.10.1.6 0.5ppm Cyanide Calibration Standard: To a 100mL volumetric flask, add 5.0mL of Cyanide 10ppm Working Calibration Standard (Section 8.10.1). Bring to volume with 0.1N NaOH (Section 8.6.2). This standard is prepared fresh at the time of calibration.

8.11 Chloramine-T Solution: Dissolve 1.0g of Chloramine-T in 100mL DI water. Prepare fresh on each day of use.

8.12 Sodium dihydrogen phosphate monohydrate, $NaH_2PO_4 \cdot H_2O$

8.12.1 Phosphate Buffer: Dissolve 138g sodium dihydrogen phosphate monohydrate (Section 8.12) in DI water and dilute to 1L. Store at room temperature. Expires one month from the date of preparation.

8.13 Barbituric acid: Store at room temperature. No expiration date.

8.14 Pyridine: Store in Flammables cabinet. Expires on manufacturer's specified expiration date.

8.15 Concentrated Hydrochloric Acid, HCl: Store in Acids cabinet. No expiration date.

8.16 6M Hydrochloric Acid (HCl): Dilute 500mL of concentrated HCl (Section 8.20) into 500mL DI water. Store at room temperature. No expiration date.

8.17 Pyridine-Barbituric Acid Reagent: Place 60g barbituric acid (Section 8.13) in a 1000mL volumetric flask and add just enough DI water to wash sides of flask and wet barbituric acid. Add 300mL pyridine (Section 8.14) and mix. Add 60mL conc HCl (Section 8.15), mix and cool to room temperature. Dilute to volume and mix until barbituric acid is dissolved. The solution is stable for one month if stored in an amber bottle at 4 ± 2 °C. Discard if precipitate develops.

8.18 Concentrated Sulfuric Acid, H_2SO_4: Store in Acids cabinet. Expires on manufacturer's specified expiration date.

8.19 Reactivity Stock Solution: Add 2.8mL of conc H_2SO_4 (Section 8.18) to a 1L volumetric flask and bring to volume with DI. Prepare fresh on each day of use.

8.19.1 Reactivity Working Solution, 0.025M: Add 100mL of reactivity stock solution (Section 8.19) to a 1L volumetric flask and bring to volume DI.

8.20 Ottawa sand

8.21 Boiling chips

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Prepare and analyze one Method Blank per batch of 20 samples or less. Blank results must be less than the RL. If this criterion is not met, a batch narrative is included with the Client Report and/or the sample batch is reanalyzed. Refer to Section 12 for corrective actions.

For soils: weigh 1.0 g of Ottawa sand, for water- use 10 ml if DI and analyze with batch.

9.2 Laboratory Control Sample (LCS)

9.2.1 Reactive Sulfide

Run one LCS using the LCS Working Solution per batch of 20 samples or less. .

For waters: to 10 ml of DI add 2.0 ml of LCS Standard (8.8); final concentration is 4.4 mg/l

For soils: to 1.0 g of Ottawa sand add 2.0 ml of LCS Standard (8.8); final concentration 44 mg/kg

The LCS must be recovered within 60 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.2.2 Reactive Cyanide

Run one LCS using the LCS Working Solution per batch of 20 samples or less.

For waters: to 10 ml of DI add 1.0 ml of LCS Standard (8.9.1); final concentration is 4.0 mg/l

For soils: to 1.0 g of Ottawa sand add 1.0 ml of LCS Standard (8.9.1); final concentration 40 mg/kg

The LCS must be recovered within 30 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.3 Initial Calibration Verification (ICV)

9.3.1 Reactive Sulfide

The ICV is analyzed at the beginning of each sample batch. The ICV must be recovered within 75 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.3.2 Reactive Cyanide

An ICV is analyzed at the beginning of each sample batch. The ICV must be recovered within 75 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.4 Continuing Calibration Verification (CCV)

9.4.1 Reactive Sulfide

A CCV is read on the spectrophotometer after every 10 samples and at the end of each sample batch. The CCV must be recovered within 75 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.4.2 Reactive Cyanide

A CCV is read on the spectrophotometer after every 10 samples and at the end of each sample batch. The CCV must be recovered within 75 – 125%, otherwise the entire sample batch is reanalyzed. Refer to Section 12 for corrective actions.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

Prepare and analyze one sample in duplicate per batch of 20 samples or less. The %RPD for liquid samples must be $\leq$ 25%. For solid/soil samples, the %RPD must be $\leq$ 40%. If %RPD is not within the specified range, the sample and its duplicate must be narrated and/or reanalyzed. Refer to Section 12 for corrective actions.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Generate calibration curves.
- Prepare samples and QC samples.
- Analyze samples and QC samples.
- Calculate results.

10. Procedure

10.1 Equipment Set-up

10.1.1 Calibration Curve Generation

Calibration curves are generated yearly or when ICV/CCV are not passing acceptance criteria.

10.1.1.1 Reactive Sulfide

Prepare a calibration curve using the standards in Section 8.5. Analyze the standards as described in Section 10.3.1. Plot the concentration vs. absorbance. Determine the slope and the y-intercept. The correlation coefficient must be $\geq$ 0.995.

10.1.1.2 Reactive Cyanide

Prepare a calibration curve using the standards in Section 8.10. Analyze the standards as described in Section 10.3.2. Plot the concentration vs. absorbance. Determine the slope and the y-intercept. The correlation coefficient must be $\geq$ 0.995.

10.1.2 Sample Preparation

10.1.2.1 Add 10mL of 1N NaOH solution to the absorption flask, and bring to a volume of 50mL with DI.

10.1.2.2 Weigh 1g of solid sample or 10mL of liquid sample into a 50mL centrifuge tube and record the weight/volume in the laboratory notebook. Label the tube and secure the cap. For solid Blank and LCS, use PTFE boiling stones or Ottawa sand. Add the contents of the tube to the reaction vessel. Record the tube number and corresponding sample number in the laboratory notebook. Assemble the Midi Distillation Unit.

10.1.2.2.1 Ensure the pump is plugged in and connected to the Midi unit via a Teflon hose. After adding the sample to the reaction vessel (large tube in the back of the unit), place the absorption flask in the front of the unit ; flask should be filled with NaOH. Place the reflux impinger in the reaction flask and place the cold finger (or stopper) into the impinger. Make sure that all of the glass connections are tight. Place the absorption impinger into the absorption flask and make sure that the glass seal is tight. Connect the absorption impinger to the reaction impinger by connecting the front hose of the absorption impinger to the Midi Distillation unit (where the air comes out) and the rear hose to the reaction impinger. Make sure that all glass connections are tight and that all of the hoses are snug.

10.1.2.3 Turn on the pump and adjust the air flow rate of air so that the gas scrubbers produce small bubbles (approx. 60mL/minute).

10.1.2.4 Add the appropriate LCS to their respective reaction vessels. Then add the reactivity solution (Section 8.19.1) to each reaction vessel so that the final volume is about 50mL.

10.1.2.5 Make sure all of the glassware is connected and all of the tubes are connected. All of the reflux impingers should now be producing air bubbles in the reaction (rear) vessels. For any are not, the glassware is not air tight. Run the pump for 30 minutes, and then shut the pump off. Record in the laboratory notebook the time started and the time ended.

10.1.2.6 Disconect the apparatus and bring the volume of the absorption tube to 100mL in DI with a volumetric flask. The normality of this hydroxide solution will be 0.1N.

10.1.2.7 Pour off 25mL into a 50mL centrifuge tube for Sulfide analysis. Pour off 20mL into a 50mL centrifuge tube for Cyanide analysis.

10.2 Initial Calibration

Refer to Sections 10.1.1 and 9.3.

10.3 Equipment Operation and Sample Processing

10.3.1 Reactive Sulfide Analysis

Take a 25mL aliquot of the calibration standards (Section 8.5) or of the volumized scrubber solution for each sample and color as follows:

Add 1.0mL Sulfide 1 reagent (Section 8.3) and swirl to mix. Add 1mL of Sulfide 2 reagent (Section 8.4). Let the reaction continue for 5 minutes. Excessive time between the addition of the “1” and “2” Sulfide coloring reagents causes low results by loss of H_2S as a gas before it has had time to react. The presence of sulfide will be indicated by the appearance of blue color. Wait 5 minutes and read the absorbance on the spectrophotometer (below).

Absorbance Determination: Set the spectrophotometer to a wavelength of 664nm. Zero the instrument with a portion of the blank sample. Read the absorbances of the samples and QC samples and record this information in the laboratory notebook.

If the absorbance of a sample is greater than the highest standard in the calibration curve, the sample must be diluted with 0.1N NaOH and reanalyzed as in Section 10.3.1 above. Record in the laboratory notebook any dilutions that are prepared.

NOTE: Very high sulfide concentrations interfere with blue color formation. For a sample in which high sulfide is suspected, but no blue color develops, make a 1000x dilution of the scrubber solution with 0.1N Color with Reagent 1 (Section 8.3) and Reagent 2 (Section 8.4). Experiment with various dilutions until the absorbance falls within the calibration curve.

10.3.2 Reactive Cyanide Analysis

Take a 20mL aliquot of the calibration standards (Section 8.10.1) or of the volumized scrubber solution and color as follows:

Add 5mL of phosphate buffer solution (Section 8.12.1), 2mL of chloramine-T solution (Section 8.11), 5mL of pyridine (Section 8.14). Bring final volume to 50mL with DI. The presence of a pink color indicates cyanide.

Absorbance Determination: Set the spectrophotometer to a wavelength of 578nm. Zero the instrument with a portion of the blank sample. Read the absorbances of the samples and QC samples and record this information in the laboratory notebook.

If the absorbance is greater than the highest standard in the calibration curve, the sample must be diluted with 0.1N NaOH and reanalyzed as in Section 10.3.2 above. Record in the laboratory notebook any dilutions that are prepared.

10.4 Continuing Calibration

Refer to Section 9.4.

10.5 Preventive Maintenance

The spectrophotometers are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file.

11. Data Evaluation, Calculations and Reporting

11.1 Reactive Sulfide

Calculate reactive sulfide concentration using the calibration curve generated in Section 10.1.1.1:

$$\text{Reactive Sulfide (mg/L)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \text{Dilution factor}$$

$$\text{Reactive Sulfide (mg/kg)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \frac{\text{Dilution factor}}{\text{Weight (g)}}$$

11.2 Reactive Cyanide

Calculate reactive cyanide concentration using the calibration curve generated in Section 10.1.1.2:

$$\text{Reactive CN (mg/L)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \text{Dilution factor}$$

$$\text{Reactive CN (mg/kg)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \frac{\text{Dilution factor}}{\text{Weight (g)}}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence, improper preservation and sample headspace (liquid samples) are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the method blank contains a reportable amount of cyanide of Sulfide, evaluate the method blank as follows. If the samples associated with the method blank are non-detect, or if a sample has an analyte concentration that is greater than 10X the concentration of the contaminated method blank, the data may be reported and the method blank results narrated on the final report. If sample results are positive, but at a concentration less than 10X the concentration of the method blank, then those samples should be re-digested and re-analyzed.

If the LCS fails acceptance criteria, then the entire batch must be re-digested and re-analyzed. If there is insufficient sample volume to re-digest, then the Client Services Department is notified, and the batch results are narrated on the final report as estimated.

If the ICV fails acceptance criteria, a fresh aliquot is poured and re-analyzed. If the standard still fails, prepare a fresh ICV and begin analysis again. If the ICV fails again, and the spectrophotometer is working properly, prepare a new calibration curve.

If the ICB fails acceptance criteria, a fresh aliquot is poured and re-analyzed. If the blank still fails, ensure that the spectrophotometer is “blanked” correctly. If the ICB failure continues, and the spectrophotometer working properly, prepare a new calibration curve.

If the CCV fails acceptance criteria, a fresh aliquot is poured and re-analyzed. If the standard still fails, prepare and analyze a fresh CCV and re-analyze the last 10 samples up to the last passing CCV. If the CCV recovery is greater than 125%, and all associated samples are non-detect, then analysis may be continued. If the CCV recovery is greater than 125%, any associated positive samples are re-prepared and re-analyzed.

If the CCB fails acceptance criteria, a fresh aliquot is poured and re-analyzed. If the CCB still fails, ensure that the spectrophotometer is working properly. Also ensure that the cell is clean and has no smudges, and is indexed correctly. When the problem has been corrected, all samples analyzed since the last passing CCB must be re-analyzed.

If %RPD of duplicates fails acceptance criteria, the non-conformance is narrated and the information is included on the final report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1734, 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1734, 1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

Semivolatile Organic Compounds By Gas Chromatography / Mass Spectrometry (GC/MS)

References: **EPA 8270E**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision VI (Phase II), June 2018.

EPA 8000D, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision III, March 2003.

Massachusetts DEP Bureau of Waste Site Cleanup - Quality Control Requirements and Performance Standards for the Analysis of Semivolatile Organic Compounds by GC/MS in Support of Response Actions under the Massachusetts Contingency Plan (MCP), Revision 1, July 2010.

NJDEP Site Remediation Program, Data of Known Quality Protocol, Version 1, April 2014.

Connecticut Department of Environmental Protection. Recommended Reasonable Confidence Protocols. Version 2.0 July 2006

1. Scope and Application

Matrices: Solid, waste and waste water, soil, sediment, tissue and ground water.

Definitions: Refer to Alpha Analytical Quality Manual.

This method is applicable to the quantification of acidic, neutral and basic organic compounds soluble in methylene chloride. The extraction and cleanup methods that are typically used for this method are listed in Section 10.3.1. Please see the appropriate extraction SOP(s) for further information. The individual target compounds are found in Section 16, Table D, and include polynuclear aromatic hydrocarbons, chlorinated hydrocarbons, pesticides, phthalate esters, nitrosamines, aldehydes, ethers, anilines, pyridines, aromatic nitro compounds and phenols, including nitro phenols.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/MS and in the interpretation of GC/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Aqueous samples are extracted with methylene chloride in a 2 Liter Separatory Funnel (Method 3510) at the required pH. Soil samples are generally extracted by Microscale Solvent Extraction (Method 3570), however other extraction methods can be applied. The extract is concentrated to its final volume in preparation for analysis, or may now undergo cleanups.

Analytes are introduced into the GC/MS by injecting a known volume of the calibration standards, quality control samples, and sample extracts into the GC equipped with a narrow-bore capillary column. The GC column is temperature programmed to separate the analytes, which are then detected with a mass spectrometer (MS). Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of the calibration standards. Concentrations are determined using mean relative response factors from a multi-level calibration curve. Response factors for target analytes and surrogate compounds are determined relative to the internal standards.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Analytes determined are measured in the range of 0.5 to 25ug/L for water samples, and 33.3 to 1667ug/Kg for soils, sediments, and solids. Tissue samples are measured in the range of 200 to 10000ug/Kg. Detection limits will vary with the individual sample matrix, sample preparation procedures, instrument calibration range, and volume of sample analyzed. Analytes detected over these concentration ranges will be diluted and re-analyzed for accurate quantification. Reporting limits for each matrix per analyte can be viewed within the LIMS under Applications/Product Catalog

4. Interferences

4.1 Phthalate esters can be a major source of contamination if any material containing plasticizers (phthalates) comes in contact with the sample during the extraction process. Use of plastic or any material containing plasticizers (phthalates) should be avoided during extraction or analysis.

4.2 The injection port of the gas chromatograph can become contaminated with high boiling compounds resulting in the loss of sensitivity of Pentachlorophenol, 2,4-Dinitrophenol, 4-Nitrophenol, Benzoic Acid, Benzaldehyde, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 4-chloroaniline, benzyl alcohol, 2,4-Dinitrotoluene and 2, 3, and 4-Nitroaniline. It may be necessary to replace the injection port liner routinely to prevent this loss of sensitivity. Clipping off approximately four inches of the column at the injection end may also increase sensitivity of the phenolic compounds. Low instrument response can be detected during the daily tuning procedure by including pentachlorophenol and benzidine in the daily tuning mix.

4.3 Raw GC/MS data from all blanks, samples, and spikes must be evaluated for interferences or carryover. Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed.

4.4 Solvents, reagents and glassware may introduce interferences. These must be demonstrated to be free of interferences by the analysis of a method blank. See the *Reagent, Solvent and Standard Control* SOP (1816) and *Laboratory Glassware Cleaning SOP* (1753), for additional details.

4.5 Many interferences can be removed by sample cleanup. The cleanup methods performed include those listed in Section 10.3.1. Only appropriate cleanup techniques must be performed based on the suspected interference and the compounds of interest.

4.6 Benzidine and pyridine may be subject to oxidative losses during solvent concentration and demonstrate poor chromatographic behavior.

4.7 Hexachlorocyclopentadiene and pyridine are subject to thermal decomposition in the injection inlet, and hexachlorocyclopentadiene is subject to chemical reaction with acetone,

and photochemical decomposition.

4.8 N-nitrosodimethylamine is difficult to separate from the solvent front under the chromatographic conditions described within.

N-nitrosodiphenylamine decomposes in the GC inlet and cannot be separated from diphenylamine.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Solid samples: A minimum of 100 grams of sample must be collected in a glass jar with a Teflon lined screw cap.

Water samples: A minimum of 2 liters of sample must be collected in amber glass bottles.

Tissue samples: A minimum of 20 grams of sample must be collected in a glass jar with a Teflon lined screw cap.

6.2 Sample Preservation

Solid samples: The samples must be refrigerated and maintained at 4±2°C until extraction and analysis. Sediment samples can be frozen at -20±5°C until extraction to extend hold time. The extracts must be refrigerated and maintained at 4±2°C until analysis.

Water samples: The samples must not be preserved except by refrigeration at 4±2°C until extraction and analysis. The extracts must be refrigerated and maintained at 4±2°C until analysis.

Tissue samples: The samples must be stored frozen at -20±5°C until extraction. The extracts must be refrigerated and maintained at 4±2°C until analysis.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Solid samples: All solid samples must be extracted within 14 days from the date of collection. Frozen sample hold times are monitored up to 14 days from the date removed from the freezer. The extracts must be refrigerated and maintained at 4±2°C until analysis. Sample extracts must be analyzed within 40 days from date of extraction.

Water samples: All water samples must be extracted within 7 days from the date of collection. Sample extracts must be analyzed within 40 days from date of extraction.

Tissue samples: Frozen sample hold times are monitored up to 14 days from the date removed from the freezer. The extracts must be refrigerated and maintained at 4±2°C until analysis. Sample extracts must be analyzed within 40 days from date of extraction.

7. Equipment and Supplies

7.1 Gas chromatograph: Programmable, heating range from 40C to 350C; split-splitless-type inlet system, (Agilent 7890 or similar); mass selective detector (Agilent 5975 or similar); automatic injector (Agilent 7683 or similar).

7.2 Chromatography Column: Fused silica capillary column, 0.25mm ID x 30m length, .25um film thickness (Zebron ZB-SemiVolatiles, Phenomenex Corporation, 5% Polysilarylene - 95% Polydimethylsiloxane , or equivalent).

7.3 Data Acquisition System: Computerized system for collecting, storing, and processing detector output (Hewlett Packard Enviroquant target software) or equivalent.

7.4 Gases: Ultra high purity BIP helium (99.9995%); Compressed Nitrogen for N-Evap.

7.5 Syringes: 10uL to 1.0mL.

7.6 Glass: Class A volumetric glass with varying volumes

7.7 GC Injection Port Liner: Phenomenex Direct Connect Top Hole

8. Reagents and Standards

Use reagent grade chemicals for all reagents. Deionized (DI) water is ASTM Type II laboratory reagent grade water.

8.1 Methylene Chloride: ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (1816) for additional details regarding solvent purity.

8.2 Acetone: ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (1816) for additional details regarding solvent purity.

8.3 Hexane: ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (1816) for additional details regarding solvent purity.

8.4 Methanol: ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (1816) for additional details regarding solvent purity.

8.5 Ether: ACS approved, Pesticide grade, see *Reagent, Solvent and Standard Control SOP* (1816) for additional details regarding solvent purity.

8.6 Analytical Standards: Standards should be stored at –10°C or less, away from light when not in use. They should be discarded after 1 year unless the vendor expiration date states otherwise or if breakdown is observed. Stock standards are given a 1 year expiration from the preparation date, or the expiration of the primary vendor solution, whichever occurs

first. Working standards are given a 6 months expiration from the preparation date or the expiration of the primary solution whichever occurs first.

8.6.1 Surrogates: Base/Neutral stock surrogate (5000ug/mL) – includes Nitrobenzene-d5, 2-Fluorobiphenyl, and Terphenyl-d14. Acid stock surrogate (10000 ug/mL) - includes Phenol-d5, 2-Fluorophenol, and 2,4,6-Tribromophenol. Restek, Ultra Scientific, or equivalent.

8.6.1.1 To prepare the surrogate spiking solution mix for 8270: Add 4.0mL of the base neutral mix (5000 ug/mL) and 2.0mL of the acid surrogate mix (10000 ug/mL) to a 250mL volumetric flask. Dilute to volume with Acetone to provide an 80 ug/mL surrogate solution.

8.6.2 Stock Calibration Mix Standards: Pre-made, commercially available solutions.

- PAH Mix - ECS-A-032 (2000ug/mL)
- EPA 8270 Add-ons - ECS-A-031 (2000ug/mL)
- EPA 625 B/N Mix#1 - ECS-A-030 (2000ug/mL)
- Benzidines Mix - ECS-A-007 (2000ug/mL)
- Phenols Mix - ECS-A-006 (2000ug/mL)
- Custom Semivolatiles Mix (containing acetophenone, atrazine, benzaldehyde, biphenyl, and caprolactam) – Restek – cat # 57685 (2000ug/mL)
- PAH Two Component Mixture – C.I.L. (2000uf/mL)
- Custom Four Component Mixture - N.S.I Q-7610-O (2000ug/ml)

The surrogate stock mixes noted in sections 8.6.1 are also added to the stock standard mixture. Due to the highly reactive nature of Benzidine and Benzaldehyde and their tendency to degrade when mixed with all the other target analytes found in this method, two separate ICAL solutions are prepared.

8.6.2.1 Preparation of the first stock standard (100ug/mL) to ***10mL*** stock solution in methylene chloride is as follows:

8.6.2.1.1 EPA 8270 Add-ons (2000ug/mL): 0.5mL

8.6.2.1.2 EPA 625 B/N Mix #1 (2000ug/mL): 0.5mL

8.6.2.1.3 Phenols Mix (2000ug/mL): 0.5mL

8.6.2.1.4 PAH Mix (2000ug/mL): 0.5mL

8.6.2.1.5 Acid Surrogate (10000ug/mL): 100uL

8.6.2.1.6 B/N Surrogate (5000ug/mL): 200uL

8.6.2.1.7 Cust. Field Surrogate Mix (2000ug/mL): 0.5mL

8.6.2.1.8 Custom Four Component Mixture (2000ug/ml): 0.5ml

8.6.2.2 Preparation of the second stock standard (100ug/mL) to 10mL in methylene

chloride is as follows:

8.6.2.2.1 Benzidines Mix (2000ug/mL): 0.5mL

8.6.2.2.2 Custom Semivolatiles Mix (2000ug/mL): 0.5mL

8.6.2.3 Preparation of calibration levels using the above stock solutions. Eight levels are prepared using the first stock solution. Only 7 additional levels are prepared using the second stock solution.

8.6.2.3.1 **Level 8 (50ug/mL):** 0.5mL of Calibration Stock diluted to a final volume of 1.0mL in methylene chloride.

8.6.2.3.2 **Level 7 (25ug/mL):** 2.5mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride

8.6.2.3.3 **Level 6 (15ug/mL):** 1.5mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride.

8.6.2.3.4 **Level 5 (10ug/mL):** 1.0mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride.

8.6.2.3.5 **Level 4 (5.0ug/mL):** 0.5mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride.

8.6.2.3.6 **Level 3 (2.0ug/mL):** 0.20mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride.

8.6.2.3.7 **Level 2 (1.**0ug/mL): 0.1mL of Calibration Stock diluted to a final volume of 10mL in Methylene chloride.

8.6.2.3.8 **Level 1 (0.5ug/mL):** 0.05mL of Calibration stock diluted to a final volume of 10mL in methylene chloride.

8.6.3 Initial Calibration Verification (ICV):

Initial Calibration Verification primary solutions (200ug/mL):
Benzoic Acid Solution (2000ug/mL): Restek
Benzaldehyde Solution (2000ug/mL): Restek
Benzidine solution (1000ug/mL): Restek
Custom 8270 Mix (2000ug/ml): Restek
8270 Megamix (1000ug/ml): Restek

8.6.3.1 **Initial Calibration Verification solution (10ug/mL):** 0.05mL of Custom 8270 Mix and 0.1ml of 8270 Megamix diluted to a final volume of 10mL in methylene chloride.

8.6.3.2 **Initial Calibration Verification solution** for Benzoic Acid (10ug/mL): 50 uL of Benzoic Acid Solution diluted to a final volume of 10mL in Methylene chloride.

8.6.3.3 **Initial Calibration Verification solution** for Benzidine (10ug/mL): 100uL of Benzidine Solution diluted to a final volume of 10ml in Methylene Chloride.

8.6.3.4 **Initial Calibration Verification solution** for Benzaldehyde (10ug/ml): 50uL of Benzaldehdye solution diluted to a final volume of 10ml in Methylene Chloride.

8.6.4 Continuing Calibration Verification (CCV) solution (10ug/mL):

1.0mL of Calibration Stock diluted to a final volume of 10mL in methylene chloride. This solution is also used as the Level 5 of the ICAL.

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8.6.5 LCS1/Matrix Spiking Solution (100/500ug/mL): Prepared with the same standards and volumes used to formulate the first ICAL stock as seen in section 8.6.2.1., minus the 3 surrogate solutions, with additional Benzoic Acid. Brought to a final volume of 10ml in Methanol. Formulation Below.

8.6.5.1 EPA 8270 Add-ons (2000ug/mL): 0.5mL

8.6.5.2 EPA 625 B/N Mix #1 (2000ug/mL): 0.5mL

8.6.5.2.1 Phenols Mix (2000ug/mL): 0.5mL

8.6.5.2.2 PAH Mix (2000ug/mL): 0.5mL

8.6.5.2.3 Custom Four Component Mixture (2000ug/ml): 0.5ml

8.6.5.2.4 Benzoic Acid Solution (Restek 2000ug/ml): 2.0ml

8.6.6 LCS2/Matrix Spiking Solution (100ug/mL):: Prepared by diluting 0.5ml of the Restek Custom Semivolatiles Mix Cat# 57685(2000ug/ml) to 10mL in Methanol.

8.6.7 LCS3/Matrix Spiking Solution (100/250ug/mL): Prepared by diluting 0.5ml of Benzidines Mix (ECS-A-007 - 2000ug/mL), and 1.5ml of Benzidine Solution (Restek 31441 1000ug/ml) to 10mL in Methanol.

8.6.8 Internal Standard Primary Standard: Six component standard commercially available from Restek – PN 31206-510. 2000ug/mL solution containing 1,4-Dichlorobenzene-d4, Naphthalene-d8, Acenaphthene-d10, Phenanthrene-d10, Chrysene-d12, and Perylene-d12.

8.6.8.1 8270 IS working solution (250ug/mL): 3.125mL of Internal Standard Primary Standard diluted to a final volume of 25mL in methylene chloride. 20uL of the solution is spiked into 1mL sample and standard vials. The final concentration on column is 5.0ug/mL.

8.6.9 DFTPP Primary Standard: Four component standard commercially available from UltraScientific – PN GCM-150. 1000ug/mL solution containing Decafluorotriphenylphosphine, Benzidine, Pentachlorophenol, and 4,4'-DDT.

8.6.9.1 8270 GC/MS Tuning Solution (50ug/mL): 1.25mL of DFTPP Primary Standard diluted to a final volume of 25mL in methylene chloride.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

9.1.1 Method Blank: A method blank must be prepared once per every 20 samples or per extraction batch, whichever is more frequent.

Compounds of interest must not be detectable in the method blank at a concentration greater than the reporting limit.

Corrective Action: Extraction of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. The results are qualified with a "B" flag for any associated sample concentrations that are less then 10x the blank concentration for the analyte. For New Jersey regulatory work, all target analytes must be < RL except phthalates which must be < 5X the RL. Sample results are qualified with a "B" flag for analytes observed in the blank > RL and phthalates observed in the blank > 5X RL. Exceptions may be made with if the samples associated with the out of control method blank are non-detect for the compound of interest, or if sample concentrations are greater than 10x the blank levels. In such cases when the sample results are accepted, the client is notified in a project narrative associated with the sample results.

9.2 Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)

Laboratory control sample (LCS) and laboratory control duplicate (LCSD) must be prepared once per every 20 samples or per extraction batch, whichever is more frequent. The LCS/D may contain all target compounds of interest, or a smaller subset may be analyzed, and is extracted along with the samples as verification of the accuracy of the entire extraction procedure.

Project specific requirements, QAPPs, and/or other DQOs may supersede general limits and QC requirements.

Corrective Action: Base/Neutral compounds should be recovered at 40%-140% and Acid compounds at 30%-130% unless listed in Table D. If ≤10% of the total compounds are outside of these limits, and the recovery is at least 10% for these compounds, re-extraction is not required. If the LCS recovery is still out of control, re-extract and re-analyze the LCS and all associated samples. Samples cannot be reported until an acceptable LCS is obtained. Exceptions may be made in some cases for documented difficult/poor performing compounds such as Benzidine, Benzoic Acid, 4-Nitrophenol, Aniline, Pentachlorophenol, 2,4-Dinitrophenol, 2,4-Dimethylphenol, 4,6-Dinitro-2-methylphenol, Benzaldehyde, Caprolactam and Pyridine. An explanation of any out of control LCS recovery must be included in the project narrative to the client.

9.3 Initial Calibration Verification (ICV)

The initial calibration for each compound of interest must be verified prior to sample analysis. This is accomplished by analyzing a second source calibration standard (Section 8.6.3).

Calculate the percent recovery for each analyte. If the percent recovery for each analyte is within 70%-130% of their true value, then the calibration is assumed to be valid. If any compounds do not meet this criterion, attempts should be made to locate and correct the source of the problem. 10% of all analytes are allowed to be outside of 70%-130%, but must be within 60%-140%. The ICV may be reanalyzed once. If the standard failure continues, corrective action must be taken prior to sample analysis.

A new initial calibration must be performed and acceptable ICV results obtained prior to any sample analysis.

9.4 Continuing Calibration Verification (CCV)

On a daily basis after the DFTPP has passed, a mid-level (10ug/mL) continuing calibration standard which contains all of the analytes of interest is analyzed.

Refer to Section 10.4 for further information.

9.5 Matrix Spike/ Matrix Spike Duplicate (MS/MSD)

Matrix spike / matrix spike duplicate (MS/MSD) samples will be performed per batch of 20 samples of the same matrix processed together in those cases where the client does not specify omitting the MS/MSD for the analyses of their samples. In the case where the lab does not receive enough sample volume to perform an MS/MSD, an LCS and LCSD may be performed to document precision and bias.

See Section 12 for MS/MSD recovery limits and %RPD limits. Calculate the %RPD as described in Section 9.6.

Corrective Action: Repeat analysis or check to see that an analytical error has occurred. If the % recovery and/or RPD still exceeds the control limits, and the LCS/D is compliant; include a project narrative with the results to client noting that there may be potential matrix effects on the accuracy or precision of the reported results as evidenced by MS/MSD recoveries and/or %RPD outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses are performed upon client and/or QAPP request. *For Organic analyses, the matrix duplicate is usually in the form of the matrix spike duplicate, see Section 9.5.*

Acceptable relative percent differences (RPD) of duplicates is listed in Section12. Acceptance criterion is not applicable to sample concentrations less than 5 times the reporting limit. Calculate the RPD as follows:

$$RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:
R1 = sample Replicate #1
R2 = sample Replicate #2

Corrective Action: Repeat analysis if an analytical error is suspected. If the % RPD still exceeds the control limits; include a project narrative with the results to client noting that there may be potential matrix effects on the precision of the reported organic results as evidenced by the matrix duplicate % RPD outlier.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogate Spikes: Surrogate spikes must be added to QC and field samples to evaluate the extraction method performance.

See Section 12 for surrogate recovery acceptance criteria.

Corrective Action: Up to one surrogate can be out in each fraction (Acid and Base/Neutral) but not less than 10% recovery, before any corrective action is necessary. Analysis must be repeated once if an analytical error is suspected. If the percent recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm the sample matrix. If *obvious* matrix interferences are noted, consultation with the Semivolatiles Department Manager, Laboratory Director or QA Officer may be necessary to confirm the need for sample re-extraction. If no re-extraction occurs, the surrogate results and reasons for the no re-extract decision must be discussed in the project narrative to the client.

9.7.2 Internal Standards: Internal standards must be added to all sample extracts, QC samples and standards for quantitation purposes.

The internal standards in the samples should remain at constant area counts with respect to the continuing calibration analyzed at the beginning of the run. Sample IS areas must be 50% to 200% of the Internal Standards in the Continuing Calibration. Additionally, the RRT of the detected sample compounds must be within 0.06RRT units of the retention times of the associated continuing calibration standard.

Corrective Action: Analysis must be repeated once unless there are obvious sample matrix interferences, e.g. If the sample extract was very colored and viscous, or there are obvious chromatographic interferences. If *obvious* matrix interferences are noted, consultation with the Semivolatiles Department Manager, Laboratory Director or QA Officer may be necessary to confirm the need for sample re-analysis.

9.8 Method Sequence

- Tune
- CCV
- Method Blank
- LCS
- LCSD
- Samples

10. Procedure

10.1 Equipment Set-up

10.1.1 The **basic GC parameters** are as follows:

Injection Port Temp: 260°C
Oven Equib Time: 0.50 min
Oven Max: 350°C
Oven: On
Cryo: Off
Ambient: 45°C
Cryo Blast: Off

Initial Temp.: 40°C
Initial Time: 1.00 min

Level	Rate (°C /min)	Final Temp. (°C)	Final Time (min)
1	25.00	200	0
2	9.00	270	0
3	12.00	320	4

Final Time: 23.344

Please note that this may differ slightly from one instrument to another.

The **basic injection port parameters** are as follows:
Pulsed Splitless “Split” mode
Equilibrium Time: 0.50 minute
Temp: 260°C
Pressure: 9.2752 psi
Total Flow: 34.2mL/min
Split: NA
Split Flow: NA
Septum Purge Flow: 3mL/min
Injection Pulse Pressure: 30psi Until 0.2min
Purge Flow to Split Vent: 30mL/min at 0.6 min
Injection volume: 1uL

This may differ slightly from one instrument to another.

10.1.2 **Column conditions** parameters are as follows:

Constant Pressure: 9.1473 psi
Column flow: 1.2mL/min
Linear Velocity: 39.723 cm/sec

This may differ slightly from one instrument to another.

10.1.3 Mass Spectrometer Scan Parameters
Scan from mass 35.0 amu to 450 amu

10.1.4 Tuning

Tune acceptance must be verified at the beginning of every 12 hour analytical sequence. The DFTPP may be combined with the calibration verification standard as long as both tuning and calibration acceptance criteria are met.

Before the analytical standards are analyzed the mass spectrometer must be adjusted to meet the proper ion criteria for DFTPP. This is demonstrated by injecting into the GC/MS system 1 uL of a 50 ug/mL DFTPP solution.

Note: Within the Enviroquant software, Autofind is used first to evaluate Tune. Three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.

Background subtraction is required, and must be accomplished using a single scan acquired within 20 scans of the elution of DFTPP. The background subtraction should be designed only to eliminate column bleed or instrument background ions. Do not subtract part of the DFTPP peak or any other discrete peak that does not coelute with DFTPP.

Average or single scan may be used, if Autofind does not pass, but caution should be used in tuning on a single scan too close to the front or back of the DFTPP peak. If the system will not tune under ideal conditions, injection port maintenance, retuning, and/or a source clean should be performed.

The response of Benzidine and Pentachlorophenol, when monitored on a daily basis, will indicate the efficiency of the chromatography system. Benzidine and Pentachlorophenol should be present at their normal responses and no peak tailing should be visible. The tailing factor of Benzidine and Pentachlorophenol must not exceed 2. DDT breakdown must be <20%. Although moderate tailing may indicate maintenance on the instrument will be required soon, these additional compounds in the DFTPP standard are included as an aid in instrument performance evaluation.

The following DFTPP mass intensity criteria should be used:

DFTPP KEY MASSES AND ABUNDANCE CRITERIA

Mass	m/z Abundance criteria
68	Less than 2% of mass 69.
70	Less than 2% of mass 69.
197	Less than 2%of mass 198.
198	Base peak, or > 50% of Mass 442.
199	5-9% of mass 198.
365	Greater than 1% of mass 198.
441	Present but less than 24% of mass 442.

442 Base Peak, or > 50% of mass 198.
443 15-24% of mass 442.

10.2 Initial Calibration

10.2.1 After the DFTPP passes criteria, a set of multi-level calibration standards listed in Section 8.6.2.3 are analyzed, from low concentration to high. A minimum of five calibration levels are analyzed. The calibration standards are stored in the standards freezer. When the Initial Calibration is typed into a sequence, the standard ID for each of the levels must be noted in the “comments” of the sequence. (Refer to the Reagent Solvent and Standard Control SOP (1816) for further information regarding standard labeling convention.)

10.2.2 Once the standards have been analyzed, they are reduced by the search software of the Enviroquant data system. Once all the components are identified, a linear curve is calculated for the components. The criteria for evaluation are as follows:

The average response factor and the response factor of the lowest calibration standard of each target analyte should be checked against the recommended minimum response factor criteria found in Table C. This information may help to quickly identify poor responding compounds and the source of failure for initial calibrations. The %RSD for each target analyte must not exceed 20%. If the %RSD for any target exceeds 20%, the fit must be ≥0.99 quadratic or ≥.995 linear fit for all targets. A minimum of 5 levels are required for a linear calibration model and a minimum of 6 levels are required for a quadratic calibration model. When calculating calibration curves using the linear regression model, a quantitiation check must be performed on the lowest calibration point, or the point that corresponds to that compound’s established reporting limit. The recalculated concentration of the low calibration point (especially where linear regression fits are used) should be within ±50% of the standard's true concentration, and the recalculated concentrations of any calibration standards above the LLOQ should be within ±30%. If this criterion is not met then corrective action must take place and a new initial calibration must be performed prior to sample analysis.

10.2.3 Once the calibration curve is accepted, the Initial Calibration Verification (ICV) must be analyzed.

Reference standards from a separate source or different lot are analyzed after every initial calibration for evaluation of the calibration standard solutions. The response factors for all compounds should be evaluated against the minimum RF listed in Table C. The percent difference should not be greater than +/-30%D of the true value for all target compounds. If any compounds do not meet this criterion, attempts should be made to locate and correct the source of the problem. 10% of all analytes are allowed to be outside of 70%-130%, but must be within 60%-140%. The ICV may be reanalyzed once. If the standard failure continues, corrective action must be taken prior to sample analysis.

A new initial calibration must be performed and acceptable ICV results obtained prior to any sample analysis.

10.2.4 Once the calibration curve is accepted and prior to processing any samples, the analyst must demonstrate through the analysis of a MB or instrument blank that equipment and reagents are free from contaminants and interferences. If a peak is found in the blank that would prevent the identification or bias the

measurement of an analyte, the analyst should determine the source of the contaminant peak and eliminate it, if possible. Blanks are generally considered to be acceptable if target analyte concentrations are less than one half the LLOQ or are less than project specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected

10.3 Equipment Operation and Sample Processing

10.3.1 Sample Extraction and Cleanup

Samples for 8270 are generally extracted using one of the following SOPs:

- *Shaker Table Extraction* (2261)
- *Separatory Funnel Extraction* 3510C (2165)
- *Microscale Solvent Extraction* 3570 (2172)

Samples for 8270 may be cleaned up before transfer to the instrument laboratory using one of the following SOPs:

- *Gel Permeation Column Cleanup* SOP (2167)
- *Silica Gel Clean-up* SOP (2170) – limited target compounds only.

10.3.2 The preparation lab staff will transfer the samples to the instrument laboratory. The samples are generally brought to a 1.0 to 5.0mL final volume; 1.0mL is transferred and any remaining sample is put into archive. One aliquot of each sample is then logged into the Internal Chain of Custody book and placed in the sample extract holding refrigerator located in the instrument laboratory.

10.3.3 All samples and standards are spiked with the six Internal Standards (IS) compounds before analysis, Section 8.6.8.1. The internal standard is intended to be used for both quantitation and the establishment of relative retention times. Samples at a volume of 1.0mL (including the QC samples of a method blank and LCS) are spiked with 20uL of internal standard for a concentration of 5ug/mL. The samples are shaken briefly after the internal standard is added to ensure mixing. If a different volume of sample is transferred the volume of internal standard added is adjusted accordingly.

10.3.4 The analyst may decide to screen any samples to determine is an upfront dilution is needed. An analyst might decide a dilution is needed prior to analysis for high target analyte concentration or to protect the instrument from of matrix related interferences.

10.3.5 After the samples have been analyzed, the data files from the Instrument are transferred to the server. The samples are quantitated versus the proper method. The QCPRN1.MAC macro creates a form with which to easily check internal standard and surrogate criteria. The istdrpt macro may, also, be utilized to check the internal standard and surrogate criteria. The following should be reviewed initially:

10.3.5.1 Are all the surrogates within QC criteria? Please see Section 12 for surrogate information.

10.3.5.2 Are all the internal standards of the samples within 50-200% of the daily CCV standard? If not, the samples should be checked for matrix interferences that may be causing these issues. The IS peaks should also be evaluated for peak splitting or incorrect integration by the software. A sample may not need to be reanalyzed if it can be determined (with guidance from a supervisor) that the QC is exceeded due to matrix interference.

10.3.5.3 Are all target analytes within calibration range? If not, the sample should be diluted and re-analyzed. If a dilution is performed after the internal standard has already been added, it will be necessary to add additional IS in order to provide a concentration of 5ug/mL. Conversely, if a sample has been over-diluted, it may need to be analyzed at a lesser dilution to detect target analytes that may have been diluted out.

10.3.5.4 Are all analyses within 12 hour tune time? If a sample is analyzed outside tune time, it will need to be re-analyzed in another tune clock.

10.3.6 The sequence should be printed out from Environmental Data Analysis, initialed and dated, placed in the standard logbook.

10.3.7 If anything in the initial review of the data indicates that there should be a re-analysis or a re-extract, the reason for re-analysis or re-extract should be noted on the sequence.

10.3.8 If a re-extract is required, the “Organics Re-Prep Request Form” google doc should be filled out and an email should be sent to the extractions lab.

10.3.9 A standard sequence (saved as S8073001;”S” for semivolatile;”8” for BNA8; 0730 for July 30, and 01 is the first sequence that day) would appear as follows:

Tune: WG#-1, -4, etc. This is the Analytical working group number generated by the Alpha LIMS that denotes the Tune.

CCV: WG#-2, -3, etc. This is the Analytical working group number generated by the Alpha LIMS that denotes the calibration verification.

10.4 Continuing Calibration Verification (CCV)

10.4.1 On a daily basis after the DFTPP has passed, a mid-level (10ug/mL) continuing calibration standard which contains all of the analytes of interest is analyzed. The criteria for acceptance are:

10.4.1.1 The %D for all targets must be ≤20%. Up to 20% of all targets are allowed to have %D > 20% with high bias as long as all associated samples are non-detect for those analytes. In cases where compounds fail low, they may be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the LLOQ or project specific level of interest (e.g., by calibrating below the established LLOQ to confirm the non-detect, or by analyzing a standard near that level to confirm the analyte could be qualitatively identified if it were present. The retention times of the internal standards must be

within 0.5min of the mid-level standard of the most recent ICAL.

10.4.1.2 The area counts of the internal standards within the continuing calibration must be within 50 to 200% of the mid-level in previous ICAL.

10.4.1.3 Allowances may be made if in accordance with specific regional criteria.

10.4.1.3.1 New Jersey work may follow Data of Known Quality Protocols Technical Guidance whereas the %D for all target analytes must be ≤30% or corrective action must be taken.

10.4.1.3.2 Connecticut work may follow RCP criteria whereas if %D of >10% of target analytes is >30% corrective action must be taken.

10.4.1.3.3 Massachusetts work may follow MCP criteria whereas if ≤20% of compounds exceed criteria recalibration is not required as long as %D <40%.

10.5 Preventative Maintenance

All repair and non-routine maintenance records including outside service visits are maintained in the instrument maintenance logbooks.

Injection Port Maintenance: Maintenance should be done when the daily CCAL starts to demonstrate degradation. The type of samples analyzed will have an effect on how soon maintenance should be performed.

Septum Maintenance: The septum needs to be changed approximately every two-hundred injections. Unscrew the top septum nut, remove the pierced septum, and replace with a new 11mm Thermolite green septum (Restek) or equivalent. Screw the top septum nut back on.

Column Maintenance: Maintenance should be done when the daily CCAL starts to demonstrate degradation. The type of samples analyzed will have an effect on how soon maintenance should be performed. Generally maintenance is performed by trimming 6 cm off the front of the column. The column is then installed into the injection port liner, and the inlet nut is tightened.

11. Data Evaluation, Calculations and Reporting

11.1 Qualitative Analysis

11.1.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

11.1.2 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. A peak selected by the data system, based on the presence of target specific ions at a target specific retention time will be accepted as meeting this criteria.

11.1.3 The relative retention time of the sample component is within ± 0.06 RRT units of the RRT of the standard component, taking into consideration matrix effects which may cause retention time shifts.

11.1.4 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%). Outlying abundances maybe included due to the presence of non-target interference. The relative intensities are monitored daily. The relative intensities will be updated when they exceed established values from the reference spectrum.

11.1.5 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 50% the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

11.1.6 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e. a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

11.1.7 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes coelute, (i.e. only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum may contain extraneous ions contributed by the coeluting compound.

11.1.8 For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification (TICs) will be determined by the purpose of the analyses being conducted. Data system library searches must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

11.1.8.1 For example, the RCRA permit or waste delisting requirements may require the reporting of non target analytes. Only after a visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. Guidelines for tentative identification are:

11.1.8.1.1 Relative intensities of major ions in the reference spectrum (ions >10% of the most abundant ion) should be present in the sample spectrum.

11.1.8.1.2 The spectral library match must be $\geq$ 85% for a tentatively identification to be made.

11.1.8.1.3 The relative intensities of the major ions should agree within $\pm$ 20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30-70%).

11.1.8.1.4 Molecular ions in the reference spectrum should be present in

the sample spectrum.

11.1.8.1.5 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for background contamination or presence of co-eluting compounds.

11.1.8.1.6 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

11.2 Quantitative Analysis

11.2.1 Response factors and % RSD to evaluate Initial Calibration acceptability.

Calculate RF by:
where:

$$RF = \frac{area_{cmp}}{area_{is}} \times \frac{conc_{is}}{conc_{cmp}}$$

area cmp = Area of the characteristic ion for the compound being measured.
area is = Area of the characteristic ion for the specific internal standard.
conc. is = Concentration of the specific internal standard.
conc. cmp = Concentration of the compound being measured.
Calculate %RSD by:
where:

$$\%RSD = \frac{SD}{\bar{x}} \times 100 \qquad SD = \sqrt{\sum_{i=1}^{N} \frac{(x_i - \bar{x})^2}{N-1}}$$

%RSD = Percent Relative Standard Deviation
x = Average of RF's
SD = Standard Deviation
x_i = Analytical results of each level in the final reporting units
N=Number of results (levels)

11.2.2 For compounds in the initial calibration in which the Average of Response Factors calibration model is used:

Calculate %Difference (%D):

$$\%D = \frac{\overline{RF}_i - RF_c}{\overline{RF}_i} \times 100$$

where:

RFi - Initial Calibration average RF
RFc = Continuing Calibration RF

11.2.3 For compounds in the Initial Calibration for which the Linear Regression calibration model is used:

$$\%\ \text{Drift} = \frac{\text{Calculated [ ] – Theoretical [ ]}}{\text{Theoretical [ ]}} \times 100$$

11.2.4 Results of Water Analysis- calculation as performed in report form:

$$\text{Concentration (ug/L)} = \frac{(\text{Conc})\ (\text{Vf})\ (\text{DF})}{(\text{Vi})} \times 1000$$

where:
Conc =Raw on-column concentration obtained from the quantitation report using Initial Calibration results.
Vf = Final volume of extract (mL)
Vi = Volume of sample extracted (mL)
DF = Dilution factor, for manually prepared dilutions

11.2.5 Results of Sediment/Soil and Sludge Analysis- calculation as performed in report form:

$$\text{Concentration (ug/Kg)} = \frac{(\text{Conc})\ (\text{Vf})\ (\text{DF})}{(\text{W})\ (\%\text{S})} \times 1000$$

where:

Conc =Raw on-column concentration obtained from the quantitation report using Initial Calibration results.
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".
Vf = Extract final volume (mL)
W = Aliquot of sample (wet), g
%S = Sample % solid (in decimal form)

11.2.6 The calculation for percentage breakdown for DDT is:

$$\%\ \text{Breakdown DDT} = \frac{(\text{Area DDD} + \text{Area DDE})}{(\text{Area DDD} + \text{Area DDT} + \text{Area DDE})} \times 100$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All results for the organic compounds of interests are reportable without qualification if extraction and

analytical holding times are met, preservation (including cooler temperatures) are met, all QC criteria are met, and matrix interference is not suspected during extraction or analysis of the samples. If any of the below QC parameters are not met, all associated samples must be evaluated for the necessity of re-extraction and/or re-analysis.

If non-compliant organic compound results are to be reported, the Semi volatile Organics Department Manager, the Laboratory Director, and/or the QA Officer must approve the reporting of these results. The laboratory Project Manager shall be notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

QC Parameter	Acceptance Criteria
Initial Calibration Curve	≤20%RSD or ≥0.99 quadratic or ≥.995 linear fit for all targets. If the linear or quadratic calibration model is used, recalculation of the lowest calibration standard is required to be within +/-30% of the true value. Average RFs and the lowest calibration standard should be checked against the recommended minimum RFs listed in Table C as a troubleshooting tool.
Independent Check Verification	+/- 30%D for all compounds; ICV is still accepted if up to 10% of all compounds that fail to meet +/-30%D, are within 60%-140%D
Continuing Calibration Verification	Analyzed every 12hr or at the minimum of every 20 samples; ≤ 20%D for all compounds. 80% of target analytes must meet criteria. (Section 10.4)
Method Blank	No analyte detected at or above the reporting limit, “B” qualify analyte if detected concentration is less than 10X the concentration found in the associated sample(s)
Laboratory Control Sample / Lab Control Sample Duplicate	40%-140% (Base/Neutral compounds) and 30%-130% (Acid compounds) for all matrices; 30% RPD for soils; 20% RPD for waters; 10% allowed out provided %Recovery is at least 10%, exception made for “difficult” compounds (Section 9.2)
Matrix Spike / Matrix Spike Duplicate	Same as for LCS/D
Sample / Sample Duplicate	30% RPD for soils; 20% RPD for waters
Surrogates	Soil: 30-130% Waters: 30-130% for BN and 15-115% for Acid
Internal Standards	50% - 200% of the daily CCV area for the Internal Standards
Project specific QAPP and DQOs supersede the above limits.	

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOPs for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP 1732 MDL/LOD/LOQ Generation

SOP 1739 IDC/DOC Generation

SOP 1797 Waste Management and Disposal

SOP 1731 Manual Integration

SOP 1816 Solvent, Reagent, and Standard Control

SOP 1753 Laboratory Glassware Cleaning

16. Attachments

Table A: Surrogate Recovery Acceptance Criteria

Water: 15-115% for Acid compounds and 30-130% for Base/Neutral Compounds
Soils: 30-130%

Note: The above limits will be replaced by laboratory limits, when available. Limits are determined by sample DQO and matrix. The limits above may also vary with project specific requirements, QAPP, or DQOs.

Table B: Target Compounds and Quantitation Ions

Compound	Quantitation Ions Primary	Secondary
Aniline*	93	39, 93
Acenaphthene (CCC)	153	154, 152
Acenaphthene-d10 (IS)	164	162, 160
Acenaphthylene	152	151, 153
Acetophenone	105	77, 51
Anthracene	178	176, 179
Azobenzene *	182	77
Atrazine	200	173, 215
Benzaldehyde	77	105, 106
Benzo(a)Anthracene	228	229, 226
Benzo(a)pyrene D12 (SURR)	264	132
Benzidine*	184	183,92
Benzo(b)fluoranthene	252	253, 125
Benzo(k)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(a)pyrene	252	253, 125
Benzoic Acid*	105	122, 77
Biphenyl	154	153, 76
Benzyl Alcohol	108	79, 77
Bis(2-chloroethoxy)methane	93	95, 123
Bis(2-chloroethyl)ether	93	63, 95
Bis(2-chloroisopropyl)ether	45	77, 79
Bis(2-ethylhexyl)phthalate	149	167, 279
4-Bromophenylphenylether	248	250, 141
Butylbenzylphthalate	149	91, 206
Carbazole*	167	139,166
Caprolactam	113	55, 56
2-Chloroaniline	127	129
4-Chloroaniline	127	129
4-Chloro-3-methylphenol (CCC)	107	144, 142
2-Chloronaphthalene	162	127, 164
2-Chlorophenol	128	64, 130
4-Chlorophenylphenylether	204	206, 141
Hexachlorobenzene	284	142, 249
Chrysene	228	226, 229
Chrysene-d12 (IS)	240	120, 236

Table B, continued: Target Compounds and Quantitation Ions

Compound	Quantitation Ions Primary	Secondary
Dibenz(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
Di-n-butylphthalate	149	150, 104
1,2-Dichlorobenzene	146	148, 113
1,3-Dichlorobenzene	146	148, 113
1,4-Dichlorobenzene (CCC)	146	148, 113
1,4-Dichlorobenzene-d4 (IS)	152	115
3,3'-Dichlorobenzidine*	252	254, 126
2,4-Dichlorophenol (CCC)	162	164, 98
Diethylphthalate	149	177, 150
2,4-Dimethylphenol	107	122, 121
Dimethylphthalate	163	194, 164
4,6-Dinitro-2-methylphenol	198	182,77
2,4-Dinitrophenol (SPCC)	184	63, 154
2,4-Dinitrotoluene	165	63, 182
2,6-Dinitrotoluene	165	63, 121
Di-n-octylphthalate (CCC)	149	167, 150
Fluoranthene (CCC)	202	101, 100
Fluoranthene D10 (SURR)	212	106
Fluorene	166	165, 167
2-Fluorobiphenyl (SURR)	172	171,173
2-Fluorophenol (SURR)	112	64,91
Hexachlorobutadiene (CCC)	225	223, 227
Hexachlorocyclopentadiene (SPCC)	237	235, 272
Hexachloroethane	117	201, 199
Isophorone	82	95, 138
Indeno(1,2,3-cd)pyrene	276	138, 227
2-Methylnaphthalene	142	141
2-Methylphenol	108	107
4-Methylphenol	108	107
Naphthalene	128	129, 127
Naphthalene-d8 (IS)	136	68
2-Nitroaniline	65	92, 138
3-Nitroaniline	138	108, 92
4-Nitroaniline	138	108, 92
Nitrobenzene	77	123, 65
Nitrobenzene-d5 (SURR)	82	128, 54
2-Nitrophenol (CCC)	139	109, 65
4-Nitrophenol (SPCC)	109	139, 65
N-Nitrosodiphenylamine (CCC)	69	168, 167
N-Nitroso-di-n-propylamine (SPCC)	169	42, 101, 130
N-Nitrosodimethylamine*	74	42, 44
Pentachlorophenol (CCC)	266	264, 268
Pentachloronitrobenzene	237	214, 249
Perylene-d12 (IS)	264	260, 265
Phenanthrene	178	179, 176
Phenanthrene-d10 (IS)	188	94, 80
Phenol (CCC)	94	65, 66

Table B, continued: Target Compounds and Quantitation Ions

Compound	Quantitation Ions Primary	Secondary
Phenol-d5 (SURR)	99	42,71
Pyrene	202	200, 203
Pyridine	79	52
Terphenyl-d14 (SURR)	244	122. 212
1,2,4,5-Tetrachlorobenzene	216	214, 179
2,3,4,6-Tetrachlorophenol	232	131, 230
2,4,6-Tribromophenol (SURR)	330	332, 141
1,2,4-Trichlorobenzene	180	182, 145
2,4,5-Trichlorophenol	196	198, 97, 132, 99
2,4,6-Trichlorophenol (CCC)	196	198, 200

* Indicates compound is not part of standard reporting list.
Note: Target list will vary based on project specific requirements.

Table C: Recommended Minimum Response Factor Initial and Continuing Calibration

Semivolatile Compounds	Minimum Response Factor (RF)
Benzaldehyde	0.01
Phenol	0.8
Bis(2-chloroethyl)ether	0.7
2-Chlorophenol	0.8
2-Methylphenol	0.7
2,2'-Oxybis-(1-chloropropane)	0.01
Acetophenone	0.01
4-Methylphenol	0.6
N-Nitroso-di-n-propylamine	0.5
Hexachloroethane	0.3
Nitrobenzene	0.2
Isophorone	0.4
2-Nitrophenol	0.1
2,4-Dimethylphenol	0.2
Bis(2-chloroethoxy)methane	0.3
2,4-Dichlorophenol	0.2
Naphthalene	0.7
4-Chloroaniline	0.01
Hexachlorobutadiene	0.01
Caprolactam	0.01

Table C Continued: Recommended Minimum Response Factor Initial and Continuing Calibration

Semivolatile Compounds	Minimum Response Factor (RF)
Hexachlorocyclopentadiene	0.05
1,1'-Biphenyl	0.01
2-Chloronaphthalene	0.8
2-Nitroaniline	0.01
Dimethyl phthalate	0.01
2,6-Dinitrotoluene	0.2
Acenaphthylene	0.9
3-Nitroaniline	0.01
Acenaphthene	0.9
2,4-Dinitrophenol	0.01
4-Nitrophenol	0.01
Dibenzofuran	0.8
2,4-Dinitrotoluene	0.2
Diethyl phthalate	0.01
1,2,4,5-Tetrachlorobenzene	0.01
4-Chlorophenyl-phenyl ether	0.4
Fluorene	0.9
4-Nitroaniline	0.01
4,6-Dinitro-2-methylphenol	0.01
4-Bromophenyl-phenyl ether	0.1
N-Nitrosodiphenylamine	0.01
Hexachlorobenzene	0.1
Atrazine	0.01
Pentachlorophenol	0.05
Phenanthrene	0.7
Anthracene	0.7
Carbazole	0.01
Di-n-butyl phthalate	0.01
Fluoranthene	0.6
Pyrene	0.6
Butyl benzyl phthalate	0.01
3,3'-Dichlorobenzidine	0.01
Benzo(a)anthracene	0.8
Chrysene	0.7
Bis-(2-ethylhexyl)phthalate	0.01
Di-n-octyl phthalate	0.01

Table C Continued: Recommended Minimum Response Factor Initial and Continuing Calibration

Semivolatile Compounds	Minimum Response Factor (RF)
Benzo(b)fluoranthene	0.7
Indeno(1,2,3-cd)pyrene	0.5
Dibenz(a,h)anthracene	0.4
Benzo(g,h,i)perylene	0.5
2,3,4,6-Tetrachlorophenol	0.01

Table D: Difficult Target Recovery Limits

(Soils)

Semivolatile Compounds	Recovery Limit
Benzoic Acid	10 – 44%
Benzidine	10 – 72%
2,4-Dinitrophenol	10 – 96%
Pyridine	10 – 105%
Hexachlorocyclopentadiene	12 – 87%
Benzaldehyde	26 – 77%
Hexachloroethane	25 – 81%
Aniline	25 – 84%
N-Nitrosodimethylamine	27 – 88%

Aqueous)

Semivolatile Compounds	Recovery Limit
Benzoic Acid	10 – 47%
Caprolactam	15 – 40%
Benzidine	10 – 82%
Phenol	18 – 49%
Pyridine	14 – 63%
4-Nitrophenol	17 – 65%
Hexachloroethane	10 – 97%
N-Nitrosodimethylamine	27 – 70%
Hexachlorocyclopentadiene	10 – 109%

Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS)

Reference Method No.: **EPA 8270 E**

Reference: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update VI (Phase II), June, 2018.

1. Scope and Application

Matrices: This method is used to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and wastewater samples.

This method is used to quantitate most neutral, acidic, and basic organic compounds that are soluble in methylene chloride and capable of being eluted, without derivatization, as sharp peaks from a gas chromatographic fused-silica capillary column coated with a slightly polar silicone.

Table 9 lists “difficult” compounds that may require special treatment when being determined by this method.

Approval of any method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability (Section 13.2).

2. Summary of Method

The samples are introduced into the GC/MS by injecting 1µL of the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

Analytes eluted from the capillary column are introduced into the mass spectrometer via direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of standards run on the same GC/MS system. Quantitation is accomplished by comparing the response of quantitation ion relative to an internal standard using a calibration curve.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 6 lists our routine reporting limits.

4. Interferences

4.1 Only high purity helium is used in the GC system to eliminate this source of possible contamination. The helium (carrier gas) is certified by the gas supplier.

4.2 Preventive instrument maintenance is performed routinely. Section 10.5 details the maintenance steps.

4.3 Glassware must be scrupulously cleaned. This procedure is detailed in the Organic Extraction Glassware Cleaning & Handling SOP/1953.

4.4 Contaminated solvents or reagents are also possible sources of contamination. All solvents used are pesticide grade or equivalent, and reagents are purchased as certified contaminant free.

4.5 Contamination by carry-over can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered (concentrations greater than 2x the highest concentration) and the next sample has reportable hits this sample should to be re-analyzed for confirmation based on analyst discretion.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC/MS laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab for future disposal by the Health and Safety Officer. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Flammable solvent bottles must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 1L amber glass jars with teflon-lined lids. For LVI, aqueous samples are collected in two 275mL amber glass jars with teflon-lined lids. Solid samples are collected in 250mL wide-mouth glass jars with teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ± 2ºC. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ± 2ºC.

6.3 Sample Handling

Aqueous samples must be extracted within 7 days of sample collection, solid samples within 14 days of collection. Once extracted, the samples must be analyzed within 40 days of the extraction date.

7. Equipment and Supplies

7.1 Gas Chromatograph/Mass Spectrometer System:

7.1.1 Gas Chromatograph, Hewlett Packard 6890 (or equivalent): An analytical system complete with a temperature-programmable gas chromatograph configured for split/splitless-injection and all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source.

7.1.2 Column: Rxi-5Sil MS30m x 0.32mm ID, 0.25µm film thickness or column of similar configuration.

7.1.3 Mass Spectrometer, Hewlett Packard 5973 (or equivalent): Scanning from 35 to 500 amu every 1 second or less, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine (DFTPP) which meets the criteria in Table 1 when 1 µL of the GC/MS tuning standard is injected through the GC (50ng of DFTPP).

7.1.4 Data System: A computer system is interfaced to the Mass Spectrometer. The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer software allows the analyst to search for any GC/MS data file for ions of specific mass and plot such ion abundances versus time or scan number. *HP ChemServer* software is used for data acquisition and *MSD Chemstation/Enviroquant version E.02.02* is used for data reduction.

7.2 Syringe: 10 µL.

7.3 Volumetric Flasks, Class A: Appropriate sizes with ground-glass stoppers.

7.4 Vials: Glass autosampler vials with polytetrafluoroethylene (PTFE)-lined crimp top caps.

8. Reagents and Standards

8.1 Stock Standard Solutions

Certified stock standard solutions, traceable to NIST, when available, are purchased from commercial vendors. They can be replaced with different standards as long as they contain all target analytes.

All stock standards, lot number, catalog number, expiration date, preparation date and initials are recorded in a logbook. Standards are stored in the refrigerator or freezer. Store away from any light source at 6 °C when not in use (−10 °C is recommended).

Stock standard expire 6 months from the date of preparation or on the earliest expiration date of any of the stock solution used to prepare it.

Please note that the following preparation instructions and stock standards are included for illustration purposes and may be modified as needed (ex. to accommodate standard availability or client requests), however final concentrations for the initial calibration levels shall always follow the example in 8.1.4.

Vendor	Standard	Catalog#	Concentration
Restek			
	8270 Mega Mix	31850	500-1000ug/mL
	Benzoic Acid Mix	31879	2000ug/mL
	Acid Surrogate Mix	31087	10000ug/mL
	B/N Surrogate Mix	31086	5000ug/mL
	Benzaldehyde Standard	33017	2000ug/mL
	Custom AP9 ICAL Standard	571813-FL	2000ug/mL
	Custom ADP Standard	572745-FL	2000ug/mL
	Benzidine Mix	31834	2000ug/mL
	SV Internal Standard Mix	31206	2000ug/mL
	1,4-Dioxane	30287	2000ug/mL
	Custom CLP 04.1 BNA Surrogate Mix	571320-FL	1000ug/mL
Absolute	Aromatic Amines Mix	99410	2000ug/ml
Ultra	Semi-Volatiles GC/MS Tuning Standard	GCM-150-1	1000ug/mL

8.1.1 ABN Stock Standard, 200ug/mL

Use 5mL of each of the following:
- Benzoic Acid Mix
- Benzidine Mix

and use 10mL of each of the following:
- 8270 Mega Mix
- Custom CLP 04.1 BNA Surrogate Mix

Bring up to 50mL volume with DCM.

8.1.2 AP9 Additional Compounds Stock Standard, 200ug/mL

Use 5mL of each of the following:
- Custom AP9 ICAL Standard
- Benzaldehyde Standard

Bring up to 50mL volume with DCM.

8.1.3 ADP Stock Standard, 200ug/ml

Use 5ml of:
- Custom ADP Standard

Bring up to 50mL volume with DCM.

8.1.4 Calibration Standard

A minimum of 5 calibration standards must be included for each analyte:

Calibration Curve Levels	
Level	Concentration ug/mL
1	1.0
2	2.0
3	3.0
4	5.0
5	10
6	20
7	50
8	100
9	150
10	200

LVI Calibration Curve Levels	
Level	Concentration ug/mL
1	0.2
2	0.4
3	1.0
4	2.0
5	3.0
6	5.0
7	10
8	15
9	35
10	50

*LVI- Low Volume Initiative

8.2 Internal Standard Solution

The internal standards are:

1,4-dichlorobenzene-d_4
naphthalene-d_8
acenaphthene-d_{10}
phenanthrene-d_{10}
chrysene-d_{12}
perylene-d_{12}

Each 500µL of standards, blank and sample extracts are spiked with 10µL of SV Internal Standard Mix, resulting in a concentration of 40ng/ µL.

For the LVI method, a 1:10 dilution is made of the Internal Standard Stock Solution. 500µL of standards, blank and sample extracts are spiked with 10µL of this preparation, resulting in a concentration of 4ng/ µL.

8.3 GC/MS Tuning Standard

The tuning standard is a methylene chloride solution containing 50ng/µL of decafluorotriphenylphosphine (DFTPP). The standard also contains 50ng/µL each of 4,4'DDT, pentachlorophenol, and benzidine to verify injection port inertness and GC column performance.

Prepare the GC/MS Tuning Standard with 25µL GCM-150 and 475µL Dichloromethane.

8.4 Surrogate Spiking Solution

8.4.1 Extraction Surrogate Preparation

In a 1000mL volumetric flask, add 5ml of 31086 and 31087. Bring up to volume with Acetone. The final concentration is 50µg/mL for the acid surrogates and 25µg/mL for the B/N surrogates.

8.4.2 LVI Extraction Surrogate Preparation

The LVI surrogate is a 10 fold dilution of the surrogate solution prepared in 8.4.1. For example, to make 200mL of LVI surrogate, add 20mL of 8.4.1 to a 200mL volumetric flask

and fill to volume with Acetone. The resulting surrogate concentration is 5µg/mL for the acid surrogates and 2.5µg/mL for the B/N surrogates.

8.5 Spike Solution (LCS, MS, MSD)

Spike Solution Preparation

ABN SPK1:
In a 500ml volumetric flask, add 20ml of 8270 Mega Mix #31850, 10ml of Benzoic Acid Mix #31879, 10ml Custom AP9 ICAL Standard #571813-FL and 10ml Benzaldehyde Standard #33017. Bring up to volume with Acetone. The final concentration is 40µg/ml.

Note: the LVI ABN SPK1 is prepared by making an 8 fold dilution of the 40µg/ml ABN SPK (in acetone), resulting in a 5µg/ml LVI ABN SPK1.

ABN SPK2:
In a 500ml volumetric flask, add 10ml Benzidine Mix #31834 and 10mL Custom ADP Standard #572945-FL. Bring up to volume with Acetone. The final concentration is 40µg/ml.

Note: the LVI ABN SPK2 is prepared by making an 8 fold dilution of the 40µg/ml ABN SPK (in acetone), resulting in a 5µg/ml LVI ABN SPK2.

8.6 Dichloromethane (DCM): Pesticide quality.

8.7 Acetone: Pesticide quality.

9. Quality Control

9.1 Blank(s)

Extraction blanks are performed with each extraction batch of 20 or less samples. The extraction blank must not contain any of the reportable analytes above the reporting limit. Corrective actions:

- No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample

- If the blank have reportable hits and re-extraction could not be performed due to lack of additional sample volume, the sample results are reported and qualified with “B” flag for any associated samples that concentration is less than 10x the blank concentration

For NJ regulatory work the method blank must have all the target analytes less than RL except for Phthalates which must be less than 5x of the RL. Sample results are qualified with “B” flag for analytes observed in the blank greater than RL and the Phthalates observed in the blank greater than 5x RL

The surrogate recoveries must also be within the acceptance criteria listed in Table 2. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary.

9.2 Laboratory Control Sample and Laboratory Control Sample Duplicate (LCS / LCSD)

A Laboratory Control Sample/Laboratory Control Sample Duplicate pair (LCS/LCSD) are extracted and analyzed with each analytical batch of 20 or fewer samples.

The LCS/LCSD acceptance criteria are based the performance standard control limits. These performance standards are based on commonly used state programs RCP,MCP etc. See table 3. Less than 10% of total compounds may be outside of control limits provided that recoveries are >10%. Note: this does not apply to difficult analytes listed in Table 9 which may be accepted at recoveries <10. If >10% of analytes are recovered above control limits, this is deemed acceptable as long as the analytes in question are not detected in associated samples.

If these criteria are not met, the entire batch is re-extracted. If re-extraction is not possible, due to insufficient sample or holding time exceedance, the analyst must write up the failure on a narrative sheet for inclusion in the client report.

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.2.7.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike and Matrix Spike Duplicate (MS / MSD)

A matrix spike/matrix spike duplicate pair is extracted and analyzed for each batch of 20 or fewer samples per client request. The MS/MSD acceptance criteria are based on performance standards. See table 3 for individual control limits. If the recovery criteria are not met, but are met in the LCS/LCSD, this is noted on a narrative sheet for inclusion in the client report.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

All extracted samples and associated QC are spiked with surrogates. The acceptable surrogate recovery limits are listed in Table 2.

Corrective action: Up to one surrogate can be out in each fraction (Acid and Base/Neutral) but not less than 10% recovery, before any corrective action is necessary. Otherwise, analysis must be repeated once to see if an analytical error has occurred. If the % recovery still exceeds the control limits the sample must be re-extracted and re-analyzed to confirm sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

Re-extraction is not required if surrogate recoveries are high and target analytes are not detected in the sample.

9.7.2 Internal Standards

If the area for any of the internal standards in the samples changes by a factor of two (-50% to +100%) from that in the CCV, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is:

- Degradation Check
- DFTPP
- Continuing or Daily Standards (1 – 3)*
  (1) ABN 50 ppm
  (2) AP9 50 ppm
  (3) ADP 50 ppm
- Method/Instrument Blank
- Samples
- QC (as required)

***Additional Continuing standards may be run at the analyst's discretion or by client request.**

10. Procedure

10.1 Equipment Set-up

10.1.1 GC/MS Operating Conditions:

Typical GC/MS operating conditions are listed below, but may be altered as long as method performance criteria are met.

Mass range:	35 – 500 amu
Scan time:	3.15 second / scan
Initial temperature:	50°C, hold for 1.5 minutes
Temperature program:	28°C/minute to 250°C then 9°C/minute to 320°C
Final temperature:	320°C for 1.50 min
Injector temperature:	300°C
Transfer line temperature:	280°C
Source temperature:	230ºC
Injector:	split ratio 5:1; 11.7mL/min
Injection volume:	1µL
Carrier gas:	helium at 523 cm/second (2.0 mL/min) constant flow

GC/MS Operating Conditions for LVI method:

Mass range:	35 – 550 amu
Scan time:	3.15 second / scan
Initial temperature:	45°C, hold for 4 minutes
Temperature program:	25°C/minute to 250°C then 20°C/minute to 320°C
Final temperature:	320°C for 4.3 min
Injector temperature:	270°C
Transfer line temperature:	280°C
Source temperature:	320ºC
Injector:	split ratio 5:1; 8.57mL/min
Injection volume:	2µL
Carrier gas:	helium at 1.7148mL/min) constant flow

10.1.2 GC/MS Tune:

At the beginning of every 12 hour sequence, analyze DFTPP tuning solution (Section 8.3).

The resultant mass spectrum for DFTPP must meet the criteria given in Table 1 before sample analysis begins. The mass spectrum of DFTPP should be acquired in the following manner:

(1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.
(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of DFTPP.

The GC/MS tuning standard is also used to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD must not exceed 20%. Benzidine and pentachlorophenol must be present at their normal responses and no peak tailing must be visible.

The tailing factor for benzidine and pentachlorophenol must be calculated in every DFTPP run. (See Table 4)

If degradation is excessive and/or poor chromatography is noted, the system needs maintenance (see Section 10.5).

10.2 Initial Calibration

10.2.1 Prepare calibration standards for all target analytes at a minimum of five concentration levels as specified in Section 8.1.4.

10.2.2 Add 10µL of Internal Standard to each calibration standard directly into the autosampler vial containing 500µL of standard. Analyze each calibration standard under the conditions specified in Section 10.1.1.

10.2.3 Record the calibration standard, unique lab identifier code (lot), concentration, and analyst's initials in the analytical sequence list.

10.2.4 In each standard, calculate the response factor (RF) for each analyte, the average RF, and the relative standard deviation (RSD) of the RFs, using the Enviroquant data processing software. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

It is recommended that a minimum response factor for the most common target analytes, as noted in Table 8, be demonstrated for each individual calibration level as a means to ensure that these compounds are behaving as expected. In addition, meeting the minimum response factor criteria for the lowest calibration standard is critical in establishing and demonstrating the desired sensitivity.

10.2.5 Initial Calibration %RSD Criteria:

For all analytes, the RSD must be ≤ 20% for the mean response factor to be used for sample quantitation.

If the RSD for any target analyte exceeds 20%, the fit must be ≥0.99 quadratic or ≥ 0.995 linear fit for all target analytes. A minimum of 5 levels are required for a linear calibration model and a minimum of 6 levels are required for a quadratic calibration model. When calculating calibration using the linear regression model, a quantitation check must be performed on the lowest calibration point, or the point that corresponds to that compound's established reporting limit.
For RCP, the RSD must be ≤ 15% for the mean response factor to be used for sample quantitation

Percent Relative Error (%RE): For linear and quadratic fit compounds, the %RE must be calculated for the standard at or near the midpoint and at the lowest level of the initial calibration. The %RE between the calculated and expected concentration of an analyte must be ≤ 30%, otherwise recalibration is necessary.

10.2.6 Evaluation of Retention Times:

The relative retention time (RRT) of each target analyte in each calibration standard should agree within 0.06 RRT units.

10.2.7 Initial Calibration Verification (Second Source Verification)

10.2.7.1 The initial calibration (Section 10.2) for each compound of interest must be verified prior to sample analysis. This is accomplished by analyzing second source calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.2.7.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

If the % Difference for each analyte is ± 30%, then the calibration is assumed to be valid. If this criterion is not met, then corrective action must be taken prior to the analysis.

For RCP, if the % Difference for each analyte is ± 20%, then the calibration is assumed to be valid. If this criterion is not met, then corrective action must be taken prior to the analysis.

10.2.7.3 In cases where compounds fail (greater than 30% difference), they may still be reported as non-detects.

10.2.7.4 Once the calibration curve is accepted and prior to processing any samples, the analyst must demonstrate through the analysis of a Method Blank or Instrument Blank that equipment and reagents are free from contaminants and interferences. If a peak is found in the blank that would prevent the identification or bias the measurement of an analyte, the analyst should determine the source of the contaminant peak and eliminate it, if possible. Blank are generally considered to be acceptable if target analyte concentration are less than one half the LLOQ or are less than project specific requirements. Blanks may contain analyte concentrations are greater than acceptance limits if the associated samples in the batch are unaffected.

10.3 Equipment Operation and Sample Processing

10.3.1 GC/MS Analysis of Samples

10.3.1.1 Allow the sample extracts to warm to room temperature.

10.3.1.2 Transfer all of the sample extract to a 1.5mL vial. Remove 500µL of sample extract to another vial, and add 10µL of the internal standard solution (Section 8.2).

10.3.1.3 The autosampler is programmed to inject 1µL aliquot of the sample extract into the GC/MS system, using the same instrument conditions that were used for calibration. The injection volume of the sample must be the same as the volume used for the calibration standard.

10.3.1.4 If the response of any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed.

10.3.2 Qualitative Identification

Perform first level data review. Obtain the primary m/z (Table 5) masses for each parameter of interest. The following criteria must be met to make qualitative identification:

- Compare the background subtracted mass spectra for the sample to the reference spectra. The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.
- The retention time must fall within ± 0.1 minutes of the retention time of the compound in the analytical standard. However, analyst experience must be used in making the qualitative identification.
- The relative peak height of the one characteristic mass must fall within 30% of the relative intensity of the mass in a reference mass spectrum. The reference spectrum is obtained from a standard analyzed on the GC/MS system.

Structural isomers that have very similar mass spectra are identified only if the resolution between authentic isomers in a standard mix is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

10.4 Continuing Calibration

10.4.1 The initial calibration (Section 10.2) for each compound of interest must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing calibration standards at a concentration near the midpoint concentration for the calibrating range of the GC/MS.

10.4.2 Analyze the standards and calculate the % Difference for each analyte according to the formula in Alpha's Quality Manual.

If the % Difference for each CCV analyte is $\leq$ 20%, then the calibration is assumed to be valid. If the criterion is not met for more than 20% of the compounds then corrective action must be taken.

Due to the large number of analytes present, allowances may be made for a RF that drifts out high, as long as there are no positive hits for that particular analyte in any of the associated samples.

10.4.3 If this criterion is exceeded, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.4.4 If routine maintenance does not return the instrument performance to meet the QC requirements based on the last initial calibration, then a new initial calibration must be performed.

10.4.5 Internal Standard Retention Time

The retention times of the internal standards in the calibration verification standard is evaluated after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard of the most recent initial calibration, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.6 Internal Standard Response

Refer to section 9.7.2

10.5 Preventive Maintenance

When poor sensitivity is observed, replacement of the injector liner and seal may solve the problem. If not, clip approximately 3 – 6 inches from the injector end of the GC column. If the sensitivity does not improve it may be necessary to replace the split line or the injector weldment assembly. If the problem persists, it may be necessary to replace the GC column.

Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

11. Data Evaluation, Calculations and Reporting

When a parameter is identified, the quantitation of that parameter must be based on the integrated abundance of the quantitation characteristic m/z given in Table 5

Calculate the concentration in the sample using the average response factor (RF) from the initial calibration curve according to the formula in Alpha's Quality Manual.

After performing technical data review, validating that all QC criteria have been met and confirming all positive hits, the data report is sent electronically to the LIMS computer for generation of the client report. There are two levels of review of the data in the LIMS system prior to release of data. These reviews must be done by two separate individuals.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument logbook.

All batch and sample specific QC criteria outlined in Section 9 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample

volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

Alpha SOP/1732 DL/LOD/LOQ Generation

Alpha SOP/1739 IDC/DOC Generation

Alpha SOP/1729 Waste Management and Disposal SOP

16. Attachments

Table 1: DFTPP Key Ions and Ion Abundance Criteria

Table 2: Acceptable Surrogate Spike Recovery Limits

Table 3A: Acceptable Aqueous QC Limits

Table 3B: Acceptable Soil QC Limits

Table 4: Tailing Factor Calculation

Table 5: Characteristic Ions for Semivolatile Compounds

Table 6: Reported Detection Limits

Table 7: Semivolatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Table 8: Recommended Minimum Response Factor Criteria

Table 9: Difficult analytes

TABLE 1

DFTPP KEY IONS AND ION ABUNDANCE CRITERIA

Mass	Ion Abundance Criteria
68	< 2% of mass 69
69	Present
70	< 2% of mass 69
197	< 2% of mass 198
198	Base peak or present
199	5-9% of mass 198
365	> 1% of Base Peak
441	Present but less than 24% mass 442
442	Base peak, or present
443	15-24% of mass 442

TABLE 2

ACCEPTABLE SURROGATE SPIKE RECOVERY LIMITS

Analytical Fraction	Surrogate Compound	Water	Soil/Sediment
BN-8270D	Nitrobenzene-d_5	23-120%	23-120%
BN-8270D	2-Fluorobiphenyl	15-120%	30-120%
BN-8270D	p-Terphenyl-d_{14}	41-149%	18-120%
Acid-8270D	Phenol-d_6	10-120%	10-120%
Acid-8270D	2-Fluorophenol	21-120%	25-120%
Acid-8270D	2,4,6-Tribromophenol	10-120%	10-136%

It is allowable for one surrogate from each fraction be outside acceptance criteria, provided a minimum recovery of 10% has been achieved.

TABLE 3A

ACCEPTABLE AQUEOUS QC LIMITS

	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)		CT TARGET COMPOUND LIST (Aqueous)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
1,2,4,5-Tetrachlorobenzene			70-130	20	40-140	20
1,2,4-Trichlorobenzene	39-98	30	70-130	20	40-140	20
1,2-Dichlorobenzene	40-140	30	70-130	20		
1,3-Dichlorobenzene	40-140	30	70-130	20		
1,3-Dinitrobenzene	15-130	30				
1,4-Dichlorobenzene	36-97	30	70-130	20		
1-Methylnaphthalene	41-103	30				
2,3,4,6-Tetrachlorophenol			70-130	20		
2,4,5-Trichlorophenol	30-130	30	70-130	20	30-130	20
2,4,6-Trichlorophenol	30-130	30	70-130	20	30-130	20
2,4-Dichlorophenol	30-130	30	70-130	20	30-130	20
2,4-Dimethylphenol	30-130	30	70-130	20	30-130	20
2,4- Dimethylaniline	40-140	30	70-130	20		
3,4- Dimethylaniline	40-140	30	70-130	20		
2,3- Dimethylaniline	40-140	30	70-130	20		
2,4,5-Dimethylaniline	40-140	30	70-130	20		
4-Chlorotoluidine	40-140	30	70-130	20		
2-Ethylaniline	40-140	30	70-130	20		
O-toluidine	40-140	30	70-130	20		
2-Napthylamine	40-140	30	70-130	20		
2,4-Dinitrophenol	20-130	30	20-130	20	30-130	20
2,4-Dinitrotoluene	24-96	30	70-130	20	40-140	20
2,6-Dinitrotoluene	40-140	30	70-130	20	40-140	20
2-Chloronaphthalene	40-140	30	70-130	20	40-140	20
2-Chlorophenol	27-123	30	70-130	20	30-130	20
2-Methylnaphthalene	40-140	30	70-130	20	40-140	20
2-Methylphenol	30-130	30	70-130	20	30-130	20
2-Nitroaniline	52-143	30	70-130	20	40-140	20
2-Nitrophenol	30-130	30	70-130	20	30-130	20
3,3'-Dichlorobenzidine	40-140	30	70-130	20	40-140	20
3,3'-Dimethylbenzidine			20-160	20		
3-Methylphenol/4-Methylphenol	30-130	30	20-160	20	30-130	20
3-Nitroaniline	25-145	30	70-130	20	40-140	20
4,6-Dinitro-o-cresol	20-164	30	70-130	20	30-130	20
4-Bromophenyl phenyl ether	40-140	30	70-130	20	40-140	20
4-Chloroaniline	40-140	30	20-160	20	40-140	20
4-Chlorophenyl phenyl ether	40-140	30	70-130	20	40-140	20
4-Nitroaniline	51-143	30	70-130	20	40-140	20
4-Nitrophenol	10-80	30	20-160	20	30-130	20
Acenaphthene	37-111	30	70-130	20	40-140	20
Acenaphthylene	45-123	30	70-130	20	40-140	20

	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)		CT TARGET COMPOUND LIST (Aqueous)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Acetophenone	39-129	30	70-130	20		
Aniline	40-140	30	20-160	20	40-140	20
Anthracene	40-140	30	70-130	20	40-140	20
Atrazine			70-130	20		
Azobenzene	40-140	30	70-130	20		
Benzaldehyde			20-160	20		
Benzidine	10-75	30	20-160	20		
Benzo(a)anthracene	40-140	30	70-130	20	40-140	20
Benzo(a)pyrene	40-140	30	70-130	20	40-140	20
Benzo(b)fluoranthene	40-140	30	70-130	20	40-140	20
Benzo(ghi)perylene	40-140	30	70-130	20	40-140	20
Benzo(k)fluoranthene	40-140	30	70-130	20	40-140	20
Benzoic Acid	10-164	30	20-160	20		
Benzyl Alcohol	26-116	30	20-160	20		
Biphenyl	40-140	30	70-130	20		
Bis(2-chloroethoxy)methane	40-140	30	70-130	20	40-140	20
Bis(2-chloroethyl)ether	40-140	30	70-130	20	40-140	20
Bis(2-chloroisopropyl)ether	40-140	30	70-130	20	40-140	20
Bis(2-Ethylhexyl)phthalate	40-140	30	70-130	20	40-140	20
Butyl benzyl phthalate	40-140	30	70-130	20	40-140	20
Caprolactam			20-160	20		
Carbazole	55-144	30	70-130	20	40-140	20
Chrysene	40-140	30	70-130	20	40-140	20
Dibenzo(a,h)anthracene	40-140	30	70-130	20	40-140	20
Dibenzofuran	40-140	30	70-130	20	40-140	20
Diethyl phthalate	40-140	30	70-130	20	40-140	20
Dimethyl phthalate	40-140	30	70-130	20	40-140	20
Di-n-butylphthalate	40-140	30	70-130	20	40-140	20
Di-n-octylphthalate	40-140	30	70-130	20	40-140	20
Fluoranthene	40-140	30	70-130	20	40-140	20
Fluorene	40-140	30	70-130	20	40-140	20
Hexachlorobenzene	40-140	30	70-130	20	40-140	20
Hexachlorobutadiene	40-140	30	70-130	20	40-140	20
Hexachlorocyclopentadiene	40-140	30	20-160	20	40-140	20
Hexachloroethane	40-140	30	20-160	20	40-140	20
Indeno(1,2,3-cd)Pyrene	40-140	30	70-130	20	40-140	20
Isophorone	40-140	30	70-130	20	40-140	20
Naphthalene	40-140	30	70-130	20	40-140	20
Nitrobenzene	40-140	30	70-130	20	40-140	20
NitrosoDiPhenylAmine(NDPA)/ Diphenylamine (DPA)	40-140	30	70-130	20	40-140	20
n-Nitrosodimethylamine	22-74	30	20-160	20		
n-Nitrosodi-n-propylamine	29-132	30	70-130	20	40-140	20
P-Chloro-M-Cresol	23-97	30	70-130	20	30-130	20
Pentachlorophenol	9-103	30	20-160	20	30-130	20
Pentachloronitrobenzene					40-140	20

	STANDARD TARGET COMPOUND LIST (Aqueous)		NEW JERSEY TARGET COMPOUND LIST (Aqueous)		CT TARGET COMPOUND LIST (Aqueous)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Phenanthrene	40-140	30	70-130	20	40-140	20
Phenol	12-110	30	20-160	20	30-130	20
Pyrene	26-127	30	70-130	20	40-140	20
Pyridine	10-66	30			40-140	20
2-Fluorophenol	*21-120*		*15-110*		*15-110*	
Phenol-d6	*10-120*		*15-110*		*15-110*	
Nitrobenzene-d5	*23-120*		*30-130*		*30-130*	
2-Fluorobiphenyl	*15-120*		*30-130*		*30-130*	
2,4,6-Tribromophenol	*10-120*		*15-110*		*15-110*	
4-Terphenyl-d14	*41-149*		*30-130*		*30-130*	

TABLE 3B

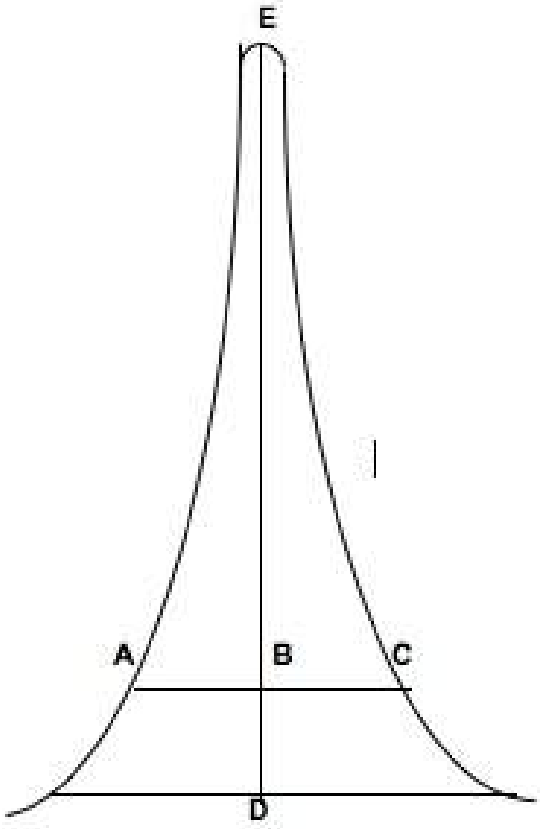
ACCEPTABLE SOIL QC LIMITS

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)		CT TARGET COMPOUND LIST (Soil)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
1,2,4,5-Tetrachlorobenzene	40-117	50	70-130	30	40-140	30
1,2,4-Trichlorobenzene	38-107	50	70-130	30	40-140	30
1,2-Dichlorobenzene	40-140	50	70-130	30		
1,3-Dichlorobenzene	40-140	50	70-130	30		
1,3-Dinitrobenzene	40-140	50				
1,4-Dichlorobenzene	28-104	50	70-130	30		
1-Methylnaphthalene	26-130	50				
2,3,4,6-Tetrachlorophenol	40-140	50	70-130	30		
2,4,5-Trichlorophenol	30-130	50	70-130	30	30-130	30
2,4,6-Trichlorophenol	30-130	50	70-130	30	30-130	30
2,4-Dichlorophenol	30-130	50	70-130	30	30-130	30
2,4-Dimethylphenol	30-130	50	70-130	30	30-130	30
2,4-Dinitrophenol	4-130	50	20-160	30	30-130	30
2,4-Dinitrotoluene	28-89	50	70-130	30	40-140	30
2,6-Dinitrotoluene	40-140	50	70-130	30	40-140	30
2-Chloroaniline	30-130	50				
2-Chloronaphthalene	40-140	50	70-130	30	40-140	30
2-Chlorophenol	25-102	50	70-130	30	30-130	30
2-Methylnaphthalene	40-140	50	70-130	30	40-140	30
2-Methylphenol	30-130.	50	70-130	30	30-130	30
2-Nitroaniline	47-134	50	70-130	30	40-140	30
2-Nitrophenol	30-130	50	70-130	30	30-130	30
3,3'-Dichlorobenzidine	40-140	50	70-130	30	40-140	30
3,3'-Dimethylbenzidine	15-115	50				
3-Methylphenol/4-Methylphenol	30-130	50	20-160	30	30-130	30
3-Nitroaniline	26-129	50	70-130	30	40-140	30
4,6-Dinitro-o-cresol	10-130	50	70-130	30	30-130	30
4-Bromophenyl phenyl ether	40-140	50	70-130	30	40-140	30
4-Chloroaniline	40-140	50	20-160	30	40-140	30
4-Chlorophenyl phenyl ether	40-140	50	70-130	30	40-140	30
4-Nitroaniline	41-125	50	70-130	30	40-140	30
4-Nitrophenol	11-114	50	20-160	30	30-130	30
Acenaphthene	31-137	50	70-130	30	40-140	30

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)		CT TARGET COMPOUND LIST (Soil)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Acenaphthylene	40-140	50	70-130	30	40-140	30
Acetophenone	14-144	50	70-130	30	40-140	30
Aniline	40-140	50	20-160	30	40-140	30
Anthracene	40-140	50	70-130	30	40-140	30
Atrazine	40-140	50	70-130	30		
Azobenzene	40-140	50	70-130	30		
Benzaldehyde	40-140	50	20-160	30		
Benzidine	10-66	50	20-160	30		
Benzo(a)anthracene	40-140	50	70-130	30	40-140	30
Benzo(a)pyrene	40-140	50	70-130	30	40-140	30
Benzo(b)fluoranthene	40-140	50	70-130	30	40-140	30
Benzo(e)pyrene	40-140	50				
Benzo(ghi)perylene	40-140	50	70-130	30	40-140	30
Benzo(k)fluoranthene	40-140	50	70-130	30	40-140	30
Benzoic Acid	10-110	50	20-160	30		
Benzyl Alcohol	40-140	50	20-160	30		
Biphenyl	37-127	50	70-130	30		
Bis(2-chloroethoxy)methane	40-117	50	70-130	30	40-140	30
Bis(2-chloroethyl)ether	40-140	50	70-130	30	40-140	30
Bis(2-chloroisopropyl)ether	40-140	50	70-130	30	40-140	30
Bis(2-Ethylhexyl)phthalate	40-140	50	70-130	30	40-140	30
Butyl benzyl phthalate	40-140	50	70-130	30	40-140	30
Caprolactam	15-130	50	20-160	30		
Carbazole	54-128	50	70-130	30	40-140	30
Chrysene	40-140	50	70-130	30	40-140	30
Dibenzo(a,h)anthracene	40-140	50	70-130	30	40-140	30
Dibenzofuran	40-140	50	70-130	30	40-140	30
Diethyl phthalate	40-140	50	70-130	30	40-140	30
Dimethyl phthalate	40-140	50	70-130	30	40-140	30
Di-n-butylphthalate	40-140	50	70-130	30	40-140	30
Di-n-octylphthalate	40-140	50	70-130	30	40-140	30
Diphenamid	40-140	50				
Fluoranthene	40-140	50	70-130	30	40-140	30
Fluorene	40-140	50	70-130	30	40-140	30
Hexachlorobenzene	40-140	50	70-130	30	40-140	30
Hexachlorobutadiene	40-140	50	70-130	30	40-140	30
Hexachlorocyclopentadiene	40-140	50	20-160	30	40-140	30
Hexachloroethane	40-140	50	20-160	30	40-140	30
Indeno(1,2,3-cd)Pyrene	40-140	50	70-130	30	40-140	30
Isophorone	40-140	50	70-130	30	40-140	30

	STANDARD TARGET COMPOUND LIST (Soil)		NEW JERSEY TARGET COMPOUND LIST (Soil)		CT TARGET COMPOUND LIST (Soil)	
Analyte	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**	**Acceptance Criteria**	**Duplicate RPD**
Naphthalene	40-140	50	70-130	30	40-140	30
Nitrobenzene	40-140	50	70-130	30	40-140	30
NitrosoDiPhenylAmine(NDPA)/ Diphenylamine (DPA)	36-157	50	70-130	30	40-140	30
n-Nitrosodimethylamine	22-100	50	20-160	30		
n-Nitrosodi-n-propylamine	32-121	50	70-130	30	40-140	30
Parathion, ethyl	40-140	50	20-160	30		
P-Chloro-M-Cresol	26-103	50	70-130	30	30-130	30
Pentachloronitrobenzene	42-153	50			40-140	30
Pentachlorophenol	17-109	50	20-160	30	30-130	30
Phenanthrene	40-140	50	70-130	30	40-140	30
Phenol	26-90	50	20-160	30	30-130	30
Pyrene	35-142	50	70-130	30	40-140	30
Pyridine	10-93	50	20-160	30	40-140	30
Thionazin	40-140	50				
2-Fluorophenol	*25-120*		*30-130*		*30-130*	
Phenol-d6	*10-120*		*30-130*		*30-130*	
Nitrobenzene-d5	*23-120*		*30-130*		*30-130*	
2-Fluorobiphenyl	*30-120*		*30-130*		*30-130*	
2,4,6-Tribromophenol	*10-136*		*30-130*		*30-130*	
4-Terphenyl-d14	*18-120*		*30-130*		*30-130*	

TABLE 4 – Tailing Factor Calculation



Tailing Factor = $\frac{BC}{AB}$

Example calculation:

Peak Height = DE = 100mm
10% Peak Height = BD = 10mm
Peak Width at 10% Peak Height = AC = 23mm

AB = 11mm
BC = 12mm

Therefore: Tailing Factor = $\frac{12}{11}$ = 1.1

Tailing factor for benzidine < 2.0

Tailing factor for pentachlorophenol <2.0

TABLE 5

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
Acenaphthene	154	153, 152
Acenaphthylene	152	151, 153
Acetophenone	105	71, 51, 120
Aniline	93	66, 65
Anthracene	178	176, 179
Atrazine	200	202, 215
Azobenzene	77	182, 105
Benzaldehyde	105	77
Benzidine	184	92, 185
Benzo(a)anthracene	228	229, 226
Benzo(a)pyrene	252	253, 125
Benzo(b)fluoranthene	252	253, 125
Benzo(g,h,i)perylene	276	138, 277
Benzo(k)fluoranthene	252	253, 125
Benzoic acid	105	122, 77
Benzyl alcohol	79	77,108
Biphenyl	154	153,152
Bis (2-chloroethoxy) methane	93	95, 123
Bis (2-chloroethyl) ether	93	63, 95
Bis (2-chloroisopropyl) ether	45	77, 121
Bis (2-ethylhexyl) phthalate	149	167, 279
4-Bromophenyl phenyl ether	248	250, 141
Butyl Benzyl phthalate	149	91, 206
Caprolactam	55	85, 113
Carbazole	167	168, 166
4-Chloro-3-methylphenol	107	144, 142
2-Chloroaniline	127	129, 65
3-Chloroaniline	65	127, 129
4-Chloroaniline	65	127,129
2-Chloronaphthalene	162	127, 164
4-Chlorophenyl phenyl ether	204	206, 141
2-Chlorophenol	128	64,130
Chrysene	228	226, 229
Dibenzo(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
1,2-Dichlorobenzene	146	148, 111

1,3-Dichlorobenzene	**146**	**148, 111**
1,4-Dichlorobenzene	**146**	**148, 111**
3,3'-Dichlorobenzidine	**252**	**254, 126**
2,4-Dichlorophenol	**162**	**164, 98**
Diethyl phthalate	**149**	**177, 150**

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
3,3-Dimethylbenzidine	**212**	**211, 213**
Dimethyl phthalate	**163**	**194, 164**
2,4-Dimethylphenol	**107**	**121,122**
Di-n-butyl phthalate	**149**	**150, 104**
Di-n-octyl phthalate	**149**	**167, 43**
4,6-Dinitro-2-methylphenol	**198**	**51, 105**
O-Toluidine	**106**	**107, 77**
2-Ethylaniline	**106**	**121, 77**
2,4-Dimethylaniline	**121**	**120, 106**
2,3-Dimethylaniline	**106**	**121, 120**
3,4- Dimethylaniline	**121**	**120,106**
2,4,5-Trimethylaniline	**120**	**135, 134**
4-Chlorotoluidine	**106**	**141, 140**
2-Napthylamine	**143**	**115, 116**
2,4-Dinitrophenol	**184**	**107,91**
2,4-Dinitrotoluene	**165**	**63, 89**
2,6-Dinitrotoluene	**165**	**63, 89**
Diphenamide	**167**	**72, 165**
1,4-Dioxane	**88**	**58,43**
Ethyl parathion	**109**	**97, 291**
Fluoranthene	**202**	**101, 203**
Fluorene	**166**	**165, 167**
Hexachlorobenzene	**284**	**142, 249**
Hexachlorobutadiene	**225**	**223, 227**
Hexachlorocyclopentadiene	**237**	**235, 272**
Hexachloroethane	**117**	**201, 199**
Indeno(1,2,3-cd)pyrene	**276**	**138, 227**
Isophorone	**82**	**95, 138**
1-Methylnaphthalene	**115**	**141, 142**
2-Methylnaphthalene	**142**	**141**
2-Methylphenol	**108**	**107,90**
3/4-Methylphenol	**108**	**107,90**

Naphthalene	**128**	**129, 127**
2-Nitroaniline	**65**	**92, 138**
3-Nitroaniline	**138**	**92,65**
4-Nitroaniline	**138**	**65, 108, 92, 80, 39**
Nitrobenzene	**77**	**123, 65**

TABLE 5 (continued)

CHARACTERISTIC IONS FOR SEMIVOLATILE COMPOUNDS

Compound	Primary Ion	Secondary Ion(s)
2-Nitrophenol	**139**	**109, 65**
4-Nitrophenol	**65**	**109, 139**
n-Nitrosodimethylamine	**74**	**42,44**
n-Nitrosodi-n-butylamine	**84**	**57, 41, 116, 158**
n-Nitrosodi-n-propylamine	**70**	**42, 101, 130**
n-Nitrosodiphenylamine/Diphenylamine	**169**	**168, 167**
Pentachlorobenzene	**250**	**252, 108, 248, 215, 254**
Pentachloronitrobenzene	**237**	**142, 214, 249, 295, 265**
Pentachlorophenol	**266**	**264, 268**
Phenanthrene	**178**	**179, 176**
Phenol	**94**	**65, 66**
Pyrene	**202**	**200, 203**
Pyridine	**79**	**52**
1,2,4,5-Tetrachlorobenzene	**216**	**214, 179, 108, 143, 218**
2,3,4,6-Tetrachlorophenol	**232**	**131, 230, 166, 234, 168**
m-Toluidine	**106**	**107, 79**
1,2,4-Trichlorobenzene	**180**	**182, 145**
2,4,5-Trichlorophenol	**196**	**200,198**
2,4,6-Trichlorophenol	**196**	**198, 200**
Acenaphthene-d_{10} (IS)	**164**	**162, 160**
Chrysene-d_{12} (IS)	**240**	**120, 236**
1,4-Dichlorobenzene-d_4 (IS)	**152**	**150, 115**
Naphthalene-d_8 (IS)	**136**	**68**
Perylene-d_{12} (IS)	**264**	**260, 265**
Phenanthrene-d_{10} (IS)	**188**	**94, 80**
2-Fluorobiphenyl (Surrogate)	**172**	**171**
2-Fluoroophenol (Surrogate)	**112**	**64**
Nitrobenzene-d_5 (Surrogate)	**82**	**128, 54**
Phenol-d_6 (Surrogate)	**99**	**42, 71**
Terphenyl-d_{14} (Surrogate)	**244**	**122, 212**
2,4,6-Tribromophenol (Surrogate)	**330**	**62,141**

TABLE 6

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
Acenaphthene	2	133.34
Acenaphthylene	2	133.34
Acetophenone	5	333.34
Aniline	2	133.34
Anthracene	2	133.34
Atrazine	10	666.67
Azobenzene	2	500
Benzaldehyde	5	333.34
Benzidine	20	1333.34
Benzo(a)anthracene	2	133.34
Benzo(b)fluoranthene	2	133.34
Benzo(k)fluoranthene	2	133.34
Benzo(ghi)perylene	2	133.34
Benzo(a)pyrene	2	133.34
Benzoic acid	50.0	3333.34
Benzyl alcohol	2	133.34
Biphenyl	2	366.67
Bis(2-chloroethyl)ether	2	133.34
Bis(2-chloroisopropyl)ether	2	133.34
Bis(2-chloroethoxy)methane	5.0	333.34
Bis(2-ethylhexyl)phthalate	3	200
4-Bromophenyl phenyl ether	2	133.34
Butyl benzyl phthalate	5.0	333.34
Caprolactam	10	666.67
Carbazole	2	166.67
2-Chloroaniline	2	na
3-Chloroaniline	10	na
4-Chloroaniline	5	333.34
p-Chloro-m-cresol (4-chloro-3-cresol)	2	133.34
2-Chloronaphthalene	2	133.34
2-Chlorophenol	2	133.34
4-Chlorophenyl phenyl ether	2	133.34
Chrysene	2	133.34
m/p-Methylphenol (3/4-methylphenol)	5.0	333.34
o-Methylphenol (2-methylphenol)	5.0	333.34

Dibenzo(a,h)anthracene	2	133.34
Dibenzofuran	2	133.34
Di-n-butylphthalate	5.0	333.34
1,2-Dichlorobenzene	2	133.34

TABLE 6 (continued)

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
1,3-Dichlorobenzene	2	133.34
1,3-Dinitrobenzene	2	N/A
1,4-Dichlorobenzene	2	133.34
3,3-Dichlorobenzidine	5	333.34
2,4-Dichlorophenol	5	333.34
O-Toluidine	2	N/A
2-Ethylaniline	2	N/A
2,4-Dimethylaniline	2	N/A
2,3-Dimethylaniline	2	N/A
3,4-Dimetylaniline	2	N/A
2,4,5-Trimethylaniline	2	N/A
4-Chlorotoluidine	2	N/A
2-Napthylamine	2	N/A
2,6-Dichlorophenol	10.0	666.67
Diethyl phthalate	5.0	333.34
3,3-Dimethylbenzidine	4	500
2,4-Dimethylphenol	5	333.34
Dimethyl phthalate	5.0	333.34
4,6-Dinitro-o-cresol	10	666.67
2,4-Dinitrophenol	20	1333.4
2,4-Dinitrotoluene	5.0	333.34
2,6-Dinitrotoluene	5.0	333.34
Di-n-octylphthalate	5.0	333.34
Diphenamide	5	N/A
1,4-Dioxane	5	166.67
Ethyl Parathion	N/A	166.67
Fluoranthene	2	133.34
Fluorene	2	133.34
Hexachlorobenzene	2	133.34
Hexachlorobutadiene	2	133.34
Hexachlorocyclopentadiene	20	1333.34
Hexachloroethane	2	133.34
Indeno(1,2,3-cd)pyrene	2	133.34
Isophorone	5.0	333.34

1-Methylnaphthalene	2	166.67
2-Methylnaphthalene	2	133.34
Naphthalene	2	133.34
2-Nitroaniline	5.0	333.34

TABLE 6 (continued)

REPORTED DETECTION LIMITS FOR SEMIVOLATILE ORGANIC COMPOUNDS*

Analyte	RDL (µg/L)	RDL (µg/Kg)
3-Nitroaniline	5.0	333.34
4-Nitroaniline	5.0	333.34
Nitrobenzene	2	133.34
2-Nitrophenol	10.0	666.67
4-Nitrophenol	10.0	666.67
Nitrosodi-n-butylamine	10.0	666.67
n-Nitrosodimethylamine	2	133.34
n-Nitrosodiphenylamine/Diphenylamine	2	133.34
Nitrosodipiperidine	20.0	2000
n-Nitrosodi-n-propylamine	5.0	333.34
Pentachlorobenzene	20.0	1333.34
Pentachloronitrobenzene	10.0	150
Pentachlorophenol	10.0	666.67
Phenanthrene	2	133.34
Phenol	5.0	333.34
Pyrene	2	133.34
Piridine	5	666.67
1,2,4,5-Tetrachlorobenzene	10	666.67
1,2,4-Trichlorobenzene	5.0	333.34
2,4,5-Trichlorophenol	5.0	333.34
2,4,6-Trichlorophenol	5.0	333.34
2,3,4,6-Tetrachlorophenol	5.0	166.66
m-Toluidine	5	300

*** Note**: Reporting Limits are based on standard 8270 reporting list. RLs may vary for other reporting lists.

Table 7
Semivolatile Internal Standards with Corresponding
Target Compounds and Surrogates Assigned for Quantitation

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
O-Toluidine	2-Ethylaniline	2-Naphthylamine	3,3-Dimethylbenzidine	3,3'-Dichlorobenzidine	Benzo(g,h,i)perylene
1,2,4-Trichlorobenzene	2,4-Dimethylaniline	2,3,4,6-Tetrachlorophenol	Anthracene	Benzo(a)Anthracene	Dibenzo(a,h)anthracene
1,2-Dichlorobenzene	3,4-Dimethylaniline	2,3,5,6-Tetrachlorophenol	Benzidine	Benzo(a)pyrene	Indeno(1,2,3-cd)pyrene
1,3-Dichlorobenzene	2,3-Dimethylaniline	2,4,6-Tribromophenol, surr	Benzyl butyl phthalate	Benzo(b)fluoranthene	
1,4-Dichlorobenezne	2,4,5-Trimethylaniline	2,4-Dinitrophenol	Carbazole	Benzo(k)fluoranthene	
2,4-Dichlorophenol	4-Chlorotoludine	2,4-Dinitrotoluene	Di-n-Butylphthalate	Bis(2-ethylhexyl) phthalate	
2,4-Dimethylphenol	1,2,4,5-Tetrachlorobenzene	3-Nitroaniline	Diphenamid	Chrysene	
2-Chloroaniline	1,2-Dichlorobenzene	4,6-Dinitro-2-methylphenol	Fluoranthene	Di-n-octylphthalate	
2-Chlorophenol	1,3-Dichlorobenzene	4-Bromophenyl-phenyl ether	n-Octadecane		
2-Fluorophenol, surr	1,4-Diclorobenzene	4-Chlorophenyl-phenyl ether	Parathion		
2-Methylphenol	1-chloror-2-nitrobenzene	4-Nitroaniline	Phenanthrene		
2-Nitrophenol	1-Methylnapthalene	4-Nitrophenol	Pyrene		
3-Methylphenol / 4-Methylphenol	2,4,5-Trichlorphenol	Acenaphthene	Terphenyl-d14, surr		
Acetophenone	2,4,6-Trichlorophenol	Atrazine			
Aniline	2,6-Dichlorophenol	Azobenzene			
Benzaldehyde	2,6-Dinitrotoluene	Dibenzofuran			
Benzyl Alcohol	2-Chloronaphthalene	Dichloran			
Bis(2-chloroethoxy)methane	2-Fluorobiphenyl, surr	Diethyl phthalate			
Bis(2-chloroethyl)ether	2-Methylnaphthalene	Fluorene			

Table 7 (cont.)
Semivolatile Internal Standards with Corresponding
Target Compounds and Surrogates Assigned for Quantitation

1,4-dichlorobenzene-d4	Naphthalene-d8	Acenaphthene-d10	Phenanthrene-d10	Chrysene-d12	Perylene-d12
bis(2-Chloroisopropyl)ether	2-Nitroaniline	Hexachlorobenzene			
Hexachloroethane	3-Choloroaniline	NDPA/DPA			
Isophorone	4-Chloro-3-Methylphenol	Pentachloronitrobenzene			
m-Toluidine	4-Chloroaniline	Pentachlorophenol			
n-Decane	Acenaphthylene				
Nitrobenzene	a-Terpineol				
Nitrobenzene-d5, surr	Benzoic Acid				
N-Nitrosodimethylamine	Biphenyl				
N-Nitrosodi-n-propylamine	Caprolactam				
Phenol	Dimethyl Phthalate				
Phenol-d6, surr	Hexachlorobutadiene				
Pyridine 1,4-Dioxane	Hexachlorocyclopentadiene				
Phenol-d6, surr	Naphthalene				

Table 8

Recommended Minimum Response Factor Criteria from Initial and Continuing Calibration Verification Using the Suggested Ions in Table 5

Analyte	MRF
Benzaldehyde	0.010
Phenol	0.800
Bis(2-chloroethyl)ether	0.700
2-Chlorophenol	0.800
2-Methylphenol	0.700
2,2'-Oxybis-(1-chloropropane)	0.010
Acetophenone	0.010
4-Methylphenol	0.600
N-Nitroso-di-n-propylamine	0.500
Hexachloroethane	0.300
Nitrobenzene	0.200
Isophorone	0.400
2-Nitrophenol	0.100
2,4-Dimethylphenol	0.200
Bis(2-chloroethoxy)methane	0.300
2,4-Dichlorophenol	0.200
Naphthalene	0.700
4-Chloroaniline	0.010
Hexachlorobutadiene	0.010
Caprolactam	0.010
4-Chloro-3-methylphenol	0.200
2-Methylnaphthalene	0.400
Hexachlorocyclopentadiene	0.050
2,4,6-Trichlorophenol	0.200
2,4,5-Trichlorophenol	0.200
1,1'-Biphenyl	0.010
2-Chloronaphthalene	0.800
2-Nitroaniline	0.010
Dimethyl phthalate	0.010
2,6-Dinitrotoluene	0.200
Acenaphthylene	0.900
3-Nitroaniline	0.010
Acenaphthene	0.900
2,4-Dinitrophenol	0.010
4-Nitrophenol	0.010
Dibenzofuran	0.800
2,4-Dinitrotoluene	0.200
Diethyl phthalate	0.010
1,2,4,5-Tetrachlorobenzene	0.010

Table 8 (cont.)

Recommended Minimum Response Factor Criteria from Initial and Continuing Calibration Verification Using the Suggested Ions in Table 5

Analyte	MRF
4-Chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroaniline	0.010
4,6-Dinitro-2-methylphenol	0.010
4-Bromophenyl-phenyl ether	0.100
N-Nitrosodiphenylamine	0.010
Hexachlorobenzene	0.100
Atrazine	0.010
Pentachlorophenol	0.050
Phenanthrene	0.700
Anthracene	0.700
Carbazole	0.010
Di-n-butyl phthalate	0.010
Fluoranthene	0.600
Pyrene	0.600
Butyl benzyl phthalate	0.010
3,3'-Dichlorobenzidine	0.010
Benzo(a)anthracene	0.800
Chrysene	0.700
Bis-(2-ethylhexyl)phthalate	0.010
Di-n-octyl phthalate	0.010
Benzo(b)fluoranthene	0.700
Benzo(k)fluoranthene	0.700
Benzo(a)pyrene	0.700
Indeno(1,2,3-cd)pyrene	0.500
Dibenz(a,h)anthracene	0.400
Benzo(g,h,i)perylene	0.500
2,3,4,6-Tetrachlorophenol	0.010

Table 9
Difficult analytes

Aniline

Benzaldehyde
Benzidine
Benzoic acid
Benzyl alcohol

Caprolactam
4-Chloroaniline
4-chloro-3-methylphenol (p-chloro-m-cresol)

3,3-Dimethylbenzidine
1,4-Dioxane
Dimethylphthalate
2,4 Dinitrophenol
4,6-dinitro-2-methylphenol (4,6-dinitro-o-cresol)

Hexachlorocyclopentadiene
Hexachloroethane

2-Methylphenol
3-Methylphenol/4-Methylphenol

2-nitroaniline
3-nitroaniline
4-nitroaniline
4-Nitrophenol
Nitrosodiphenylamine and diphenylamine (NDPA/DPA)
n-Nitrosodimethylamine

Parathion
Pentachloronitrobenzene
Pentachlorophenol
Phenol
Pyridine

Specific Conductance and Salinity

Reference Methods.: **SM 2510 B, SM 2520B (Salinity),** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

EPA 120.1, Methods for Chemical Analysis of Water and Waste, EPA-600/4-79-020, U.S. Environmental Protection agency, 1982.

SW-846 9050A, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1996.

1. Scope and Application

Matrices: This method is applicable to aqueous solutions. Soils samples can be analyzed using water based extractions for preparation prior to analysis. (Sec 2.1)

Definitions: See Alpha Quality Manual Appendix A

Conductance, G, is defined as the reciprocal of resistance, R:

$$G = 1 / R$$

Where the unit of R is ohm and G is ohm^{-1} (sometimes written mho). Conductance of a solution is measured between two spatially fixed and chemically inert electrodes. To avoid polarization at the electrode surfaces, the conductance measurement is made with an alternating current signal. The conductance of a solution, G, is directly proportional to the electrode surface area, A, cm^2, and inversely proportional to the distance between the electrodes, L, cm. The constant of proportionality, k, such that:

$$G = k (A / L)$$

is called “conductivity” (preferred to “specific conductance”). It is a characteristic property of the solution between the electrodes. The units of k are 1/ohm-cm or mho per centimeter. Conductivity is customarily reported in micromhos per centimeter (µmho/cm).

Distilled water produced in a laboratory generally has a conductivity in the range of 0.5 to 3µmhos/cm. The conductivity increases shortly after exposure to both air and the water container.

The conductivity of potable waters in the United States ranges generally from 50 to 1500µmhos/cm. The conductivity of domestic wastewaters may be near that of the local water supply, although some industrial wastes have conductivities above 10,000µmhos/cm. Conductivity instruments are used in pipeline, channels, flowing streams, and lakes and can be incorporated in multiple-parameter monitoring stations using recorders.

Laboratory conductivity measurements are used to:

- Establish the degree of mineralization to assess the effect of the total concentration of ions on chemical equilibria, physiological effect on plants or animals, corrosion rates, etc.
- Assess the degree of mineralization of distilled and deionized water.
- Evaluate variations in dissolved mineral concentration of raw water or wastewater. Minor seasonal variations found in reservoir waters contrast sharply with the daily fluctuations in some polluted river waters. Wastewater containing significant trade wastes also may show a considerable daily variation.
- Estimate sample size to be used for common chemical determinations and to check results of a chemical analysis.

- Determine amount of ionic reagent needed in certain precipitation and neutralization reactions, the end point being denoted by a change in slope of the curve resulting from plotting conductivity against buret readings.
- Estimate total dissolved solids (mg/L) in a sample, multiplying conductivity (in micromhos per centimeter) by an empirical factor. This factor may vary from 0.55 to 0.9, depending on the soluble components of the water and on the temperature of measurement. Relatively high factors may be required for saline or boiler waters, whereas lower factors may apply where considerable hydroxide or free acid is present. Even though sample evaporation results in the change of bicarbonate to carbonate, the empirical factor is derived for a comparatively constant water supply by dividing dissolved solids by conductivity.
- Approximate the milliequivalents per liter of either cations or anions in some waters by multiplying conductivity in units of micromhos per centimeter by 0.01.

Salinity is an important unit less property of industrial and natural waters. It was originally conceived as a measure of the mass of dissolved salts in a given mass of solution. The experimental determination of the salt content by drying and weighing presents some difficulties due to the loss of some components.

To determine salinity, one normally uses indirect methods involving the measurement of a physical property such as conductivity. From an empirical relationship of salinity and the physical property determined for a standard solution it is possible to calculate salinity. Because of its high sensitivity and ease of measurement, the conductivity method is most commonly used to determine salinity

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Conductivity Instrument and in the interpretation of data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Conductivity is a measure of the ability of an aqueous solution to carry an electric current. This ability depends on the presence of ions; on their total concentration, mobility, and valence; and on the temperature of measurement. Solutions of most inorganic compounds are relatively good conductors. Conversely, molecules of organic compounds that do not dissociate in aqueous solution conduct a current very poorly, if at all.

The Corning Model 441 conductivity meter currently in use has had the traditional platonized conductivity cell replaced with sensors that operate on a 4-ring principle, where alternating voltage is applied to the 2 outer rings. This produces a current in the solution. The size of the current is determined by the quantity of ions in the solution.

2.1 Method Modifications from Reference

A Method Blank is not analyzed.

Analysis of soil samples is performed using water based extraction with ratio 1:10 soil to water.

3. Reporting Limits

The reporting limit is 10 µmho/cm for both aqueous and soil matrices. Salinity is reported in SU with RL of 2 SU

4. Interferences

4.1 Most problems in obtaining good data with conductivity-monitoring equipment are related to electrode fouling and to inadequate sample circulation.

4.2 Conductivities greater than 10,000 to 50,000µmho/cm or less than about 10µmho/cm may be difficult to measure with usual measurement electronics.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in either glass or plastic containers, preferably with no headspace.

6.2 Sample Preservation

None.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Refrigerate at 4±2ºC and analyze within 24 hours of sample collection. If analysis cannot be completed within 24 hours of collection then the sample must be filtered through a 0.45micron filter and stored at 4±2ºC and analysis completed within 28 days of collection. The filter apparatus should be rinsed with DI followed by a pre-rinse with the sample before use.

7. Equipment and Supplies

7.1 Conductivity Meter: Corning Model 441, SN 001262. With auto temperature compensation from 0 – 40 °C or equivalent.

7.2 Conductivity Probe: Corning, PN 476501.

7.3 Analytical balances

7.4 Plastic cups: 150 ml

7.5 Graduated cylinder: 100 ml

8. Reagents and Standards

8.1 1413 µmhos / cm conductivity standard: Dissolve 744.7 mg of anhydrous KCl in Deionized Water and bring up to 1000mL in a volumetric flask. Alternately, Oakton, PN 00653-18, a commercially prepared standard may be used, with a certificate of analysis kept on file. Store at room temperature. Expires upon manufacturer's specified date.

8.2 84 µmhos / cm conductivity standard: Dissolve 40.38 mg of anhydrous KCl in Deionized Water and bring up to 1000mL in a volumetric flask. Alternately, Oakton, PN 00653-16, a commercially prepared standard may be used, with a certificate of analysis kept on file. Store at room temperature. Expires upon manufacturer's specified date.

8.3 Alternative conductivity standards: Standards with alternative concentrations (1500 umhos/cm, 10,049 umhos/cm , 996 umhos/com) may be used for CCV's . All standards are commercially prepared, a certificate of analysis kept on file. Standards are stored at room temperature. Expires upon manufacturer's specified date.

8.4 DI water

9. Quality Control

9.1 Blank(s)

Not applicable.

9.2 Laboratory Control Sample (LCS)

The calibration must be verified with a third party standard. When possible, choose a standard in the same range as the samples being analyzed. The LCS is analyzed at the beginning of a batch of 20 samples or less. % Recovery must be within ± 1% of the true value. If % recovery falls outside of acceptance criteria, the meter is re-calibrated and LCS is re-analyzed.

9.3 Initial Calibration Verification (ICV)

See LCS Section 9.2.

9.4 Continuing Calibration Verification (CCV)

The calibration must be verified with a third party standard. The CCV is analyzed at the end of a batch of 20 samples or less. % Recovery must be within ± 1% of the true value. If % recovery falls outside of acceptance criteria, the standard may be analyzed again. If failure continues, all samples analyzed since the last acceptable calibration verification must be reanalyzed.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

Method 120.1: Analyze one duplicate sample per batch of 20 samples or less. Method 9050A: Analyze one duplicate sample per batch of 10 samples or less. % RPD must fall within acceptance criteria generated based on in-house performance data. If results fall outside of acceptance criteria, the data is evaluated and a narrative is included in the report.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Calibrate conductivity sensor
- Analyze an appropriate conductivity standard, LCS
- Rinse conductivity cell with sample or DI
- Measure conductivity of samples and duplicate
- Analyze an appropriate conductivity standard, CCV

10. Procedure

10.1 Equipment Set-up

Press “On/Off” button to turn instrument on.

10.2 Initial Calibration

10.2.1 Daily Calibration of Conductivity Sensor

NOTE: The following procedure satisfies the requirement to determine the cell constant, as outlined in SM2510B.4.a and section 7.1 in 9050A

2 Point Calibration :

- Hold the sensor in air and press “CAL”.
- Place the tip of the sensor in the conductivity standard (either 84 or 1413 µmhos/cm) and press “CAL” .

  NOTE: For greater accuracy 1) calibrate using a standard close in value to the expected results of the samples and 2) have the standards at the reference temperature of 25 °C.
- The meter will automatically endpoint when the reading is stable.
- Rinse the sensor with deionized water.
- The analyst does not need to set the reference temperature.

10.3 Equipment Operation and Sample Processing

10.3.1 Rinse the conductivity cell with one or more portions of sample or DI water.

10.3.2 For soil samples: in plastic cup measure 10 g of homogenized soil material, add 100 ml of DI and stirring bar. Stir for 30 minutes, let set for 30 minutes, pour off 25 mL of extract and then proceed to section 10.3.3.

Note: Alternative weight may be used for soil preparation; in this case amount of DI water for extraction has to be adjusted to ensure 1:10 ratio for soil: H2O

For water samples pour off 25 mL of sample and proceed to section 10.3.3

10.3.3 Place the sensor in the sample and press “READ” to start the sample measurement. The decimal point will be flashing whenever the sensor is reading. The flashing will stop when the reading is stable and the instrument will “beep”. In the Laboratory notebook record the reading with the correct units as displayed on the instrument.

10.3.4 When measuring, make sure the solution is above the cell chamber slot.

10.3.5 Make sure the cell chamber is free of bubbles when measuring. To reduce air bubbles, immerse the sensor at an angle and then raise to the vertical position.

10.3.6 For greater accuracy, have the samples at the reference temperature of 25 °C.

10.4 Continuing Calibration

The calibration must be verified with a third party standard. The CCV is analyzed at the end of a batch of 20 samples or less. % Recovery must be within ± 20% of the true value. If % recovery falls outside of acceptance criteria, the standard may be analyzed again. If failure continues, all samples analyzed since the last acceptable calibration verification must be reanalyzed.

10.5 Preventative Maintenance

10.5.1 The conductivity meter is calibrated annually by an instrument service company. Certificates are kept on file.

10.5.2 Sensor maintenance:

10.5.2.1 To prevent static damage to the Model 441, avoid touching the cell chamber area of the sensor. ALWAYS disconnect the sensor from the Model 441 meter before cleaning.

10.5.2.2 Solids built up inside the cell chamber can be carefully removed with a cotton wool bud soaked in a detergent solution, then rinsing the sensor with DI water.

10.5.2.3 Do not use organic solvents to clean the sensor.

11. Data Evaluation, Calculations and Reporting

The temperature coefficient of most waters is approximately the same as that of the standard KCl solution. The more the temperature deviates from 25.0ºC, the greater the uncertainty in applying the temperature correction. Report all conductivities at 25 °C.

Results are reported directly from the Corning Model 441 instrument.

Conductivity results are used to calculate salinity using Practical Salinity Scale. This scale was developed relative to a KCl solution. A seawater with a conductivity, C, at 15°C equal to that of a KCl solution containing a mass of 32.4356 g in a mass of 1 kg of solution is defined as having a practical salinity of 35. This value was determined as an average of three independent laboratory studies. The salinity dependence of the conductivity ratio, Rt, as a function of temperature (t °C, International Practical Temperature Scale 1968) of a given sample to a standard S 35 seawater is used to determine the salinity.

$$S = a_0 + a_1 R_t^{1/2} + a_2 R_t + a_3 R_t^{3/2} + a_4 R_t^2 + a_5 R_t^{5/2} + \Delta S$$

where ΔS is given by

$$\Delta S = \left[\frac{t - 15}{1 + 0.0162\,(t - 15)}\right](b_0 + b_1 R_t^{1/2} + b_2 R_t + b_3 R_t^{3/2} + b_4 R_t^2 + b_5 R_t^{5/2})$$

and:

$a_0 = 0.0080$	$b_0 = 0.0005$
$a_1 = -0.1692$	$b_1 = -0.0056$
$a_2 = 25.3851$	$b_2 = -0.0066$
$a_3 = 14.0941$	$b_3 = -0.0375$
$a_4 = -7.0261$	$b_4 = 0.0636$
$a_5 = 2.7081$	$b_5 = -0.0144$

valid from $S = 2$ to 42, where:

$$R_t = \frac{C\,(\text{sample at } t)}{C\,(\text{KCl solution at } t)}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. Analysis must be repeated. If duplicate results fall outside of acceptance criteria, the data is evaluated and a narrative is included in the report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

Sulfate, Turbidimetric Method

Method References: **SM 4500SO_4-E,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

EPA 9038, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1997.

HACH Water Analysis Handbook, Sulfa Ver 4 Method: Copyright 1989

1. Scope and Application

Matrices: This method is applicable to water and wastewater samples. The samples should be free of sediment and color. However, small amounts of color and/or sediment may be corrected for, see interference section.

Definitions: See Alpha Laboratories Quality Manual Appendix A.

Sulfate is widely distributed in nature and may be present in natural waters in concentrations ranging from a few to several thousand milligrams per liter. Mine drainage wastes may contribute large amounts of sulfate through pyrite oxidation. Sodium and magnesium sulfate exert a cathartic action.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

25mL of sample is mixed with 1 Sulfa Ver 4 Reagent powder pillow or ampule to react with sulfate in the sample. The sample absorbance is read on a Spectrophotometer at 420nm and calculated against a daily-made calibration curve.

2.1 Method Modifications from Reference

A HACH sulfa Ver 4 powder pillow is added to each sample in place of the individual reagents. Development time: at least 5 minutes.

Lab uses a 4 point calibration curve.

Lab is analyzing CCV and CCB after every 10 samples.

3. Reporting Limits

The reported detection limit for Sulfate is 10mg SO_4 /L for liquids, and 100mg/Kg for soils.

4. Interferences

4.1 Color or suspended matter in large amounts will interfere. Some suspended matter may be removed by filtration (Section 9.2.1). If both are small in comparison with the sulfate ion concentration, correct for interference as indicated in the procedure (Section 9.2.1). Silica in

excess of 500mg/L will interfere, and in waters containing large quantities of organic material it may not be possible to precipitate $BaSO_4$ satisfactorily.

4.2 Sulfite may be oxidized to sulfate during analysis to give a positive error.

4.3 Sample temperature control is important for reproducibility of results. Prepare calibration curve and analyze samples adjusted to room temperature. Variations over 10 °C will cause appreciable error.

4.4 In the presence of organic matter certain bacteria may reduce sulfate to sulfide. To avoid this, store all samples at low temperatures up to a maximum holding time of 28 days

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in plastic bottles.

6.2 Sample Preservation

None.

6.3 Sample Shipping

NA

6.4 Sample Handling

Samples are stored at 4 ± 2 °C. Analyze within 28 days of collection. Bring samples to room temperature prior to analysis

7. Equipment and Supplies

7.1 Centrifuge tubes: 50mL plastic, with covers.

7.2 Spectrophotometer: For use at 420nm, with 2.5cm absorption cell.

7.3 Digital clock.

7.4 Glass fiber filters: Any size.

7.5 Membrane Filters, 0.45 μm.

7.6 Syringes

7.7 Pipets: Volumetric, various sizes.

7.8 Plastic Cups

7.9 Stirring Plate

7.10 Magnets of the same shape/size (for universal mixing)

8. Reagents and Standards

8.1 1000ppm Calibration Stock Sulfate Solution: Commercially prepared standard. A certificate of analysis is kept on file. Store at room temperature. Expires upon manufacturer's specified date.

8.1.1 Calibration Standards

Calibration standards are stored at room temperature and expire upon the manufacturer's specified date of the Stock Solution.

8.1.1.1 10ppm Calibration Standard: To a 50mL volumetric flask, add 0.5mL of the 1000ppm Stock Sulfate Solution (Section **8.1**). Bring to volume with Reagent water.

8.1.1.2 20ppm Calibration Standard: To a 50mL volumetric flask, add 1mL of the 1000ppm Stock Sulfate Solution (Section **8.1**). Bring to volume with Reagent water.

8.1.1.3 40ppm Calibration Standard: To a 50mL volumetric flask, add 2mL of the 1000ppm Stock Sulfate Solution (Section **8.1**). Bring to volume with Reagent water.

8.2 1000ppm ICV/LCS Stock Sulfate Solution: Commercially prepared standard. A certificate of analysis is kept on file. This solution is purchased as a third party standard; it is not a dilution of Section **8.1**. Store at room temperature. Expires upon manufacturer's specified date.

8.2.1 20ppm ICV/LCS Standard: To a 25mL volumetric flask, add 0.5 mL of the 1000ppm Stock Sulfate Solution (Section **8.2**). Bring to volume with Reagent water.

8.3 Sulfa Ver 4 Sulfate Reagent: HACH brand powder pillows, for a 25mL sample size. Store at room temperature. Expires upon manufacturer's specified date. Alternatively, Hanna Sulfate Reagent packets can be used (Part # HI93751-03).

8.4 Ottawa sand

8.5 Boiling Chips.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Analyze one Blank consisting of 25 ml DI plus a Sulfa Ver4 powder pillow per batch of 20 samples or less.

For soils: weigh 5g of sand; add 50 ml of DI, stir for 30 min then let sit for 1h. Filter water portion for analysis.

The results of the Blank must be below the reporting limit. If acceptance criteria are not met, the entire batch is re-prepared and reanalyzed.

9.2 Initial Calibration Verification (ICV)

Analyze one ICV (Section **8.2.1**) at the beginning of analytical run. % Recovery must be within +/- 10% of true value. If the ICV fails this criterion, the ICV is remade and reanalyzed. If failure continues, a new calibration curve is generated.

9.3 Laboratory Control Sample (LCS)

Analyze one LCS (the same as ICV; Section **8.2.1**) per batch of 20 samples or less. % Recovery must be within +/-10% of true value. If the LCS fails this criterion, the LCS is remade and reanalyzed. If failure continues, a new calibration curve is generated.

For soils: weigh 5g of sand; add 49 ml of DI and 1.0 ml of 1000 ppm ICV/LCS stock (Section **8.2**), stir for 30 min then let sit for 1h. Filter water portion for analysis.

9.4 Continuing Calibration Verification (CCV)

Analyze one CCV (20 ppm calibration standard **8.1.1.2**) after every 10th sample and at the end of the batch to verify the calibration curve. % Recovery must be within +/- 10% of true value. If the CCV fails this criterion, the CCV is remade and reanalyzed. If failure continues, a new calibration curve is generated, and all samples analyzed since the last acceptable CCV are re-prepared and reanalyzed.

9.5 Matrix Spike

Analyze one Spike per batch of **20** samples or less for method **4500** and one Spike for every **10** samples for method **9038**.

For spiked sample: in centrifuge tube transfer 20 ml of sample, add 0.5 ml of 1000 ppm Calibration Stock (Section **8.1**), bring up to 25 ml with sample.

For soils: weigh 5g of sample; add 49 ml of DI and 1.0 ml of 1000 ppm ICV/LCS stock (Section **8.1**), stir for 30 min then let sit for 1h. Filter water portion for analysis.

% Recovery must be within in-house control chart limits. If acceptance criteria are not met, a narrative is included with the data for inclusion on the Client report.

9.6 Laboratory Duplicate

Analyze one sample in duplicate per batch of **20** samples or less for method **4500** and one Duplicate for every **10** sample for method **9038**. % RPD must be within in-house control chart limits. If acceptance criteria are not met, a narrative is included with the data for inclusion on the Client report.

9.7 Method-specific Quality Control Samples

NA

9.8 Method Sequence

1) Generate Calibration Curve, ICV/LCS and ICB.
2) Measure sample background absorbance.
3) Filter samples if necessary and dilute if necessary.
4) Measure 25mL of sample or QC sample into a centrifuge tube.
5) Add one Sulfa Ver 4 Sulfate Reagent powder pillow to each centrifuge tube and mix.
6) Wait 5 minutes.
7) Measure the sample absorbance at 420nm in 2.5cm cell.
8) If absorbance is above curve, dilute the original sample and return to .2.
9) Subtract the background absorbance from the sample absorbance.
10) Calculate final results.

10. Procedure

10.1 Equipment Set-up

10.1.1 Calibration Curve Generation

A calibration curve is prepared fresh on each day of analysis. The curve is generated using the three calibration standards from Section **8.1.1**, and Reagent water blank.

Analyze these standards and the blank according to Section **10.3.5**.

Plot the absorbance values of the standards versus the corresponding sulfate concentrations. The correlation coefficient must be $\geq$ 0.995 before analysis of samples may begin.

All calibration points are back calculated (on excel form) and should be within 10% from true concentration, except lowest point of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse then 0.995.

10.2 Initial Calibration

The calibration curve must be initially verified using a 20ppm LCS Solution as prepared in Section **10.3.4.1** and analyzed per Section **10.3.5**

% Recovery must be within +/- 10% of true value. If acceptance criteria are not met, a new LCS is prepared and analyzed. If failure continues, a new calibration curve is prepared.

10.3 Equipment Operation and Sample Processing

10.3.1 Sample Preparation

Samples that contain heavy suspended material are filtered prior to analysis through a glass fiber filter. If substantial turbidity or color remains, the sample may be filtered again through a 0.45μm filter. Otherwise, if samples are slightly turbid or are colored, the background absorbance is determined as in Section **10.3.3**, following the Spectrophotometer Preparation in Section **10.3.2**.

For soil samples: Weigh 5g of sample in a plastic cup; add 50mL of DI. Stir for 30 minutes, then let sit for one hour (sample may be left overnight). Filter the water portion through a 0.45um filter and then analyze below as a water sample. Samples with a high background color may need additional dilution.

10.3.2 Spectrophotometer Preparation The spec is set to zero absorbance using only DI in a 2.5cm cell at a wavelength of 420 nm.

10.3.3 Sample Background Absorbance:

Determine the background absorbance of each sample by measuring an aliquot of the sample into the 2.5cm absorption cell and recording the sample absorbance at 420nm in the laboratory notebook.

If the sample background absorbance is > 0.2 , filter sample. Record filtration information on excel Sheet.

If the background is still too high following filtration, the filtrate may be diluted with Reagent Water.

10.3.4 QC Preparation

Refer to Section 10 for QC sample frequency.

10.3.4.1 20ppm LCS / CCV: Into a centrifuge tube, pipette 0.5ml of 1000ppm LCS Stock Sulfate Solution (sect **8.2**) and bring up to 25 ml with DI.

For Soil LCS (200mg/Kg): Weigh 5g of sand; add 1mL of 1000ppm LCS Stock Sulfate Solution and 49 ml of DI in a plastic cup. Stir and filter. Analyze as a liquid LCS.

10.3.4.2 20ppm Spike: Choose a sample in the batch to spike. Add 25mL of this sample to a centrifuge tube; add 0.5mL of the 1000ppm Calibration Stock Sulfate Solution (Section **8.1**).

200mg/Kg soil spike: Weigh 5g of sample; add 1mL of 1000ppm Spike stock Sulfate Solution and 49mL of DI in a plastic cup. Stir and filter. Analyze as a liquid.

10.3.5 Sample Analysis

Measure 25mL of prepared analytical samples and QC samples into a centrifuge tubes. Add one Sulfa Ver 4 Reagent powder pillow (Section **8.3**) to each centrifuge tube, cap and shake until reagent is dissolved. Wait 5 minutes.

Determine each sample absorbance by measuring an aliquot of the mixed sample into the 2.5cm absorption cell and recording the sample absorbance in the laboratory notebook.

If the sample absorbance is greater than the absorbance of the highest calibration standard, the original sample must be diluted with Reagent water, and a new background absorbance determined. Return to Sections **10.3.3** and then **10.3.5**

10.3.6 Correction for sample color and turbidity

Subtract the sample background absorbance determined in Section **10.3.3**, from the corresponding sample absorbance determined in Section **10.3.5**. This result is the net absorbance value, which is used for calculation in Section **11**.

10.4 Continuing Calibration

The calibration curve must be verified using a 20ppm CCV Solution as prepared in Section **8.1.1.2** and analyzed per Section **10.3.5**

% Recovery must be within +/- 10% of true value.. If acceptance criteria are not met, a new CCV is prepared and analyzed. If failure continues, a new calibration curve is prepared and all samples analyzed since the last acceptable CCV are re-prepared and reanalyzed against the new curve.

10.5 Preventive Maintenance

An instrument service company calibrates the Spectrophotometers on a semi-annual basis. Certificates are kept on file.

11. Data Evaluation, Calculations and Reporting

$$\text{Sulfate as mg } SO_4 \text{ / L} = \frac{\text{Net Absorbance - Y-intercept}}{\text{Slope}} \times \text{dilution factor}$$

To calculate soils:

To calculate result in mg/kg on received basis, multiply result in mgSO4/L on Extraction (Final) volume (ml) and divide on sample weight (g)

mgSO4/kg = mgSO4/L x Extraction volume (ml) / weight (g)

To calculate final result for soil samples based on dry weight, multiply final result on 100 and divide on %TS (in %)

SO4 mg/kg (dry weight) = SO4 mg/kg x 100 / TS (%)

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedence and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of LCSs, blanks and spike response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the LCS recovery falls outside the designated acceptance range, the laboratory is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

The quarterly method reporting limit (MRL) verification requirement is satisfied by the quarterly LOQ analysis, as per SOP/1732.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP ID 1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ)

SOP ID 1739 IDC/DOC Generation

SOP ID 1728 Waste Management and Disposal SOP

16. Attachments

None.

Tannin & Lignin

References: **HACH Method 8193,** adapted from Journal American Water Works Association, vol.66 #1, p. 44, (1974).

SM 5550B, Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

1. Scope and Application

Matrices: Drinking waters, wastewaters, boiler/cooling waters

Definitions: Refer to Alpha Analytical Labs Quality Manual.

Lignin is a plant constituent that often is discharged as a waste during the manufacture of paper pulp. Another plant constituent, tannin, may enter the water supply through the process of vegetable matter degradation or through the wastes of the tanning industry. Tannin also is applied in the so-called treatment of boiler waters, where it reduces scale formation by causing the production of a more easily handled sludge.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the spectrophotometer and in the interpretation of spectrophotometric data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

This method measures all hydroxylated aromatic compounds, including tannin, lignin, phenol and cresol. Analytically these compounds are reported as Tannic Acid. Upon adding the coloring reagents, a blue color is produced which is proportional to the amount of above compounds present in the sample.

2.1 Method Modifications from Reference

The following reagents from the Hach Method 8193 are used in place of reagents described in Standard Method 5550B: 0.5 mL of Hach Tanni Ver 3 Tannin-Lignin reagent instead of 1 mL of Folin Phenol reagent and 5 mL Sodium Carbonate solution instead of 10 mL Carbonate-Tartrate reagent. Method 5550B allows using commercially prepared reagent (see sec 3.a of the method).
25min time period is used for color development (instead of 30 min).

3. Reporting Limits

The reporting limit is 0.2mg/L Tannic Acid.

4. Interferences

Any substances able to reduce Folin phenol reagent will produce a false positive response.

Organic chemicals known to interfere include hydroxylated aromatic, proteins, humic substances, nucleic acid bases, fructose and amines.

Inorganic substances known to interfere include iron II, manganese II, nitrite, cyanide, bi sulfite, sulfite, sulfide, hydrazine, and hydroxylamine hydrochloride.

Both 2 mg ferrous iron/L and 125mg sodium sulfite/L individually produce a color equivalent to 1 mg tannic acid.

Sulfide interference is eliminated by adding 1 mL of formaldehyde to the 25 mL sample size before testing.

To eliminate ferrous iron interference of up to 20mg/L, add 0.2g of sodium pyrophosphate to the sample before testing.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection: Collect samples in clean plastic or glass containers.

6.2 Sample Preservation: None.

6.3 Sample Shipping: None.

6.4 Sample Handling: Store samples at 4 degrees (C) . Bring up to 20 ± 2 ° C just prior to testing. No hold time restrictions.

7. Equipment and Supplies

7.1 Genesys 10 Vis Spectrophotometer or equivalent : Capable of reading at 700nm using a 2.5cm cell.

7.2 Centrifuge

7.3 Centrifuge tubes: 50 mL

7.4 Pipets: Class A; sizes 0.5mL, 1.0ml, 2.0 mL, 5.0 mL 6.0 mL, 8.0 mL

7.5 Volumetric Flasks: Class A; sizes 25mL, 50mL and 1000 mL, with ground glass stoppers.

8. Reagents and Standards

8.1 Sodium carbonate solution: HACH Cat. # 675-49

8.2 TanniVer3 Tannin-Lignin Reagent: HACH Catalog # 2560-42; store at room temperature, use vendor's expiration date.

8.3 Tannic Acid: Fisher Cat. # A310, HACH Catalog # 791-14 or equivalent; store at room temperature no expiration date. Keep tightly sealed. (NOTE: Two different sources of Tannic Acid are required, one for preparation of Calibration Standards and Spike (Section 8.7), and the other for preparation of the LCS (Section 8.8).)

8.4 Formaldehyde solution: HACH Catalog # 2059-32. Store at room temperature, expires 6 months from date opened. Used to remove possible Sulfite interference.

8.5 Sodium pyrophosphate: HACH Catalog # 1429525. Store at room temperature. No expiration date. Keep tightly sealed.

8.6 De-ionized Water (DI)

8.7 1000ppm Tannic Acid Stock Calibration Standard and Stock Spiking Standard: To a 1000mL volumetric flask, add 500mL of DI and dissolve 1.0 g of Tannic Acid (Section 8.3). Bring to volume with DI. Prepare every 6 months. Store at room temperature.

8.7.1 40ppm Working Standard : Pipet 2 mL of 1000ppm Tannic Acid Stock Calibration Standard (Section 8.7) into a 50 mL volumetric flask. Bring to volume with DI. Prepare fresh daily. Store at room temperature.

8.7.2 Calibration Standards: Prepare these standards fresh whenever it is necessary to generate a new calibration curve.

8.7.2.1 0.1ppm Calibration Standard: Into a 200mL volumetric flask add 0.5mL of 40ppm Working Standard (Section 8.7.1). Bring to volume with DI water.

8.7.2.2 0.2 ppm Calibration Standard: Into a 200mL volumetric flask add 1.0mL of 40ppm Working Standard (Section 8.7.1). Bring to volume with DI water

8.7.2.3 0.8ppm Calibration Standard: Into a 50mL volumetric flask add 1.0mL 40ppm Working Standard (Section 8.7.1). Bring to volume with DI water.

8.7.2.4 1.6ppm Calibration Standard: Into a 50mL volumetric flask add 2.0mL 40ppm Working Standard (Section 8.7.1). Bring to volume with DI water.

8.7.2.5 4.0ppm Calibration Standard: Into a 50mL volumetric flask add 5.0mL 40ppm Working Standard (Section 8.7.1). Bring to volume with DI water.

8.7.2.6 6.4ppm Calibration Standard: Into a 50mL volumetric flask add 8.0mL 40ppm Working Standard (Section 8.7.1). Bring to volume with DI water.

8.8 1000ppm Tannic Acid Stock LCS: (NOTE: Use an alternate source of Tannic acid than that used in Section 8.7.) To a 1000mL volumetric flask, add 500mL of DI and dissolve 1.0 g of Tannic Acid (Section 8.3). Bring to volume with DI. Prepare every 6 months. Store at room temperature.

8.8.1 40ppm Working LCS / Spiking Standard: Pipet 2 mL of 1000ppm Tannic Acid Stock LCS Standard (Section 8.8) into a 50 mL volumetric flask. Bring to volume with DI. Prepare daily before each use. Store at room temperature.

8.8.1.1 1.6 ppm LCS: Pipet 1 mL of the 40ppm working LCS standard (Section 8.8.1) into a 25 mL volumetric flask and bring up to volume with DI. The 25mL aliquot of this solution is used for the LCS sample. Prepare the LCS on a daily basis or whenever the analysis is performed.

8.8.1.2 Spiking solution: To a 25 mL sample aliquot, pipet 1.0 mL of the 40ppm Working LCS Standard (Section 8.8.1).

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank: The Blank QC sample consists of a 25mL aliquot of DI water. Analyze one Blank sample at the beginning of each batch of 20 or less samples. Blank sample results must be less than the Reporting Limit (RL). If results are greater than the RL, another aliquot is re-poured and re-analyzed. If results continue to be greater than the RL, the entire sample batch must be re-processed.

9.2 Laboratory Control Sample (LCS): Following the Blank, analyze one LCS (Section 8.8.1.1) at the beginning of each batch of 20 samples or less. % Recovery must be within 80-120% before continuing sample analysis. If the LCS recovery is outside of the control limits, then a new LCS sample aliquot is re-poured and re-analyzed. If the LCS failure continues, a new LCS solution is prepared (Section 8.8.1.1) and re-analyzed. If failure continues, a new calibration curve must be prepared and analyzed (Section 10.2).

9.3 Initial Calibration Verification (ICV): Refer to LCS (Section 9.2).

9.4 Continuing Calibration Verification (CCV): The LCS (Section 9.2) is utilized for the CCV in this analysis. A CCV and Continuing Calibration Blank (Section 9.1) QC sample pair are read on the spectrophotometer following every 10 samples and also at end of the sample batch. % Recovery for the CCV must be within 80-120%. If the CCV recovery is outside of the control limits, then a new CCV sample aliquot is re-poured and re-analyzed. If the CCV failure continues, a new CCV solution is prepared (Section 8.8.1.1) and re-analyzed. If failure continues, a new calibration curve must be prepared and analyzed (Section 10.2). All samples analyzed since the last acceptable CCV must be reanalyzed.

The Continuing Calibration Blank (CCB) results must be less than the Reporting Limit (RL). If results are greater than the RL, the CCB is re-poured and re-analyzed. If failure continues, the problem must be found and corrected, and all samples analyzed since the last acceptable CCB must be reanalyzed.

9.5 Matrix Spike: Analyze one Matrix Spike sample per batch of 20 samples or less. (Section 8.8.1.2.) % Recovery must be within 75-125%. If recovery is outside the acceptance criteria, the results must be narrated for inclusion on the Client report.

9.6 Laboratory Duplicate: Analyze one sample in Duplicate per batch of 20 samples or less. The RPD must be $\leq$ 25%. If the RPD is outside the acceptance criteria, the results must be narrated for inclusion on the Client report.

9.7 Method-specific Quality Control Samples: None.

9.8 Method Sequence

- Blank
- LCS/ICV
- 10 samples/QC samples
- CCV/CCB
- 10 samples
- CCV/CCB

10. Procedure.

10.1 Equipment Set-up: Turn on the spectrophotometer to warm up the instrument. Set the wavelength at 700nm.

10.2 Initial Calibration:

10.2.1 Prepare calibration standards per Section 8.7.

10.2.2 Pour 25mL of each standard and a DI blank into new centrifuge tubes. Add coloring reagents as described in Sections 10.3.2 – 10.3.4, and wait 25 minutes.

10.2.3 Using the 2.5cm cell, zero the Spectrophotometer at 700nm with an aliquot of the colored Blank sample.

10.2.4 Read each standard using the 2.5cm cell at 700nm. Record the absorbance values in the Laboratory notebook.

10.2.5 Plot the absorbance values of the standards versus the corresponding concentrations.

Correlation coefficient will be calculated. All calibration points are back calculated on excel and should be within 10% from true concentration, except lowest point of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 50% and correlation coefficient must be > or = 0.995. If calibration curve criteria failed, then new calibration standards have to be re-analyzed and new calibration curve has to be generated and all calibration points have to be recalculated to meet criteria. If second calibration curve fails acceptance criteria, then analysis have to stopped, problem has to be identified; new calibration standards made and analyzed. The calibration curve is valid for a maximum of 1 year, unless the LCS % Recovery falls outside of the acceptance criteria of 80%-120% (Section 9.2).

10.3 Equipment Operation and Sample Processing:

10.3.1 Pour 25 mL of each well-homogenized sample and QC sample into a labeled centrifuge tube. Make sure that sample is brought up to 20 ± 2 ° C (see sec. 6.4)

10.3.2 Add 1 mL of Formaldehyde Solution (Section 8.4) to each centrifuge tube. Swirl to mix. Formaldehyde solution is added to remove potential Sulfite interference.

10.3.3 Add one 0.2g scoop of Sodium Pyrophosphate (Section 8.5) to each centrifuge tube and swirl until crystals dissolve.

10.3.4 Add 0.5mL of TanniVer3 Reagent (Section 8.2) and 5.0mL of Sodium Carbonate solution (Section 8.1) to each centrifuge tube. Swirl to mix.

10.3.5 Wait 25 minutes for full color development.

NOTE: If the colored sample is cloudy or turbid, cap the tube and centrifuge prior to proceeding.

10.3.6 Using the 2.5cm cell, zero the Spectrophotometer at 700nm with an aliquot of the colored Blank sample.

10.3.7 Read each sample and QC sample using the 2.5cm cell at 700nm. Record the absorbance values in the Laboratory notebook.

10.3.7.1 If a sample result is greater than the highest standard used for the calibration curve, prepare an appropriate dilution of the original sample using DI water. Proceed back to Section 10.3.1 to prepare and analyze the diluted sample.

10.4 Continuing Calibration:

Refer to Section 9.4. The continuing calibration standard and blank are processed as in Section 10.3.

10.5 Preventive Maintenance

Spectrophotometers are calibrated semiannually by an instrument service company. Certificates are kept on file.

11. Data Evaluation, Calculations and Reporting

All absorbance values obtained in Section 10.3 are recorded in the Tannin & Lignin Elecronic Laboratory notebook (ELN). Electronically prepare a record using the ELN by following the Alpha Work Instructions WI/11-01.

The ELN calculates the concentration of tannic acid in each sample and QC sample, using the following formula:

$$\text{mg Tannic Acid/L} = \frac{(\text{Absorbance}) - (\text{y-intercept})}{\text{Slope}} \times \text{Dilution factor}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Refer to Section 9 for Quality Control Criteria and processes.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1734 and 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method

14. Pollution Prevention and Waste Management

Samples and standards that have been prepared and analyzed must be disposed of in the Organic –Wet Chemistry waste stream.

Refer to Alpha's Chemical Hygiene Plan and SOP 1728 Waste Management and Disposal for further pollution prevention and waste management information.

15. Referenced Documents

SOP 1732MDL Generation

SOP 1734, 1739 IDC Generation

SOP 1728 Waste Management and Disposal

WI/11-01 Electronic Laboratory Notebook Procedure – Wet Chemistry

16. Attachments

None.

TCLP Extraction

Metals and Semi-Volatile Organics

Reference Method: **EPA 1311**

Reference: **SW-846**, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Update I, July 1992

State of Connecticut, DEP, TCLP by SW-846 Method 1311, Version 2.0, December 2006

1. Scope and Application

The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

Definitions: See Alpha Laboratories Quality Manual Appendix A.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Extractions Manager, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

For liquid wastes, (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP extract.

For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

For additional detailed instruction, see TCLP and SPLP Work Instruction (ID# 17618).

2.1 Method Modifications from Reference

- 10.1.1.3.3 – Shaker table is used instead of stir bars. Samples not covered when shaking.
- 8.2 – 4N NaOH is used instead of 1N NaOH.

3. Reporting Limits

The Reported Detection Limit is determined by the amount of sample used for preparation. Therefore a review of Client requirements for Reporting Limits is necessary prior to sample preparation. Refer to analytical method SOPs.

4. Interferences

4.1 The most common cause of contamination is from improperly cleaned glassware and lab supplies. All glassware and re-useable extraction equipment must be scrupulously cleaned, following the Organic Extraction Glassware Cleaning and Handling SOP/1953 and Work Instruction 10995, Solvent rinsing guide.

4.2 Impurities in solvents and reagents may also yield artifacts and/or interferences that may compromise the results of sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of extract preparation and analysis by preparing method blanks with each extraction batch. The same solvents and reagents are used for the method blank and the associated samples.

4.3 Phthalate esters contaminate many types of products used in the laboratory. Plastic materials must not contact the samples or extracts, as phthalates could be easily leached from the plastic. The exception is in the use of various pre-packed reagent cartridges (Florisil, Silica gel) used in the extract cleanup steps. Each new lot of cartridges is checked for contamination, and is monitored on an on-going basis through the analysis of method blanks.

4.4 Additional specific interference or contamination concerns are addressed in the various analytical SOPs.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. This includes wearing personal protective equipment such as a labcoat, safety glasses, gloves and respirator (as necessary).

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards or solvents and when washing glassware.

5.2 All extract concentration steps must be performed in the extraction hoods. All solvent and extract transfers must also be handled in the hood.

5.3 All expired stock standards, working standards, and spent sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be properly labeled with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet or in the vented cabinets found under the hoods.

5.5 All waste solvents must be transferred to the satellite waste storage containers located in the extraction lab. Separate containers are provided for chlorinated and non-chlorinated solvents and must be used accordingly. Under no circumstances are solvents to be poured down the sink drains.

5.6 Inspect all glassware prior to use. Do not use any glassware that is chipped, cracked or etched if it could present a safety hazard. Damaged glassware is put aside for repair, otherwise discard the piece.

5.7 All Field Samples must be opened and handled in a hood.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass containers. Soils are collected in 8oz amber glass jars, and aqueous samples are collected in 500mL amber glass jars.

6.2 Sample Preservation

Preservatives are not added to samples before extraction.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples may be refrigerated at 4 ± 2°C, unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

Samples must undergo TCLP extraction within the time periods listed in Table 1.

7. Equipment and Supplies

7.1 Agitation Apparatus: End-over-end 30 ± 2 rpm. Calibrated Quarterly.

7.2 Vacuum Filtration Unit

7.3 0.6 to 0.8 µm Glass Fiber Filter Paper: 70 mm, 90 mm, and 120 mm, and acid prewashed 47 mm for all metals analysis.

7.4 2 L Glass/Plastic-coated Bottles

7.5 7 L Plastic Extraction Vessel: For Lamp Extraction

7.6 pH Meter: +/- 0.01 units resolution

7.7 pH test paper: 0-14 pH range

7.8 Laboratory Balance: +/- 0.1 g tolerance

7.9 250 mL Beaker or equivalent

7.10 Watch Glass: Used with Beaker (Section 7.8)

7.11 Stir Bars

7.12 Hot Plate: 1x1 ft (120 V) Barnestead-Thermolyne – Type 2200

7.13 2 L Graduated Cylinder Class A

7.14 250 mL Graduated Cylinder Class A

7.15 Teflon coated Sieve: 9.5 mm (0.375-inch)

7.16 Oven: Capable of maintaining 100 ± 20°C

7.17 Automatic Shaker Table

7.18 Vessel to store fluid #1

7.19 Vessel to store fluid #2

7.20 Calibrated thermometer: Temperature calibration point 50°C. Range: 0-100°C.

7.21 Bottle Top Dispenser: 0-5 mL, 0-100 mL

7.22 1 L Amber Bottle: Thermo Scientific, Item # 341-0950, Jar Tall Amber WM.

7.23 120 mL Plastic Bottle: Greenwood, Product # 07-120WMF24503, Natural HDPE WM Packer assembled w/38-400 F-217 Lined Cap.

7.24 250 mL Plastic Bottle: Greenwood, Product # 07-250OB45F22603, Natural HDPE WM Oblong Bottle Assembled w/45-400 F-217 Lined Cap.

8. Reagents and Standards

8.1 Hydrochloric Acid: 1N: Dilute 83 mL conc. HCl to 1 liter with reagent water. Store at room temperature; expires one year from date of prep.

8.2 Sodium Hydroxide: 4N: Dissolve 160 grams NaOH in 1 liter of reagent water. If larger volumes are required, prepare using similar ratios. Store at room temperature; expires one year from date of prep.

8.3 Glacial acetic acid: CH_3COOH. Store at room temperature; expires according to manufacturer's specifications.

8.4 Reagent Water, (ASTM Type II): All references to water within this SOP refer to reagent water unless otherwise specified. Reagent water is interference-free.

8.5 Extraction Fluid #1: Add 5.7 mL glacial acetic acid to 500 mL of reagent water, add 64.3 mL of 4N NaOH, and dilute to a volume of 4 liters. Check the pH of the solution after preparation and record the value in the Extraction Fluid Logbook. The pH is within 4.93 $\pm$ 0.05 when correctly prepared. In addition, the pH of the fluid is verified prior to each use and the pH recorded in the extraction logbook. Store at room temperature; no expiration date, however the pH must be maintained.

NOTE: Larger volumes or extraction fluid may be prepared, provided the ratios remain the same. For example, a typical preparation of 200 L of Extraction Fluid #1:

1140 mL Glacial Acetic Acid + 3215 mL 4N NaOH diluted to 200 L with reagent water

8.6 Extraction Fluid #2: Dilute 5.7 mL glacial acetic with reagent water to a volume of 1 liter. Check pH of solution which should be 2.88 ± 0.05 when correctly prepared. Record the pH in the Extraction Fluid Logbook.

The pH is checked prior to use to ensure that this fluid is prepared accurately. If the pH is not within the above specifications, the fluid is discarded and fresh extraction fluid prepared. Store at room temperature; no expiration date, however the pH must be maintained.

NOTE: Larger volumes or extraction fluid may be prepared, provided the ratios remain the same. For example, a typical preparation of 20L or 50L Extraction Fluid #2:

19,886 mL of reagent water + 114 mL Glacial Acetic Acid = 20 L of fluid.

49,715 mL of reagent water + 285 mL Glacial Acetic Acid = 50 L of fluid

8.7 Trace Nitric Acid (1:1 HNO3): 500 mL HNO3 diluted to 1 liter with DI water. Store at room temperature in hood. Expires 3 months from prep.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

A minimum of **one blank** (using the same extraction fluid lot number as used for the samples) must be prepared and analyzed for every 20 extracts or every 48 hours, whichever comes first. The Blank vessel is a randomly selected container from the stock and is not one designated for blank use only.

9.2 Laboratory Control Sample (LCS)

Not applicable.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

- Determine analysis to be performed (Metals, ABN, Pest, PCB, Herbicides)
- Check sample; ensure there is enough sample for the extraction
- Determine the %solids of the waste sample

- Determine particle size reduction requirement
- Determine the extraction fluid to be used
- Extract the sample for TCLP
- Filter the extract.
- Aliquot extract to proper preparation departments
- Prepare extracts according to analytical protocols

10. Procedure

10.1 Equipment Set-up

10.1.1 Preliminary Evaluation

Perform preliminary TCLP evaluations on a minimum of 100g aliquot of waste. If there is limited sample volume, the chemist must contact the project manager. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include:

(1) determination of the percent solids (Section 10.1.1.1);

(2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Section 10.1.1.1.2);

(3) determination of whether the solid portion of the waste requires particle size reduction (Section 10.1.1.2); and

(4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste (Section 10.1.1.3)

10.1.1.1 Determine % Solids

10.1.1.1.1 If waste contains no free liquid, proceed to Section 10.1.1.2 where the waste is the solid portion. If the waste contains less than 0.5% solids, proceed to Section 10.1.1.1.2.

10.1.1.1.2 Mulitphasic waste - separate liquid and solid portion

10.1.1.1.2.1 Pre-weigh a filter. Pre-weigh a beaker or graduated cylinder that will hold the initial filtrate. Pre-weigh empty filtrate container. Record weights in the extraction logbook.

10.1.1.1.2.2 Assemble the vacuum filtration unit.

10.1.1.1.2.3 Weigh a subsample of the waste (100 g minimum), or pour 100 mL of the sample into a cylinder, and record the weight or volume in the extraction logbook.

10.1.1.1.2.4 Transfer the waste sample to the filter holder, spreading evenly over the filter.

10.1.1.1.2.5 Allow enough time for the liquid to pass through the filter (at least 15 minutes).

10.1.1.1.2.6 The material in the filter holder is defined as the SOLID PHASE of waste and the filtrate is defined as the LIQUID PHASE.

Note: The SOLID PHASE may appear liquid in some samples. Do Not replace original filter. Use only one filter.

10.1.1.1.2.7 Weigh the filtrate filled container and calculate the weight of the LIQUID PHASE as follows:

$$W_{LP} = W_F - W_C$$

Where:

W_{LP} = Weight of the LIQUID PHASE
W_F = Weight of the filtrate filled container
W_C = Weight of container

10.1.1.1.2.8 Calculate the weight of the SOLID PHASE using the following formula:

$$W_{SP} = W_W - W_{LP}$$

Where:

W_{SP} = Weight of the SOLID PHASE
W_W = Weight of the waste sample (Section 10.1.1.1.2.3)
W_{LP} = Weight of the LIQUID PHASE (Section 10.1.1.1.2.7)

10.1.1.1.2.9 Calculate the percent solids using the following formula:

$$\%\ Solids = \frac{Wsp}{Ww} \times 100$$

Where:

W_{SP} = Weight of the SOLID PHASE (Section 10.1.1.1.2.8)
W_W = Weight of the waste sample (Section 10.1.1.1.2.3)

10.1.1.1.2.10 If the % Solids determined above (section 10.1.1.1.2.9) is less than 0.5%, then the filtrate is the TCLP extract. Filter sample until enough filtrate is obtained for analysis and proceed to Section 10.3.1.3.6.

10.1.1.1.2.11 If the % Solids determined above in section is greater than or equal to 0.5%, all liquids entrained in the filter and waste must be removed to determine the % dry solids. Remove the solid phase and filter from the filtration apparatus. Heat at 100 ± 20° C in an oven (section 7.16) until two successive weights yield the same value within ±1%. Record weights in the extraction logbook.

10.1.1.1.2.12 Calculate the percent dry solids using the following formula:

$$\%\ Dry\ Solids = \frac{W_{DW} - W_F}{W_W} \times 100$$

Where:

W_{DW} = Weight of dried waste + filter (Section 10.1.1.1.2.11)
W_F = Weight of the filter (Section 10.1.1.1.2.1)
W_W = Weight of waste sample (Section 10.1.1.1.2.3)

10.1.1.1.2.13 If the % dry solids determined above (section 10.1.1.1.2.12), is less than 0.5%, then the filtrate is the TCLP extract. Filter sample until enough filtrate is obtained for analysis and proceed to Section 10.3.1.3.6.

10.1.1.1.2.14 If the % dry solids determined above is greater than or equal to 0.5%, the sample is multi-phasic. Determine the weight of the dry waste by subtracting the weight of the filter from the weight of the dry waste and filter as determined follows and record in extraction logbook:

Weight of dry solids= **W_{DW} . W_F**

Where:

W_{DW} = Weight of dried waste + filter (Section 10.1.1.1.2.11)
W_F = Weight of the filter (Section 10.1.1.1.2.1)

Proceed to Section 10.3.1.3. (**NOTE:** Do not dispose of dried waste + filter.)

10.1.1.2 Determine whether waste requires particle size reduction

Evaluate the solid phase of the waste sample (Section 10.1.1.1.2.6), to determine if particle size reduction is necessary. Particle size reduction is required unless the solid has a surface area greater than 3.1 cm^2 per gram or is smaller than 1 cm in its narrowest dimension (i.e.: will not pass through a 9.5 mm (0.375-inch) standard sieve).

If the surface area is smaller or the particle size is larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting or grinding the waste to a surface area or particle size as described above.

NOTE: Surface area criteria is meant for filamentous (e.g. paper, cloth, etc.) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, those samples should be reduced to meet the 1 cm dimension criteria.

10.1.1.3 Extraction Fluid Determination

10.1.1.3.1 Weigh out a 5 g subsample of the solid phase waste, if the sample is all solid. If the sample is multiphasic, weigh out a 5 g subsample of the solid waste from Section 10.1.1.1.2.6.

10.1.1.3.2 Transfer the 5 g aliquot to a 250 mL beaker (or equivalent) and add 96.5 mL of reagent water. (NOTE: Less weight and volume may be used, provided the ratio remains the same.)

10.1.1.3.3 Place beaker (or equivalent) in the automatic shaker (Section 7.17) for 5 minutes.

10.1.1.3.4 Measure using pH strip and record the pH in the extraction logbook.

10.1.1.3.5 If pH is less than 5.0, use extraction fluid #1, proceed to Section 10.3.

10.1.1.3.6 If pH is greater than 5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watch glass, heat to 50°C+/- 2°C on a hotplate and hold for 10 minutes.

Note: heating block load tests have shown heating times of up to 25 minutes prior to the sample reaching the specified temperature. The 10 minute hold begins only after the temperature is reached and monitored using a calibrated thermometer placed in a sample or one representative of the batch.

Cool solution to room temperature and record pH. If the pH is less than 5.0, use extraction fluid #1. Otherwise, use extraction fluid #2.

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Extraction of SOLID PHASE

10.3.1.1 A 100 gram minimum aliquot of SOLID PHASE is extracted to produce the TCLP extract, as outlined below. In some cases, a larger sample size may be required in order to produce enough liquid for the required analysis. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analysis, more than one extraction may be performed and the extracts combined for analysis. If there is limited sample volume, the chemist must contact the project manager.

10.3.1.2 100% Solid Waste

10.3.1.2.1 If the waste is 100% solid, homogenize the sample and weigh out an aliquot (100 gram minimum), follow Sections 10.4.1.2 (particle size reduction) and then proceed to 10.3.1.3.2 (extraction fluid amount).

10.3.1.3 Liquid or Multiphasic Waste

10.3.1.3.1 Quantitatively transfer the solid material, along with the filter (from Section 10.1.1.1.2.14), into a glass extractor bottle (plastic may be used if only metals are being analyzed).

Repeat sections 10.1.1.1.2 through .6 if additional final volume is needed for the intended analysis.

10.3.1.3.2 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$V_{EF} = \frac{20 \times \%\,Solid \times W_W}{100}$$

Where:

V_{EF} = Volume of extraction fluid required.
(Density = 0.998g/L, therefore assume volume is equivalent to weight.)
%Solid = Percent solid as determined in section 10.1.1.1.2.9
(100% if waste contains no free liquid as determined in section 10.1.1.1.1)
W_W = Weight of waste (Section 10.1.1.1.2.3)

10.3.1.3.3 Add the volume of the appropriate extraction fluid (determined in Section 10.1.1.3) to the extraction vessel. Glass amber 2.5 L for organics and metals, 2.5 L plastic if for metals only analysis. New (never used) 2.5 L HPDE plastic vessels for PFAS (Reference: PFAS by SPE and LC/MS/MS Isotope Dilution (ID#23528).

10.3.1.3.4 Close the extractor bottle tightly. Secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours while maintaining room temperature at 23 ± 2° C. Record the agitation device (Tumbler) ID in the extraction logbook.

10.3.1.3.4.1 The room temperature (°C) and the extraction time are recorded in the extraction logbook at both the beginning and the end of extraction.

10.3.1.3.4.2 To verify that the proper room temperature is maintained during extraction, a Maximum/Minimum thermometer is reset at the beginning of the extraction period. This thermometer is reviewed at the end of the extraction period and the Maximum and Minimum temperatures are noted in the extraction logbook.

10.3.1.3.4.3 If proper room temperature is not maintained during the extraction period, it is considered a variation from the method and must be written into a laboratory narrative and submitted with the reported data. The Department Supervisor is immediately notified to determine the proper corrective action to be taken.

10.3.1.3.4.4 If determination is made that a MAX/MIN thermometer reading is outside of the acceptance range (21C-25C), it must be confirmed by comparison to the average temperature of the room during the extraction period recorded in the data logger records. Any MAX/MIN temperature reading that exceeds acceptance criteria must be reviewed by the department manager or team leader and QA.

10.3.1.3.5 Following extraction, vacuum filter the extract through glass fiber filters (acid precleaned for metals analysis). For this final filtration, the glass fiber filter may be changed to facilitate quicker filtration but should be kept to a minimum when practical. Use of pre-filters is prohibited for TCLP.

10.3.1.3.6 Prepare the TCLP extract as follows:

10.3.1.3.6.1 If the waste contained no initial liquid phase, the filtered extract obtained is defined as the TCLP extract. Proceed to 10.3.1.3.7.

10.3.1.3.6.2 If the waste contained a liquid phase, check its compatibility with the extraction fluid determined in Section 10.1.1.3. If the liquids are compatible (miscible), then the initial liquid phase (Section 10.1.1.1.2.6) and the filtered extract are combined and analyzed together.

10.3.1.3.6.3 If the initial liquid phase of the waste obtained in Section 10.1.1.1.2.6, is not compatible with the filtered extract, do not combine the liquids. Analyze these liquids, collectively defined as the TCLP extract, separately and mathematically combine the results, as described below. Determine the volume of the individual phases, conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$(\mathrm{mg/L}),\ \mathrm{Final\ Analyte\ Concentration} = \frac{(\mathbf{V_1})(\mathbf{C_1}) + (\mathbf{V_2})(\mathbf{C_2})}{\mathbf{V_1} + \mathbf{V_2}}$$

Where:

$\mathbf{V_1}$ = Volume of the first phase (L)
$\mathbf{C_1}$ = Analyte concentration first phase (mg/L)
$\mathbf{V_2}$ = Volume of the second phase (L)
$\mathbf{C_2}$ = Analyte concentration second phase(mg/L)

10.3.1.3.7 Record the pH of the TCLP extract in the extraction logbook prior to preservation of samples.

10.3.1.3.8 Metals analysis: transfer filtrate (or combined filtrates) to a 120 mL plastic bottle and add 2.5 mL of 1:1 HNO_3 preservative to all samples.

10.3.1.3.8.1 Check the pH of the preserved samples to ensure that the sample is properly preserved at a pH of less than 2.

10.3.1.3.9 Non-volatile Organics: transfer filtrate (or combined filtrates) to two 1 L glass amber bottles, adhere pre-made sample ID labels and transfer to extraction department refrigerators.

10.3.2 Extraction of Lamps

10.3.2.1 Lamps (for both TCLP extraction and 120x extraction) are broken down into small particles (See Section 10.1.1.2) and extracted using Extraction Fluid #1. The extraction procedure from Section 10.3.1.3.2 through 10.3.1.3.8 is followed with the exception that the entire lamp weight including endcaps is used. 7 L plastic extraction vessels are utilized for larger lamps that exceed 100 g total weight. The vessel size must be recorded in the logbook for lamp samples.

10.3.2.1.1 Check the sample comments for whether Mercuric Oxide (HgO) is required during the extraction. If so, make note in the logbook that the HgO packet was added to the extraction vessel prior to tumbling. Contact the project manager if HgO is requested but no packet is supplied.

10.3.2.2 The excess unfiltered extract is immediately decanted into the TCLP waste drum, making sure to leave behind in the extraction vessel the solid material that was extracted.

10.3.2.3 The remaining solid material is transferred to a plastic bag, labeled as TCLP Lamp Waste and brought to the Hazardous Waste department for proper disposal.

10.4 Continuing Calibration

Not Applicable.

10.5 Preventive Maintenance

None.

11. Data Evaluation, Calculations and Reporting

None.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

12.1 All Holding time exceedences, improper preservation and Extraction Anomalies are to be reported to a Supervisor or Manager. Non Conformance Reports may need to be issues through the Qualtrax System.

12.2 Refer to determinative method SOPs for additional Corrective Action information.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP 1732 These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

Not Applicable.

13.2.2 Continuing (DOC)

Not Applicable.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

SOP 2121 Chemical Hygiene Plan

SOP 1732 MDL/LOD/LOQ Generation

SOP 1739 IDC/DOC Generation

SOP 1728 Waste Management and Disposal SOP

WI 17618 TCLP and SPLP Work Instruction

16. Attachments

Table 1: Maximum Holding Times

Table 1

MAXIMUM HOLDING TIMES

Sample Maximum Holding Times (Days)

	From: Field collection **To:** TCLP extraction	**From:** TCLP extraction **To:** Preparative extraction	**From:** Preparative extraction **To:** Determinative analysis	**Total Elapsed Time**
Semi-volatiles	14	7	40	61
Mercury	28	N/A	28	56
Metals, except mercury	180	N/A	180	360

TCLP/SPLP Extraction - Volatile Organics EPA 1311/1312

Reference: EPA 1311, EPA 1312 SW-846, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Update I, July 1992.

1. Scope and Application

Matrices: The TCLP/SPLP extraction process is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

Definitions: Refer to Alpha Analytical Quality Manual.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of experienced analysts Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

For liquid wastes, (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 μm glass fiber filter, is defined as the TCLP/SPLP extract.

For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. A special extractor vessel is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 μm glass fiber filter.

If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately, and the results are mathematically combined to yield a volume-weighted average concentration.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

The analytical method detection limits determined by the laboratory are on file for review. See the 8260 SOP 32639.

4. Interferences

Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. This includes wearing personal protective equipment such as a lab coat, safety glasses, gloves and respirator (as necessary).

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

When the waste is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples are collected in Teflon-lined septum capped vials and stored at 4 ± 2° C). Samples are opened only immediately prior to extraction.

SPLP samples may be collected in 25g En Core sampling device.

6.2 Sample Preservation

Preservatives shall not be added to samples before extraction. For SPLP samples arriving in En Cores they must be extruded and tumbled within 48 hours of collection. Extrude the entire sample, but no more 25g since the vessel can only hold 500 mLs of fluid.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) must be extracted.

TCLP/SPLP extracts are prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.

Samples must undergo TCLP/SPLP extraction within the time periods listed in Table 1.

7. Equipment and Supplies

7.1 Agitation Apparatus: End-over-end 30 ± 2 rpm.

7.2 Zero Headspace Extraction Vessel

7.3 Glass Fiber Filters: 0.6 to 0.8µm, 90mm

7.4 50mL Gastight Syringe

7.5 Graduated Cylinder: 500 mL, 1L

7.6 Laboratory Balance: 0.01g tolerance

7.7 Pressure Gauge: 0-60 psi

7.8 Pressure Filtration Device

7.9 Drying Oven: 100 ± 2 °F

7.10 Fluid Metering Pump

7.11 pH Meter: The meter should be accurate to + 0.05 units at 25°C

8. Reagents and Standards

8.1 Analytical Standards: The standards shall be prepared according to the appropriate volatile organic analysis method 8260 SOP 32639.

8.2 Hydrochloric Acid (HCl), 1N: Dilute 83 mL conc. HCl to 1L with DI water.

8.3 Nitric Acid (HNO_3), 1N: Dilute 64 mL conc. HNO_3 to 1L with DI water.

8.4 Sodium Hydroxide (NaOH), 1N: Dissolve 40 gs NaOH in 1L of water.

8.5 Glacial acetic acid: CH_3COOH.

8.6 Ottawa sand: SiO_2

8.7 Extraction Fluid #1: Add 5.7 mL glacial acetic acid to 500 mL of DI water, add 64.3 mL of 1N NaOH, and dilute to a volume of 1L. Check pH of solution which must be 4.93 $\pm$ 0.05 when correctly prepared. The laboratory reagent number is referenced in the TCLP sample logbook.

NOTE: This extraction fluid is monitored frequently for impurities. The pH is checked prior to use to ensure that this fluid is prepared accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

8.8 Extraction Fluid #2: Organic-free reagent water.

8.9 Methanol: Purge and Trap Grade or equivalent. Store in flammables cabinet.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

A minimum of **one blank** must be analyzed for every batch or 20 extracts, whichever comes first. The blank is prepared by weighing 20g of Ottawa sand (SiO_2) and utilizing the same extraction fluid as used for the samples. The blank is then analyzed following the same procedures as analytical samples.

9.2 Laboratory Control Sample (LCS)

A laboratory control sample is not extracted. Refer to the 8260 procedure for the LCS information.

9.3 Initial Calibration Verification (ICV)

Not Applicable.

9.4 Continuing Calibration Verification (CCV)

Not Applicable.

9.5 Matrix Spike

Upon a client's request, a spike solution composed of all regulated compounds can be added to the TCLP/SPLP extract before analysis. See Method 8260 for more information.

9.6 Laboratory Duplicate

Not Applicable.

9.7 Method-specific Quality Control Samples

Not Applicable.

9.8 Temperature Measurement Guidelines

The ambient temperature of the tumbler rooms must be monitored from the beginning to the end of the TCLP, SPLP and ZHE extractions by use of a MIN/MAX thermometer to determine room temperature outages.

If a determination is made that a MAX/MIN thermometer reading is outside of the acceptance range (21C-25C), it must be confirmed by comparison to the average temperature of the room during the extraction period recorded in the datalogger records.

Any MAX/MIN temperature reading that exceeds acceptance criteria must be reviewed by the Department Manager or Team Leader and QA.

If a temperature exceedance is confirmed, the following narrative must be included on the Alpha job report.

Narrative # 2068

TCLP / SPLP TEMPERATURE EXCEEDANCE

[list] [qcsamplist]: The ambient temperature of the area in which the TCLP/SPLP/ZHE extraction was performed was outside the required 21-25 degrees C temperature range.

9.9 Method Sequence

The extraction sequence is:

- Disassemble ZHE; clean all parts with warm H_2O and soap; dry.
- Check sample; ensure there is enough sample for analysis.
- Determine %solids of the waste.
- Extract the TCLP/SPLP.
    - Record the temperature of the extraction room.
    - Following extraction, perform a leak test on the ZHE canisters.
- Draw sample with syringe from ZHE after extraction.
- Prepare sample for 8260 analysis

10. Procedure

10.1 Equipment Set-up

10.1.1 Preliminary Evaluation

10.1.1.1 Determine % Solids :

10.1.1.1.1 If waste contains no free liquid, proceed to Section 10.1.1.2 where the waste is the solid portion.

10.1.1.1.2 If the waste contains less than 0.5% solids, proceed to Section 10.3.2.

10.1.1.1.3 Multiphasic waste - separate liquid and solid portion

The following is performed by the Login Department.

10.1.1.1.3.1 Using a pressure filtration device pre-weigh the filter and the container to receive the initial filtrate. Record the weight in the laboratory notebook.

10.1.1.1.3.2 Assemble the pressure filter according to the manufacturer's directions.

10.1.1.1.3.3 Weigh a subsample of the waste (100 g minimum) and record the weight.

10.1.1.1.3.4 Transfer the waste sample to the filter holder spreading evenly over the filter.

10.1.1.1.3.5 Gradually apply a gentle pressure of 1-10psi until the pressurizing gas moves through the filter, proceed to Section 10.1.1.1.3.8.

10.1.1.1.3.6 If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10psi increments.

10.1.1.1.3.7 Stop the filtration when pressurizing gas passes through the filter, or 50psi is reached and no additional liquid passes through the filter in any 2 minute interval.

10.1.1.1.3.8 The material in the filter holder is defined as the SOLID PHASE of waste and the filtrate is defined as the LIQUID PHASE.

Note: the SOLID PHASE may appear liquid in some samples. Do Not replace original filter. Use only one filter.

10.1.1.1.3.9 Weigh the filtrate filled container and record the weight in the laboratory notebook. Calculate the weight of the LIQUID PHASE as follows:

$$W_{LP} = W_F - W_C$$

Where:

W_{LP} = Weight of the LIQUID PHASE
W_F = Weight of the filtrate filled container
W_C = Weight of container

10.1.1.1.3.10 Calculate the weight of the SOLID PHASE using the following formula:

$$W_{SP} = W_W - W_{LP}$$

Where:

W_{SP} = Weight of the SOLID PHASE
W_W = Weight of the waste sample
W_{LP} = Weight of the LIQUID PHASE

10.1.1.1.3.11 Calculate the percent solids using the following formula:

$$\%Solids = \frac{W_{sp}}{W_w x 100}$$

Where:

W_{SP} = Weight of the SOLID PHASE
W_W = Weight of the waste sample

10.1.1.1.3.12 If the % Solids determined in Section 10.1.1.1.3.11, is less than 0.5%, then proceed to Section 10.3.2, where the filtrate is the TCLP/SPLP extract.

10.1.1.1.3.13 Remove the solid phase and filter from the filtration apparatus, dry at 100 ± 2° F, cool and weigh. Record the weight in the laboratory notebook.

10.1.1.1.3.14 Calculate the percent dry solids using the following formula:

$$\% \text{ dry solids} = \frac{W_{DW} - W_F}{W_W \times 100}$$

Where:

W_{DW} = Weight of dried waste + filter
W_F = Weight of the filter
W_W = Weight of waste sample

10.1.1.1.3.15 If the % dry solids determine above, 10.1.1.3.14, is less than 0.5%, then proceed to Section 10.3.2, where the filtrate is the TCLP extract.

10.1.1.2 Determine whether waste requires particle size reduction

The following is performed by the Metals Prep Department.

Using fresh portion of the waste, obtain another solid phase of the waste sample by pressure filtration as determined in Sections 10.1.1.1.1. Use enough wet sample to obtain a dry portion for tumbling: at least 100 g. Evaluate the solid phase for particle size. Particle size reduction is required unless the solid has a surface area greater to 3.1 cm^2 per g, or is smaller than 1 cm in its narrowest dimension.

10.1.1.3 Determine Compatibility of Liquids

The following is performed by the Metals Prep Department.

If a LIQUID PHASE was filtered, check its compatibility with the extraction fluid. If the liquids are compatible (miscible), then only one TCLP/SPLP extract needs to be analyzed. If the liquids are not compatible, then the two liquids will have to be analyzed separately. Record in the laboratory notebook.

10.2 Initial Calibration

Not Applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 After the preliminary examination has been completed, the sample used in the following step comes from supplied VOA sample vials. The extraction for Volatile Organics is accomplished in a zero headspace extractor, ZHE.

10.3.2 Waste containing less than 5% dry solids

10.3.2.1 For waste containing less than 5% dry solids, weigh out up to a 500 g subsample and record its weight. Filter enough of the sample so that two 40 mL VOA vials may be filled with the filtered liquid. The liquid portion of the waste, after filtration, is defined as the TCLP/SPLP extract for volatile organic analysis. Proceed to Section 10.3.4.20.

10.3.3 100% Solid Waste

10.3.3.1 If particle size reduction is required (Section 10.1.1.2.), prepare the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 10.1.1.2. Waste and appropriate reduction equipment should be refrigerated to 4 ± 2° C prior to particle size reduction.

10.3.3.2 Weigh 20 gs of prepared waste and record the weight. If a 25 g Encore is supplied extrude and record the weight.

10.3.3.3 Quantitatively transfer the sample to the ZHE.

10.3.3.4 Assemble the ZHE according to manufacturer's instructions. Add the appropriate type and amount of fluid using the fluid metering pump. The fluid added is 20 times the amount of sample. For example, for a 20 g sample, add 400 mL of extraction fluid for TCLPs or 400 mL of organic free DI water for SPLPs. For a 25 g sample, 500 mLs of the appropriate fluid is added.

10.3.3.5 Position the ZHE in the vertical position with the liquid inlet/outlet valve on top and open.

10.3.3.6 Gradually apply pressure to the ZHE and bleed out any headspace.

10.3.3.7 Proceed to Section 10.3.4.16.

10.3.4 Waste containing greater than 5% dry solids

10.3.4.1 Determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge the ZHE} = \frac{20 \times 100}{\%\ \text{solids}}$$

Weigh out a subsample of the waste of the appropriate size and record the weight

10.3.4.2 If particle size reduction of the solid portion of the waste was required in Section 10.1.1.2, prepare the waste for extraction by crushing, cutting or grinding the solid portion of the waste to a surface area or particle size as described in Section 10.1.1.2. Waste and appropriate reduction equipment should be refrigerated to 4 ± 2° C prior to particle size reduction.

10.3.4.3 Quantitatively transfer the entire sample quickly to the ZHE. Assemble the ZHE in accordance with the manufacturer's instructions.

10.3.4.4 Place the device in the vertical position with the inlet/outlet valve on top and open.

10.3.4.5 Gradually apply pressure to the ZHE and bleed out any headspace quickly.

10.3.4.6 At the first appearance of liquid from the liquid inlet/outlet valve quickly close the valve.

10.3.4.7 Attach a 50mL gastight syringe to the liquid inlet/outlet valve and open the valve.

10.3.4.8 Begin applying gentle pressure to force the liquid phase of the sample into the filtrate collection container. When no additional liquid has passed through the filter in any 2-minute interval, proceed to Section 10.3.4.9.

10.3.4.9 Stop filtration by closing the liquid inlet/outlet valve.

10.3.4.10 Disconnect and weigh the 50mL gastight syringe. Record the weight in the laboratory notebook.

10.3.4.11 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

10.3.4.12 If the liquid phase is compatible with the extract (Section 10.1.1.3), store the filtrate at 4 ± 2°C until analysis. Then combine and analyze the filtrate as one extract.

10.3.4.13 If the liquid phase is not compatible with the solid extract, then analyze the filtrates separately and mathematically combine the results (Section 10.3.4.22).

10.3.4.14 Determine the weight of extraction fluid #1 for TCLP or organic free DI water for SPLP to add to the ZHE as follows:

Weight of extraction fluid = $20(W_S + W_T - W_{TF})$

Where:

W_S = Weight Sample
W_T = Weight of pre-weighed 50mL syringe
W_{TF} = Weight of filtrate in 50mL syringe

For 100% solid samples, the above formula reduces to:

Weight of extraction fluid = 20 x weight of sample

10.3.4.15 Add extraction fluid by backing piston off the filter and sucking fluid into the ZHE. Do not open the ZHE.

10.3.4.16 Re-pressurize the ZHE to 15 psi (1.03 bar) and place in the rotary agitation apparatus. Rotate at 30 ± 2 rpm for 18 ± 2 hours.

10.3.4.17 Post-extraction measure the final pressure of the ZHE canister. If pressure has decreased below 15 PSI the sample must be reset.

10.3.4.18 Following the agitation period separate the material in the extractor vessel into its liquid and solid phases.

10.3.4.18.1 100% solid waste samples: Filter enough liquid phase to fill two 40 mL vials. The filtrate is defined as the TCLP/SPLP extract. Proceed to Section 10.3.4.20.

10.3.4.18.2 Compatible liquid phases: Filter the entire extract into a 50mL gastight syringe. Transfer the contents of the syringe into 40mL VOA vials. Combine the extracts (Section 10.3.4.22) and analyze together.

10.3.4.18.3 Non-compatible liquid phases. Filter a sufficient quantity of the solid extract to fill two 40 mL VOA vials and analyze. The two liquids are collectively defined as the TCLP/SPLP extract and results are mathematically combined after individual analysis.

10.3.4.19 Following collection of the TCLP/SPLP extract immediately prepare the extract for analysis and store with minimal headspace at 4 ± 2°C until analyzed.

10.3.4.20 Follow the volatile organic analyses method 8260 SOP 32639 to analyze the TCLP/SPLP extract.

10.3.4.21 If the individual liquid phases (i.e. non-compatible liquids) are analyzed separately, determine the volume of the individual phases, conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:

V_1 = Volume of the first phase, (L)
C_1 = Analyte concentration first phase, (mg/L)
V_2 = Volume of the second phase, (L)

C_2 = Analyte concentration second phase, (mg/L)

10.4 Continuing Calibration

Not Applicable.

10.5 Preventive Maintenance

Tumbler rotation speed is verified annually and documented in the TCLP Annual Calibration Log, Form No.: 13855.

Balances are calibrated semi-annually by an instrument service company. Certificates are kept on file.

ZHEs are cleaned after extraction in soapy water. Filter screens and o-rings may be changed if they show degradation or the extraction vessel can't maintain pressure. Extremely contaminated vessels maybe further solvent rinsed with methanol

11. Data Evaluation, Calculations and Reporting

Calculations are included in Section 10.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

Not Applicable.

13.2.2 Continuing (DOC)

Not Applicable.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

32639 8260 SOP

13855 TCLP Annual Calibration Form

16. Attachments

Table1: Maximum Holding Times
Table 2: TCLP Analyte List

Table 1

MAXIMUM HOLDING TIMES

Sample Maximum Holding Times (Days)

	From: Field collection **To:** TCLP/SPLP extraction	**From:** TCLP/SPLP extraction **To:** Preparative extraction	**From:** Preparative extraction **To:** Determinative analysis	**Total Elapsed Time**
Volatiles	14/48 (hrs.)	N/A	14	28
Semi-volatiles	14	7	40	61
Mercury	28	N/A	28	56
Metals, except mercury	180	N/A	180	360

Table 2
TCLP Analyte List

Analyte	CAS No.
1,1 - Dichloroethene	75-35-4
1,2 - Dichloroethane	0107-06-02
1,4 - Dichlorobenzene	106-46-7
2-Butanone	78-93-3
Benzene	71-43-2
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
Tetrachloroethene	127-18-4
Trichloroethene	79-01-6
Vinyl chloride	75-01-4

Total Organic Carbon and Soot in Soil, Sediment

References: Perkin Elmer, "PE 2400 Series II CHNS/O Analyzer User's Manual," The Perkin-Elmer Corporation, April 1998.

USEPA, "Method 9060A Total Organic Carbon," in Test Methods for Evaluating Solid Waste, SW846, Revision 1 November 2004.

"Quantification of the Dilute Sedimentary Soot Phase: Implications for PAH Speciation and Bioavailability" published in Environmental Science and Technology, Vol. 31, No. 1, 1997 by Gustafsson, Haghseta, Chan, McFarlane and Gschwend.

"Reinterpreting Literature Sorption Data Considering Both Absorption into Organic Carbon and Adsorption onto Black Carbon" published in Environmental Science and Technology, Vol. 37, No. 1, 2003 by Accardi-Dey and Gschwend

1. Scope and Application

Matrices: This method is applicable for solid samples (soils, sediments, sludges).

Definitions: Refer to Alpha Analytical Quality Manual.

The organic carbon in a sample consists of a variety of organic compounds in various oxidation states. Some of these compounds may be oxidized by biological or chemical processes and can be measured by the Biochemical Oxygen Demand or Chemical Oxygen Demand tests. To measure the amount of organically bound carbon, the organic carbon molecules are broken down into single carbon units, and converted into a form that can be measured quantitatively.

The PE 2400 Series II CHNS/O Analyzer can simultaneously determine carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and oxygen (O) in organic material. In the CHN mode, the PE 2400 Series II CHNS/O Analyzer uses a combustion method to convert the sample elements to simple gases (CO_2, H_2O, and N_2). The resulting gases are homogenized and controlled to exact conditions of pressure, temperature, and volume. The homogenized gases are allowed to de-pressurize through a column where they are separated in a stepwise steady-state manner and detected as a function of their thermal conductivity and reported as a percentage for solid samples. Treated with hydrochloric acid prior to analysis, the Total Organic Carbon (TOC) content is determined by the amount of CO_2 in the sample. If the sample is analyzed without pretreatment, the Total Carbon (TC) content is determined. Total Inorganic Carbon can be determined by subtraction of the TOC from the TC values.

This method is applicable to the measurement of TOC and Soot in solid samples $\geq$ 100mg/Kg or $\geq$ 0.01%, dry weight. According to the instrument manufacturer, a sample of up to 70% carbon can be analyzed without dilution.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the TOC Analyzer and in the interpretation of the data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

An aliquot of solid sample is dried, homogenized, weighed into a tin capsule, pre-treated with hydrochloric acid and heated to 75°C to convert the inorganic carbon (*i.e.,* carbonate and bicarbonate in the form of CO_2) prior to analysis.

Organic carbon is measured using combustion and a carbon analyzer. The sample, of approximately 5-10 (masses less than 5 mg must be avoided since balance is not calibrated below 5 mg)mg corrected sample weight (a larger aliquot may be used for heterogeneous, or low- carbon samples to improve reproducibility), is oxidized in a pure oxygen environment, introduced into a furnace by a 60-slot Autosampler, then combusted. A greater mass may be weighed if the sample observations indicate low carbon content. As much as 20-25mg may be utilized. The carrier gas (O_2) is combined with the carbon content of the combusted sample to form CO_2. Elements, such as halogens and sulfur, are removed by scrubbing reagents in the combustion zone. A thermal conductivity detector then measures the CO_2. The amount of CO_2 derived from a sample is directly proportional to the concentration of organic carbon material in the sample.

For Soot, the sample will be transferred to an aluminum tin. The tins will be placed in an oven or muffle furnace for 24 hours at 375°C. After sample is in the muffle furnace for 24hrs sample is reduced to a powder with a mortar and pestle, weighed into a tin capsule, then treated with acid then re-dried and analyzed on the TOC Analyzer.

2.1 Method Modifications from Reference

Samples are analyzed using 2 replicates unless otherwise specified by the Client. Hydrochloric acid is substituted for phosphoric acid. The use of this acid has been validated and documented by the laboratory.

3. Reporting Limits

The Practical Quantitation Limit (PQL) or Reporting Limit (RL) is 0.01 (or 100mg/Kg) for solid samples.

4. Interferences

4.1 To determine the TOC/Soot content, the inorganic fractions (carbonate and bicarbonate) must be removed prior to analysis by lowering the pH of the sample to $\leq$ 2. When removing the inorganic carbon, care must be taken in sample pretreatment to minimize the potential of loss of volatile organic carbon present in the sample.

4.2 Maintaining the samples at 4±2°C, and analyzing within the specified holding time minimizes bacterial decomposition and volatilization of the organic material.

4.3 Elements, such as halogens and sulfur, are removed by scrubbing reagents in the combustion zone. Large and/or complex organic molecules such as tannins, lignins, or humic acids may be oxidized slowly. If these compounds are suspected to be present, it is advisable to check the efficiency of the oxidation procedure with a selected representative sample, and adjust the analysis (sample size) as needed.

4.4 Oils from skin, or contact with any organic material, such as plastic containers and rubber tubing, can contaminate samples. Handle the weighing tins with tweezers.

4.5 Sample heterogeneity can be a major source of imprecision in TOC/Soot results. Recommend to clients to perform a client-specific duplicate, to evaluate precision and representativeness of the TOC/Soot results to the site.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. All relevant Material Safety Data Sheets (MSDSs) are kept on file.

Approved PPE, which includes Safety Glasses, Gloves and Lab Coats, must be worn at ***all*** times when handling samples, reagents, chemicals, or when in the vicinity of others handling these items, so that dermal contact is avoided. All standards, reagents and solvents shall be handled using the proper PPE. All flammable solvents must be kept in the flammable storage cabinet, and returned to the cabinet immediately after use. When transporting chemicals, use a secure transporting device and/or secondary outer container. Chemical storage is properly segregated and adequately ventilated to reduce the possibility of hazardous reactions. Chemical storage in work areas shall be kept to a minimum. Storage on bench tops or other work surfaces, except temporary, is not permitted.

All standards and reagents shall be prepared in a hood while using the proper PPE.

Analytical instrumentation poses the unique possibility of exposure to high voltages. Other than the *routine* instrument maintenance, as listed in the front of every Instrument Maintenance Logbook, at no time shall an instrument operator attempt to maintenance an instrument alone, or without the proper training, supervision or instruction. Caution must always be used in the presence of moving parts (autosamplers) and hot surfaces (injection ports).

All additional company safety practices shall be followed at all times as written in the *Chemical Hygiene Plan*.

All TOC standard solutions must be handled with caution. See pages 5-3 through 5-5 of the PE 2400 Series II CHNS/O Analyzer User's Manual for additional and specific Warnings and Precautions.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

A minimum of 5 grams of sample must be collected in a glass jar.

6.2 Sample Preservation

Soil samples must be refrigerated and maintained at 4°±2°C until drying and analysis.

Sediment samples are either processed within 28 days (or can be frozen (for programs with an EPA approved holding time extension for frozen storage) at -15C to -20C until processing.

6.3 Sample Shipping

The *Sample Receipt & Login SOP* (1559) describes how samples are normally shipped or obtained by the laboratory, precautions to be used in opening sample shipments, and sample storage conditions.

6.4 Sample Handling

Soil samples must be refrigerated and maintained at 4°±2°C until drying and analysis.

Sediment samples are either processed within 28 days or can be frozen at -15C to -20C until processing (for programs with an EPA approved holding time extension for frozen storage)

All solid samples must be analyzed within 28 days from the date of collection unless extended hold times are provided in client specific QAPP.

The *Sample Receipt & Login SOP* (1559) describes the responsibilities of sample custody including all proper documentation, verification, and tracking procedures, following Chain of Custody (COC) protocols and sample receipt procedures using the *Sample Receipt Checklist*, which includes the check for proper sample preservation and cooler temperature verification.

Internal COC procedures for sample tracking include the use of sample tracking logbooks. These procedures are also described in the *Sample Receipt & Login SOP* (1559).

7. Equipment and Supplies

7.1 Instrument: Perkin Elmer 2400 Series II CHNS/O Analyzer with computer and PE software

7.2 Detector: Thermal Conductivity

7.3 Mortar and pestle

7.4 Oven: 75 +/- 2 °C

7.5 Muffle Furnace: Capable of maintaining temperature of 375°C

7.6 Dessicator

7.7 Volumetric Flasks: Class-A, various volumes

7.8 Microbalance: Perkin-Elmer, Model AD-6. See the Manufacturer User's Manual, Section 1.2, pages 3-4, for Microbalance and weighing instructions. The Microbalance calibration is verified every day using 5.0mg - 200mg weights, in the range of daily use, and be accurate within +/1%. If the balance needs to be re-calibrated, see the Manufacturer User's Manual SOP, Section 1.1, page 3, for details.

7.9 Tweezers: Anti-Magnetic

7.10 Tin Boats: 5 X 8 mm.

7.11 Polypropylene beakers: 50 mL, for weighing wet samples.

7.12 Glass Beads: Filter Aid 400, High-density; from Perkin Elmer or 3M. Used as the CCB/Method Blank media. Beads must be muffled at 375°C prior to use to remove any trace carbon.

7.13 Gas-tight syringes: Various measuring sizes, including 10μL for direct sample spiking

7.14 Transfer pipets and/or Eppendorf pipettor for delivering HCl: Polypropylene.

7.15 Printer : OKIDATA Microline 320 Turbo-9 Pin or equivalent

Note: All maintenance records including routine upkeep and outside service visits are maintained in the instrument maintenance logbooks. See the Manufacturers User's Manual for detailed troubleshooting instructions and routine maintenance, as needed.

8. Reagents and Standards

Use reagent grade chemicals for all reagents. All reagents and standards are stored at room temperature and expire 6 months from preparation, unless otherwise indicated below.

8.1 Deionized (DI) water: All references to DI water refer to ASTM Type II reagent grade water which has been boiled, cooled and capped to eliminate CO_2. Water must be replaced weekly.

8.2 10% Hydrochloric Acid solution: Add 300mL of concentrated HCl (34-37%) to 700mL of DI water. Mix thoroughly and cool to room temperature before use. Store in a glass bottle and keep at room temperature. This solution is stable for 6 months.

8.3 Potassium Hydrogen Phthalate (KHP), CCV or K-Factor for TOC:

CCV- 10,000mg/L equivalent to 10,000mg/Kg Carbon, or 1.0 % TOC:

Dry potassium hydrogen phthalate (KHP) (primary standard grade) at 110°C for 1 hour. Cool to room temperature in a dessicator. Dissolve 2.128g of KHP (primary standard grade) in ASTM DI water, and dilute to 100mL in a volumetric flask. This standard is good for 6 months, stored at room temperature. Discard solution if discoloration or any signs of bacterial growth are observed.

8.4 Potassium Hydrogen Phthalate (KHP) Spiking Solution/MS/ICV: 10,000mg/L equivalent to 10,000mg/Kg Carbon, or 1.0 % TOC. From a separate source than the solution in Section 8.3, Sigma Aldrich or equivalent.

Dry KHP (primary standard grade) at 110°C for 1 hour. Cool to room temperature in a dessicator. Dissolve 2.128g of KHP (primary standard grade) in ASTM DI water, and dilute to 100mL in a volumetric flask. *Use a separate source or different lot of KHP from that used to prepare the calibration standards.*

This stock may be stored up to six months at room temperature. Discard solution if discoloration or any signs of bacterial growth are observed. Use at 1.0 % TOC concentration for solid samples, and at 10,000mg/L for aqueous samples.

8.5 Solid Standard Reference Materials: Standard Reference Material (SRM) NIST 1944, a separate source from the calibration standard that is certified at the concentration of 4.4% TOC for solid samples. SRM 1650b for Soot, this material has no certified value, however has a reference value of 78% soot carbon. ("Quantification of the Dilute Sedimentary Soot Phase: Implications for PAH Speciation and Bioavailability" published in Environmental Science and Technology, Vol. 31, No. 1, 1997 by Gustafsson, Haghseta, Chan, McFarlane and Gschwend.)

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

9.1.1 Method Blank: A method blank must be analyzed once per every 20 samples or per TOC/Soot batch, whichever is more frequent. The Blank consists of 30-50mg of Filter Aid acidified with HCl and dried. (Section 7.11).

TOC must not be detectable in the method blank at a concentration greater than the reporting limit for the given matrix (Section 3).

Corrective Action: Analysis of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Department Manager, if the samples associated with the out of control method blank are non-detect for TOC/Soot, or if sample TOC/Soot concentrations are greater than 10X the blank levels. In such cases, the sample results are accepted without corrective action for the high method blank and the client is notified in a project narrative associated with the sample results.

9.1.2 Continuing Calibration Blank: A CCB must be analyzed immediately after every CCV. The CCB is 30-50mg of Filter Aid acidified with HCl and dried. (Section 7.10).

The CCB concentration must not be greater than the reporting limit for the given matrix (Section 3).

Corrective Action: Repeat analysis once to see if an analytical error occurred. If the CCB still exceeds the control limits, re-calibrate and/or re-analyze a fresh

blank. All samples associated with the out of control CCB must be re-analyzed (since the last acceptable CCB). Exceptions may be made with approval of the Department Manager if the samples associated with the out of control method blank are non-detect for TOC/Soot or if sample TOC/Soot concentrations are greater than 10X the blank levels. In such cases, the sample results are accepted without corrective action for the high CCB and the client is notified in a project narrative associated with the sample results.

9.1.3 Instrument Blank: An Instrument Blank must be analyzed during Calibration.run2. The Instrument Blank consists of 30-50mg of Filter Aid acidified with HCl and dried at 75°C. (Section 7.10).

9.2 Laboratory Control Sample (LCS)

Laboratory control sample (LCS) must be from a second source or lot number to verify the accuracy of the standard curve. The LCS is analyzed along with the samples. A LCS must be analyzed once per every 20 samples or per TOC batch, whichever is more frequent. For *solid* samples, the LCS is the NIST SRM at 4.4% TOC. The LCS is treated identically as samples with HCl and dried at 75°C.

LCS consists of approximately 5.0mg of SRM 1944 with a true value of 4.4% TOC (Section 8.6). This SRM is NOT evaluated for Soot since no certified concentration for Soot is published.

The acceptable recovery QC range is 75%-125% for the LCS.

Corrective Action: Repeat analysis once to see if an analytical error has occurred. If the LCS recovery is still out of control, re-calibrate and re-analyze the LCS and all associated samples. Samples cannot be analyzed until an acceptable LCS is obtained. Exceptions may be made with approval of the Department Manager if the samples associated with the out of control LCS are also associated with a matrix spike that is in control. This is an acceptable measure of accuracy of the analytical procedures. An explanation of this out of control LCS recovery must be included in the project narrative to the client and the sample data reported with the acceptable MS results as batch QC.

9.3 Initial Calibration Verification (ICV)

Second source analyzed at the beginning of the run. Sample matrix of ICV is a 1.0% carbon solution of KHP.

The acceptable recovery QC range for the ICV is 80-120%.

The **ICV** is prepared by adding 10μL of *Spiking Solution* (Section 8.4), (with a 1% TOC true value). The weight recorded is the Calibration Solution only. (10μL of solution should weigh approximately 10mg).

Corrective Action: Repeat analysis once to see if an analytical error occurred. If the ICV still exceeds the control limits, re-calibrate and re-analyze all samples since last acceptable ICV.

9.4 Continuing Calibration Verification (CCV)

A CCV must be analyzed at a minimum of every 10 burns and at the close of an analytical sequence. This standard monitors instrument performance throughout the duration of the analytical run. Sample matrix of CCV is a 1.0% carbon solution of KHP.

The **CCV** is prepared by adding 10µL of *Calibration Solution* (Section 8.3), (with a 1% TOC true value). The weight recorded is the Calibration Solution only. (10µL of solution should weigh approximately 10mg).

The acceptable recovery QC range for the CCV is 80-120%.

Corrective Action: Repeat analysis once to see if an analytical error occurred. If the CCV still exceeds the control limits, re-calibrate and re-analyze all samples since last acceptable CCV.

9.5 Matrix Spike

A matrix spike must be performed once per 10 samples (10% frequency). When project specifications dictate, a Matrix Spike Duplicate (MSD) may also need to be performed at the same frequency as the MS. Prepare the MS according to Section 8.4 for the given matrix.

A **Matrix Spike** sample is spiked with 10µL of *Spiking Solution* (Section 8.4), at a 1.0 % TOC true value. The weight recorded is the sample weight prior to spiking.

The acceptable recovery QC range is 75%-125% for the MS/MSD. Calculate the %RPD as in Section 9.6 when analyzing a MS/MSD pair. The acceptable %RPD is $\leq$ 25% for solid samples.

Corrective Action: If sample concentration exceeds 4X the spike amount, narrate. Repeat analysis once to see if an analytical error has occurred. If the % recovery or %RPD still exceeds the criteria and the LCS is compliant; include a project narrative with the results to the client noting that there may be potential matrix effects on the accuracy or precision of the TOC/Soot results as evidenced by matrix spike recovery or %RPD outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses (matrix duplicate) must be performed once per 20 samples (5% frequency).

Acceptable relative percent difference (RPD) of duplicates is $\leq$ 25%. Acceptance criterion is not applicable to sample concentrations less than 5X the reporting limit. Calculate RPD as follows:

$$\%RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:

R1 = highest result
R2 = lower result

Corrective Action: Repeat analysis once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to the client noting that there may be potential matrix effects on the precision of the TOC/Soot results as evidenced by the matrix duplicate RPD exceedance.

9.7 Method-specific Quality Control Samples

High level Initial Calibration Verification (HLICV - linearity check):

This sample consists of 4% carbon solution (high calibration point).

Aliquot 50 µL using a syringe into a tin boat, record weight and analyze. (Equivalent to a 5 mg sample containing 40% TOC).

The acceptable recovery QC range for the HLICV is 80-120%.

Corrective Action: Repeat analysis once to see if an analytical error occurred. If the HLICV still exceeds the control limits, re-calibrate.

9.8 Method Sequence

- Calibration Curve generation
- ICV
- ICB
- HLICV
- Method Blank
- LCS
- Laboratory Duplicate
- Matrix Spike
- Samples 1 – 8
- CCV
- CCB
- Samples 9 – 18
- CCV
- CCB

10. Procedure

10.1 Equipment Set-up

Samples are prioritized by the Department Manager for analysis based on hold time and client due date. Section 15.5 outlines the steps for final TOC reporting that will contain the sample analysis final results.

The analyst initials, date, sample ID #'s, sample weights (solids), or volumes (aqueous), of all Standards, QC samples and field samples, are entered into the TOC logbook. Include the standard, LCS and MS IDs and concentrations. Use TOC weight spreadsheet to record all information.

10.1.1 Pretreatment for Total Organic Carbon:

This procedure is used to remove the inorganic compounds in the sample prior to the determination of Total Organic Carbon. If Total Carbon is to be determined, addition of the acid solution is omitted.

10.1.1.1 Aliquot 10-15g of wet solid sample into a weighing vessel. Remove shells, rocks and other large non-representative materials with tweezers. Record weight of vessel and vessel + wet sample.

10.1.1.2 Heat for 4 hours at 75 +/- 2 °C. Return sample to oven if additional drying time is required(in ½ hour increments) Record time in and out of oven. Cool sample to room temperature in dessicator. Record weight of vessel + dry sample.

10.1.1.3 Add 10% HCl (Section 8.2) dropwise to dry sample to induce effervescence (removal of inorganic carbon). Do not use excessive acid.. Only add enough acid to exhaust the effervescence. In lieu of additional acid, use DI water to wet the sample in order to form a slurry if necessary, to distribute the acid throughout the sample. Record the volume of HCl used for each sample.

10.1.1.4 Return the sample to the oven. Heat for 4 hours at 75 +/- 2 °C. Return sample to oven if additional drying time is required(in ½ hour increments) Record time in and out of oven. Cool to room temperature in dessicator. Record final weight of dry sample + vessel. The change in weight of dry sample after acidification is used to adjust the weight of the aliquots used for analysis.

10.1.1.5 Take a sub-aliquot of approximately 2-3 g of dry sample and perform moisture content using SM2540D. The corrected sample weight (CSW, see Section 11.4) is corrected using this result (see Section 11.4 for calculations)

10.1.1.6 Grind sample with mortar and pestle to homogenize. Place ground sample into a 4 mL vial.

10.1.1.6.1 If Soot analysis is required, take a portion of the dried sample aliquot(prior to acidification), approximately 5-10 gm, and place into a muffle furnace at 375°C for 24 hours. Once the sample aliquot is cooled to room temperature, proceed to the acid addition step, Section 10.1.1.3. Samples for Soot are read analytically in the same way as Routine TOC.

10.1.1.7 Weigh (and record weight of) each replicate into a tin capsule (see Section 10.3 for appropriate weight range). Record the weight and use the TOC weight spreadsheet to calculate the corrected sample weight (CSW) which is entered into the analyzer.

10.2 Initial Calibration

On the accompanying computer, or "PC", Perkin Elmer software has been loaded to collect and save sample and standard analysis data. Create a file to store the data from each day's analytical sequence by clicking on the PE 2400 icon, go to "new" and "save" the file "as" the name of the date of the analysis (*i.e.,* 060503.chn). Data from each standard or sample analysis will be automatically stored in this file for future reference

10.2.1 Follow the instrument Manufacturers User's Manual for instrument set up and see Sections below for an outline of the initial, daily calibration procedure.

See the Manufacturers User's Manual, page 5-69, for the default instrument conditions when in the CHN operating mode.

10.2.2 *Routine* TOC/Soot Calibration Method:

TOC analyses use K-Factors (3 repeat analyses of a 1.0% TOC standard, Section 8.3) and Blanks, calculated and averaged by the instrument when the calibration is being performed. Follow the instrument Manufacturer's User's Manual, page 4-67. Calibration of the instrument using K-Factors and Blanks is performed once per day of analysis.

Load the Autosampler (Section 10.3.2) for combustion to analyze Blanks and Standards in the order listed below. All "Blanks" and "K-Factors" are prepared in the same way. Analyze the standards following the procedure in Section 10.3.

- **Instrument "Primer"** (~3.5mg of SRM 1944, with true value of 4.4% TOC) only if new column.
- **Blank 1** (30-50mg of Filter Aid) only if new column
- **K-Factor 1** (10µl of *Calibration Solution*, Section 10.3,)
  The weight recorded is the Calibration Solution, only.
- **Blank 2** (30.0mg of Filter Aid)
- **K-Factor 2** (10µl of *Calibration Solution*, Section 10.3,)
  The weight recorded is the Calibration Solution, only.
- **K-Factor 3** (10µl of *Calibration Solution*, Section 10.3,)
  The weight recorded is the Calibration Solution, only.

The K-Factors must reproduce from the mean value within +/- 15% of known K factor for each instrument for carbon. The calculation involves the comparison among the latest K-Factor run, the prior K-Factor run, and the current running average K-Factor. An "out of tolerance" message will be displayed if this criterion is not met.

The ICV/LCS and Method Blank are prepared to run immediately following the third K-Factor, given the instrument is “in tolerance”.

Corrective Action: If, at any time during the 6–step process of calibration, one of the K-Factors or Blanks fail, repeat analysis of failing sample.

10.3 Equipment Operation and Sample Processing

Prepare a Calibration per Section 10.2.

An ICV/ICV and LCS/Method Blank are analyzed immediately following the third K-Factor of the Routine Calibration, provided the instrument is “in tolerance”. The LCS and method blank must be run with the same number of replicates that are required for the samples in the run.

Remove all pretreated samples from the oven, and allow to cool in a dessicator.

Weigh approximately 5-10mg (to an accuracy of 0.01 mg) (masses less than 5 mg must be avoided since balance is not calibrated below 5 mg) of sample into a tin boat using the Perkin-Elmer Microbalance, Model AD-6, which is linked to the instrument. Samples that are known to be high in TOC may use a lesser sample aliquot. Samples where it is observed that the TOC may be low, weight as much as 20-25mg. Record the weights in the TOC/Soot logbook. Matrix Duplicate and Matrix Spike samples are weighed as separate samples, with their own replicates. As noted in Section 10.1.1.6, use the TOC weight spreadsheet to correct the sample weights for all replicates and re-analyses.
Any observations regarding the sample composition must be noted in the TOC/Soot logbook such as: sandy, contains shells or rocks, contains grass or roots, is light/white in coloration, etc.

Analyze a CCV/CCB (Section 9.4 and 9.1.3) after every 10 “burns”, and at the end of the analytical sequence.

If following the *Lloyd Kahn* Calibration method, analyze the first set of batch QC samples, the ICV/LCS and Method Blank, followed by field samples with a CCV/CCB analyzed every 10 “burns”, and at the end of the analytical sequence.

10.3.1 Multiple Sample Instrument Loading:

10.3.1.1 Each tin boat is carefully folded with anti-magnetic tweezers, and placed into its respective holding tray position. (See the Manufacturer User’s Manual SOP, Section 1.2, page 4, for tin boat folding details.) Weights, sample tray position and Autosampler location are recorded in the TOC/Soot logbook. The analyst must be very careful when transferring samples. Documentation of sample consistency is very important. See Figure 1 for an example logbook page.

10.3.1.2 Adjust the Autosampler tray to begin sampling at auto-slot #1 and carefully transfer the folded tins containing the samples to the correct auto-slot, using tweezers. All Instrument Calibration Standards (Blanks, K-Factors and/or *Lloyd Kahn* Calibration Standards) are loaded first. Immediately following will be the first batch QC samples, the ICV/LCS and Method Blank for the run, followed by field samples. Rotate the Autosampler in a *counter clockwise* manner.

10.3.1.3 A maximum of 60 tins can be held on the Autosampler at one time. The instrument only allows for 100 total samples (including calibration/blank samples) to be programmed for any given run.

10.3.2 Sample Analysis

10.3.2.1 Select the *Auto Run* option on the keypad on the instrument. The following menu appears:

AUTO RUN NO. 1
1B 2K 3S
where: B = Blank
K = K-Factor
S = Sample

Assuming the *Instrument Standardization* occupies auto-slots 1-6, Enter "3" for Sample, then select "Enter". Enter the weight of the "Primer" Standard (position 1) and select "Enter".

10.3.2.2 You will be prompted for AUTO RUN NO. 2 as in Section 10.3.3.1, except now there is a function "4NP" which allows the analyst to stop/erase everything by selecting this feature.

- Enter "1" for the Blank. The weight will not be asked for.
- Enter "2" for the first K-Factor, then select "Enter". Enter the weight of your K-Factor (the standard weight should be ~ 10mg). Select "Enter".
- Enter "1" for the second Blank, then "Enter".
- Enter "2" for the second K-Factor, then "Enter". Enter the weight, then "Enter".
- Enter "2" for the third K-Factor, then "Enter". Enter the weight, then "Enter".

10.3.2.3 All remaining sample weights are added by entering "3" for Sample, and then by entering the sample weight. Continue adding all information to the run sequence, using the keypad. When entering sample identifications, and letters are necessary, refer to the Manufacturer's User's Manual, page 3-4, for instructions for converting numbers to letters.

10.3.2.4 When all sequence information is entered and complete, select "Start". The analyses will begin. The computer will continue to display the "AUTO RUN" menu during sample analysis.

10.3.2.5 The Standard and Sample data prints out as it is collected on the line printer, and is also collected on the linked computer using the PE 2400 software. The data on the computer is saved in a file named as the date of the analysis (*i.e.,* 060503.chn) for future reference, as noted in 10.2.

10.3.2.6 When all analyses are complete, refer to the Manufacturers User's Manual SOP, Section 3.1, page 7, for instructions to "Shut Down" the instrument.

10.3.2.7 Evaluate all batch QC samples first, to confirm or exclude the need for re-analyses. The ICV/LCS/HLICV, Method Blank, CCB/CCV samples must meet the acceptance criteria in Section 9. Follow the corrective action for any QC failures.

10.3.2.8 Evaluate the field samples, LCS/SRM, and MS/Duplicates next. If replicate "burns" vary by more than 30%, re-weigh two additional aliquots similar to the initial weights used, and re-analyze the sample. For a 2-replicate analysis, this provides a total of four burns/samples. If the second set of replicates also has RPD > 30%, report the first set. If the re-analysis has an RPD which passes, report the re-analysis. Three and four replicate analysis may be requested, and the average of the multiple burns is reported, unless otherwise indicated by the client. Analysts should use judgement in cases where there may have been an error with the autosampler or other factor (incorrect loading, etc.) which may have led to RPD failure. The Lloyd Kahn method requires that quadruplicate analysis be performed on one sample per batch of up to 20 samples and that the RSD must meet precision criteria of 3 times the standard deviation of laboratory generated sample data. One sample per batch of up to 20 must be analyzed in quadruplicate. RSD criteria is 40%. This value was generated using 30 data points of quadruplicate analysis of various concentrations and multiple instruments. The RSD was calculated for each sample in quadruplicate, and the RSD limit is based on 3 times the average standard deviation. Failure to meet this criteria requires that the entire batch be re-analyzed.

10.4 Continuing Calibration

A CCV must be analyzed as outlined in Section 10.3.3 at a minimum of every 10 burns and at the close of an analytical sequence.

10.5 Preventive Maintenance

The reduction column is changed after every 100-200 runs. The combustion column is replaced as needed.

11. Data Evaluation, Calculations and Reporting

11.1 Procedures for data and record management for TOC/Soot analysis must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP, 08-01. All records are stored in such a manner as to be safe and accessible for at least 10 years.

11.2 The relevant TOC/Soot laboratory notebooks must follow the specifications in the *Laboratory Notebook Usage* Work Instruction 1556, and all record keeping and document control practices.

11.3 Sample results for TOC/Soot are directly reported from the instrument printout.

11.4 Calculations: The following calculations are applied by the instrument when determining the percentage of Total Organic Carbon or Soot: Note: sample weights are corrected for sample mass changes due to acidification prior to analysis and entered into the instrument software as the corrected weights, therefore, no correction is required after analysis.

Corrected Sample Weight(CSW): Weight that is entered at the instrument, corrected for sample mass change after acidification.

CSW = aliquot wt (mg) * total sample dry wt (g) (prior to acidification) * dry wt.@ (75°C)
Total sample dry wt (g) (post acidification) *dry wt.@(105°C)

Carbon Blank (CB): Used to make all other determinations, namely, K-Factor and weight percent calculations. When run alternately with samples, the instrument averages the blank values.

Carbon Blank (CB) = Carbon Read – Nitrogen Read

Carbon K-Factor (C KF): K-Factor, or detector calibration factor, is determined when a known standard is analyzed to calibrate the analyzer in terms of micrograms of carbon. This calibration factor is then used to determine unknowns.

C KF = ((CR – NR) – CB x 100) / (CSW x C Theory Wt. %)

where: CR = Carbon Read
NR = Nitrogen Read
CB = Carbon Blank
CSW= Corrected Sample Weight (mg) (using corrected weight recorded in Section 10.3)
C Theory Wt % = 1% (based on the concentration of the K-Factor standard)

Carbon (mg/Kg):

Carbon (mg/Kg) = (((CR – NR) – CB) x 1000) / (SW x C KF)

where: CR = Carbon Read
NR = Nitrogen Read
CB = Carbon Blank
CSW = Corrected Sample Weight (mg)
C KF = Carbon K-Factor

To convert to % C divide result in mg/Kg by 10000

Example Calculation of Percent TOC/Soot from the instrument, converted to mg of TOC/Soot:

If the result from the instrument is 1.083% and the sample weight is 10.76mg:

1.083% = 100 x [**X** / 10.76mg] (to solve for **X,** divide each side by 100)
0.01083 = **X** / 10.76mg (multiply each side by 10.76mg)
0.116mg = **X**

11.5 Reporting Results

The following procedures must be followed for reporting of TOC/Soot results:

Go to the next page in the bound TOC/Soot logbook.

Retain all computer printouts for the standard and sample analyses of this sequence.

Print the sequence from the computer and review it to make sure that no weight or sample ID transcription errors were made.

Calculate the recoveries for the QC samples (IVC/LCS, CCV, MS and RPD between the duplicate samples) and ensure the Method Blank and CCB meet the criteria. Remember any "QC Failures" require corrective action. See Section 9.0 or 12.0.

After reviewing the final logbook entries, make a copy of the logbook page for secondary review.

Go to the LIMS and "batch" and "associate" the set of QC samples and field samples that were just analyzed.

Enter the Final Results into the LIMS report sheet and check for transcription errors.

Transfer all associated sample paperwork to the Department Manager for the second level review and approval.

All TOC/Soot results are reportable without qualification if analytical holding times are met, preservation (including pH and cooler temperatures) are met, all QC criteria defined in the table below are met, and matrix interference was not suspected during analysis of the TOC/Soot samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-analysis.

QC Parameter	Acceptance Criteria
Initial Calibration Curve, *Lloyd Kahn*	r $\geq$ 0.995 and slope ± 10% the historical curves
Initial Calibration Verification	80-120% of True Value
Linearity check (high level ICV)	80-120% of True Value
Method Blank	< reporting limit for matrix
Laboratory Control Sample	75-125% R
Matrix Duplicate	25%RPD for results <5x reporting limit
Replicate burns (each sample)	30% RPD
Quadruplicate analysis(1 per batch)	< 40 % RSD
Matrix Spike	75-125% R
Matrix Spike Duplicate if requested	75-125%R/25%RPD
Continuing Calibration Verification	80-120% of True Value
Continuing Calibration Blank	< reporting limit for matrix

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9 outlines sample batch QC acceptance criteria. If non-compliant TOC/Soot results are to be reported, the Department Manager and/or the Laboratory Director, and the QA Manager must approve the reporting of these results. The laboratory Project Manager is notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

The *Hazardous Waste and Sample Disposal* SOP (1797) must be referenced for disposal of used standards, solvents, acids, reagents or other chemicals.

Once satisfactory TOC results have been generated, the sample containers must be scanned back into LIMS and returned to the appropriate sample refrigerator and held for 30 days.

All waste generated must be transferred to the waste disposal area. Dispose all solid samples in solid waste container. Aqueous samples must be poured into a 55-gallon drum marked acidic/non-chlorinated waste.

Refer to the *Chemical Hygiene Plan* and the *Hazardous Waste & Sample Disposal SOP* (1797) for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

PE 2400 Series II CHNS/O Analyzer User's Manual

Sample Receipt & Login SOP (1559)

Perkin-Elmer, Model AD-6 Manufacturer User's Manual

Document Control SOP 1729

Laboratory Notebook Usage Work Instruction (WI 1556)

SOP/1732 MDL Generation

SOP/1739 IDC Generation

Hazardous Waste & Sample Disposal SOP (1797)

16. Attachments

None.

Total Organic Carbon and Soot in Soil, Sediment

References: USEPA, Region II, Lloyd Kahn, "Determination of Total Organic Carbon in Sediment", July 27, 1988.

Perkin Elmer, "PE 2400 Series II CHNS/O Analyzer User's Manual," The Perkin-Elmer Corporation, April 1998.

USEPA, "Method 9060A Total Organic Carbon," in Test Methods for Evaluating Solid Waste, SW846, Revision 1 November 2004.

"Quantification of the Dilute Sedimentary Soot Phase: Implications for PAH Speciation and Bioavailability" published in Environmental Science and Technology, Vol. 31, No. 1, 1997 by Gustafsson, Haghseta, Chan, McFarlane and Gschwend.

"Reinterpreting Literature Sorption Data Considering Both Absorption into Organic Carbon and Adsorption onto Black Carbon" published in Environmental Science and Technology, Vol. 37, No. 1, 2003 by Accardi-Dey and Gschwend

1. Scope and Application

Matrices: This method is applicable for solid samples (soils, sediments, sludges).

Definitions: Refer to Alpha Analytical Quality Manual.

The organic carbon in a sample consists of a variety of organic compounds in various oxidation states. Some of these compounds may be oxidized by biological or chemical processes and can be measured by the Biochemical Oxygen Demand or Chemical Oxygen Demand tests. To measure the amount of organically bound carbon, the organic carbon molecules are broken down into single carbon units and converted into a form that can be measured quantitatively.

The PE 2400 Series II CHNS/O Analyzer can simultaneously determine carbon (C), hydrogen (H), nitrogen (N), sulfur (S), and oxygen (O) in organic material. In the CHN mode, the PE 2400 Series II CHNS/O Analyzer uses a combustion method to convert the sample elements to simple gases (CO_2, H_2O, and N_2). The resulting gases are homogenized and controlled to exact conditions of pressure, temperature, and volume. The homogenized gases are allowed to de-pressurize through a column where they are separated in a stepwise steady-state manner and detected as a function of their thermal conductivity and reported as a percentage for solid samples. Treated with hydrochloric acid prior to analysis, the Total Organic Carbon (TOC) content is determined by the amount of CO_2 in the sample. If the sample is analyzed without pretreatment, the Total Carbon (TC) content is determined. Total Inorganic Carbon can be determined by subtraction of the TOC from the TC values.

This method is applicable to the measurement of TOC and Soot in solid samples $\geq$ 500mg/Kg or $\geq$ 0.05%, dry weight. According to the instrument manufacturer, a sample of up to 70% carbon can be analyzed without dilution.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory

personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the TOC Analyzer and in the interpretation of the data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

An aliquot of solid sample is dried, homogenized, weighed into a tin capsule, pre-treated with hydrochloric acid and heated to 75°C to convert the inorganic carbon (*i.e.,* carbonate and bicarbonate in the form of CO_2) prior to analysis.

Organic carbon is measured using combustion and a carbon analyzer. The sample, of approximately 5-10 mg corrected weight(masses less than 2 mg must be avoided since balance is not calibrated below 2 mg) Aa larger aliquot may be used for heterogeneous, or low- carbon samples to improve reproducibility, is oxidized in a pure oxygen environment, introduced into a furnace by a 60-slot Autosampler, then combusted. A greater mass may be weighed if the sample observations indicate low carbon content. As much as 20-25mg may be utilized. The carrier gas (O_2) is combined with the carbon content of the combusted sample to form CO_2. Elements, such as halogens and sulfur, are removed by scrubbing reagents in the combustion zone. A thermal conductivity detector then measures the CO_2. The amount of CO_2 derived from a sample is directly proportional to the concentration of organic carbon material in the sample.

For Soot, the sample will be transferred to an aluminum tin. The tins will be placed in an oven or muffle furnace for 24 hours at 375°C. After sample is in the muffle furnace for 24hrs sample is reduced to a powder with a mortar and pestle, weighed into a tin capsule, then treated with acid then re-dried and analyzed on the TOC Analyzer.

2.1 Method Modifications from Reference

Samples are analyzed using 2 replicates unless otherwise specified by the Client. Hydrochloric acid is substituted for phosphoric acid. The use of this acid has been validated and documented by the laboratory.

3. Reporting Limits

The Practical Quantitation Limit (PQL) or Reporting Limit (RL) is 0.05% (or 500mg/Kg) for solid samples.

4. Interferences

4.1 To determine the TOC/Soot content, the inorganic fractions (carbonate and bicarbonate) must be removed prior to analysis by lowering the pH of the sample to $\leq$ 2. When removing the inorganic carbon, care must be taken in sample pretreatment to minimize the potential of loss of volatile organic carbon present in the sample.

4.2 Maintaining the samples at 4±2°C and analyzing within the specified holding time minimizes bacterial decomposition and volatilization of the organic material.

4.3 Elements, such as halogens and sulfur, are removed by scrubbing reagents in the combustion zone. Large and/or complex organic molecules such as tannins, lignins, or humic acids may be oxidized slowly. If these compounds are suspected to be present, it is advisable to check the efficiency of the oxidation procedure with a selected representative sample and adjust the analysis (sample size) as needed.

4.4 Oils from skin, or contact with any organic material, such as plastic containers and rubber tubing, can contaminate samples. Handle the weighing tins with tweezers.

4.5 Sample heterogeneity can be a major source of imprecision in TOC/Soot results. Recommend to clients to perform a client-specific duplicate, to evaluate precision and representativeness of the TOC/Soot results to the site.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The use of laboratory equipment and chemicals exposes the analyst to several potential hazards. Good laboratory techniques and safety practices shall be followed at all times. All relevant Material Safety Data Sheets (MSDSs) are kept on file.

Approved PPE, which includes Safety Glasses, Gloves and Lab Coats, must be worn at all times when handling samples, reagents, chemicals, or when in the vicinity of others handling these items, so that dermal contact is avoided. All standards, reagents and solvents shall be handled using the proper PPE. All flammable solvents must be kept in the flammable storage cabinet and returned to the cabinet immediately after use. When transporting chemicals, use a secure transporting device and/or secondary outer container. Chemical storage is properly segregated and adequately ventilated to reduce the possibility of hazardous reactions. Chemical storage in work areas shall be kept to a minimum. Storage on bench tops or other work surfaces, except temporary, is not permitted.

All standards and reagents shall be prepared in a hood while using the proper PPE.

Analytical instrumentation poses the unique possibility of exposure to high voltages. Other than the *routine* instrument maintenance, as listed in the front of every Instrument Maintenance Logbook, at no time shall an instrument operator attempt to maintenance an instrument alone, or without the proper training, supervision or instruction. Caution must always be used in the presence of moving parts (autosamplers) and hot surfaces (injection ports).

All additional company safety practices shall be followed at all times as written in the *Chemical Hygiene Plan*.

All TOC standard solutions must be handled with caution. See pages 5-3 through 5-5 of the PE 2400 Series II CHNS/O Analyzer User's Manual for additional and specific Warnings and Precautions.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

A minimum of 5 grams of sample must be collected in a glass jar.

6.2 Sample Preservation

Soil samples must be refrigerated and maintained at 4 ± 2 °C until drying and analysis.

Sediment samples are either processed within 28 days (where citing 9060 hold time reference) (or 14 days for Lloyd Kahn) or can be frozen (for programs with an EPA approved holding time extension for frozen storage) at -15 °C to -20 °C until processing.

6.3 Sample Shipping

The *Sample Receipt & Login SOP* (1559) describes how samples are normally shipped or obtained by the laboratory, precautions to be used in opening sample shipments, and sample storage conditions.

6.4 Sample Handling

All solid samples (NOT requiring the *Lloyd Kahn* Calibration method) must be analyzed within 28 days from the date of collection unless extended hold times are provided in client specific QAPP. **Note:** If samples require the *Lloyd Kahn* Calibration method, the hold time is 14 days unless otherwise indicated by client.

The *Sample Receipt & Login SOP* (1559) describes the responsibilities of sample custody including all proper documentation, verification, and tracking procedures, following Chain of Custody (COC) protocols and sample receipt procedures using the *Sample Receipt Checklist*, which includes the check for proper sample preservation and cooler temperature verification.

Internal COC procedures for sample tracking include the use of sample tracking logbooks. These procedures are also described in the *Sample Receipt & Login SOP* (1559).

7. Equipment and Supplies

7.1 Instrument: Perkin Elmer 2400 Series II CHNS/O Analyzer with computer and PE software

7.2 Detector: Thermal Conductivity

7.3 Mortar and pestle

7.4 Oven: 75 ± 2 °C

7.5 Muffle Furnace: Capable of maintaining temperature of 375°C

7.6 Desiccator

7.7 Volumetric Flasks: Class-A, various volumes

7.8 Microbalance: Perkin Elmer, Model AD-6. See the Manufacturer User's Manual, Section 1.2, pages 3-4, for Microbalance and weighing instructions. The Microbalance calibration is verified every day using 5.0mg - 200mg weights, in the range of daily use, and be accurate within ±1%. If the balance needs to be re-calibrated, see the Manufacturer User's Manual SOP, Section 1.1, page 3, for details.

7.9 Tweezers: Anti-Magnetic

7.10 Tin Boats: 5 X 8 mm.

7.11 Polypropylene beakers: 50 mL, for weighing wet samples.

7.12 Glass Beads: Filter Aid 400, High-density; from Perkin Elmer or 3M. Used as the CCB/Method Blank/LFB media. Beads must be muffled at 375°C prior to use to remove any trace carbon.

7.13 Gas-tight syringes: Various measuring sizes, including 10µL for direct sample spiking

7.14 Transfer pipets and/or Eppendorf pipettor for delivering HCl: Polypropylene.

Note: All maintenance records including routine upkeep and outside service visits are maintained in the instrument maintenance logbooks. See the Manufacturers User's Manual for detailed troubleshooting instructions and routine maintenance, as needed.

8. Reagents and Standards

Use reagent grade chemicals for all reagents. All reagents and standards are stored at room temperature and expire 6 months from preparation, unless otherwise indicated below.

8.1 Deionized (DI) water: All references to DI water refer to ASTM Type II reagent grade water which has been boiled, cooled and capped to eliminate CO_2. Water must be replaced weekly.

8.2 10% Hydrochloric Acid solution: Add 300mL of concentrated HCl (34-37%) to 700mL of DI water. Mix thoroughly and cool to room temperature before use. Store in a glass bottle and keep at room temperature. This solution is stable for 6 months.

8.3 Potassium Hydrogen Phthalate (KHP), CCV or K-Factor for TOC:

CCV- 10,000mg/L equivalent to 10,000mg/Kg Carbon, or 1.0 % TOC:

Dry potassium hydrogen phthalate (KHP) (primary standard grade) at 110°C for 1 hour. Cool to room temperature in a desiccator. Dissolve 2.128g of KHP (primary standard grade) in ASTM DI water and dilute to 100mL in a volumetric flask. This standard is good for 6 months, stored at room temperature. Discard solution if discoloration or any signs of bacterial growth are observed.
Calibration Standards-
Dry KHP (primary standard grade) at 110°C for 1 hour. Cool to room temperature in a desiccator.

- 0.1% (1000mg/Kg) – Add 10 mL of the 1.0% calibration solution (see below) to a 100 mL volumetric flask. Dilute to 100 mL using ASTM DI water.

- 0.5% (5000 mg/Kg) - Dissolve 1.064g of KHP (primary standard grade) in ASTM DI water and dilute to 100mL in a volumetric flask. This standard is good for 6 months, stored at room temperature. Discard solution if discoloration or any signs of bacterial growth are observed.

- 1.0% (10000mg/Kg) - Dissolve 2.128g of KHP (primary standard grade) in ASTM DI water and dilute to 100mL in a volumetric flask. This standard is good for 6 months, stored at room temperature. Discard solution if discoloration or any signs of bacterial growth are observed.

- 2.0% (20000mg/Kg) - Dissolve 4.256g of KHP (primary standard grade) in ASTM DI water and dilute to 100mL in a volumetric flask. This standard is good for 6 months, stored at room temperature. Discard solution if discoloration or any signs of bacterial growth are observed.

- 4.0% (40000mg/Kg) - Dissolve 8.512g of KHP (primary standard grade) in ASTM DI water and dilute to 100mL in a volumetric flask. This standard is good for 6 months, stored at room temperature. Discard solution if discoloration or any signs of bacterial growth are observed.

8.4 Potassium Hydrogen Phthalate (KHP) Spiking Solution/MS/ICV: 10,000mg/L equivalent to 10,000mg/Kg Carbon, or 1.0 % TOC. From a separate source than the solution in Section 8.3, Sigma Aldrich or equivalent.

Dry KHP (primary standard grade) at 110°C for 1 hour. Cool to room temperature in a desiccator. Dissolve 2.128g of KHP (primary standard grade) in ASTM DI water and dilute to 100mL in a volumetric flask. *Use a separate source or different lot of KHP from that used to prepare the calibration standards.*

This stock may be stored up to six months at room temperature. Discard solution if discoloration or any signs of bacterial growth are observed. Use at 1.0 % TOC concentration for solid samples, and at 10,000mg/L for aqueous samples.

8.5 Solid Standard Reference Materials (when requested): Standard Reference Material (SRM) NIST 1944, a separate source from the calibration standard that is certified at the concentration of 4.4% TOC for solid samples. SRM 1650b for Soot, this material has no certified value, however, has a reference value of 78% soot carbon. ("Quantification of the Dilute Sedimentary Soot Phase: Implications for PAH Speciation and Bioavailability" published in Environmental Science and Technology, Vol. 31, No. 1, 1997 by Gustafsson, Haghseta, Chan, McFarlane and Gschwend.)

The recovery range for SRM 1944 --3.3 - 5.5%C and SRM1650(soot) 80%-120%C (low 62.40%-high 93.60%). Corrective action for recoveries outside of the expected range: re-run once, if still failing criteria, recalibrate and rerun after confirming the calibration (passing ICV,ICB). If the SRM continues to fail, contact the department supervisor or manager.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

9.1.1 Method Blank: A method blank must be weighed out alongside samples during the pretreatment process once per every 20 samples or per TOC/Soot batch, whichever is more frequent. The Blank consists of 10-15g of Filter Aid that has been dried in the muffle furnace to remove any trace amounts of Carbon

TOC must not be detectable in the method blank at a concentration greater than the reporting limit for the given matrix (Section 3).

Corrective Action: Analysis of the method blank and all associated samples must be performed until the blank is in control. Samples cannot be analyzed until an acceptable method blank analysis is obtained. Exceptions may be made with approval of the Department Manager, if the samples associated with the out-of-control method blank are non-detect for TOC/Soot, or if sample TOC/Soot concentrations are greater than 10X the blank levels. In such cases, the sample results are accepted without corrective action for the high method blank and the client is notified in a project narrative associated with the sample results.

9.1.2 **Continuing Calibration Blank:** A CCB must be analyzed immediately after every CCV. The CCB is 30-50mg of Filter Aid acidified with HCl and dried. (Section 7.10).

The CCB concentration must not be greater than the reporting limit for the given matrix (Section 3).

Corrective Action: Repeat analysis once to see if an analytical error occurred. If the CCB still exceeds the control limits, re-calibrate and/or re-analyze a fresh blank. All samples associated with the out-of-control CCB must be re-analyzed (since the last acceptable CCB). Exceptions may be made with approval of the Department Manager if the samples associated with the out-of-control method blank are non-detect for TOC/Soot or if sample TOC/Soot concentrations are greater than 10X the blank levels. In such cases, the sample results are accepted without corrective action for the high CCB, and the client is notified in a project narrative associated with the sample results.

9.1.3 **Instrument Blank:** An Instrument Blank must be analyzed during Calibration.run2. The Instrument Blank consists of 30-50mg of Filter Aid acidified with HCl and dried at 75°C. (Section 7.10).

9.2 Laboratory Control Sample (LCS)

Laboratory control sample (LCS) must be from a second source or lot number to verify the accuracy of the standard curve. The LCS is analyzed along with the samples. A LCS must be analyzed once per every 20 samples or per TOC batch, whichever is more frequent. The LCS is an aliquot of the filter aid prepared alongside the samples and spiked at a known value. The recovery limits are 80%-120%.

Corrective Action: Repeat analysis once to see if an analytical error has occurred. If the LCS recovery is still out of control, re-calibrate and re-analyze the LCS and all associated samples. Samples cannot be analyzed until an acceptable LCS is obtained.

9.3 Initial Calibration Verification (ICV)

Second source analyzed at the beginning of the run. Sample matrix of ICV is a 1.0% carbon solution of KHP.

The acceptable recovery QC range for the ICV is 80-120%.

The **ICV** is prepared by adding 10µL of *Spiking Solution* (Section 8.4), (with a 1% TOC true value). The weight recorded is the Calibration Solution only. (10µL of solution should weigh approximately 10mg).

Corrective Action: Repeat analysis once to see if an analytical error occurred. If the ICV still exceeds the control limits, re-calibrate and re-analyze all samples since last acceptable ICV.

9.4 Continuing Calibration Verification (CCV)

A CCV must be analyzed at a minimum of every 10 burns and at the close of an analytical sequence. This standard monitors the instrument's performance throughout the duration of the analytical run. Sample matrix of CCV is a 1.0% carbon solution of KHP.

The CCV is prepared by adding 10µL of *Calibration Solution* (Section 8.3), (with a 1% TOC true value). The weight recorded is the Calibration Solution only. (10µL of solution should weigh approximately 10mg).

The acceptable recovery QC range for the CCV is 80-120%.

Corrective Action: Repeat analysis once to see if an analytical error occurred. If the CCV still exceeds the control limits, re-calibrate and re-analyze all samples since last acceptable CCV.

9.5 Matrix Spike

A matrix spike must be performed once per 20 samples (5% frequency). When project specifications dictate, a Matrix Spike Duplicate (MSD) may also need to be performed at the same frequency as the MS. Prepare the MS according to Section 8.4 for the given matrix.

A **Matrix Spike** sample is spiked with 10µL of *Spiking Solution* (Section 8.4), at a 1.0 % TOC true value. The weight recorded is the sample weight prior to spiking.

The acceptable recovery QC range is 75%-125% for the MS/MSD. Calculate the %RPD as in Section 9.6 when analyzing a MS/MSD pair. The acceptable %RPD is $\leq$ 25% for solid samples.

Corrective Action: If sample concentration exceeds 4X the spike amount, narrate. Repeat analysis once to see if an analytical error has occurred. If the % recovery or %RPD still exceeds the criteria and the LCS is compliant; include a project narrative with the results to the client noting that there may be potential matrix effects on the accuracy or precision of the TOC/Soot results as evidenced by matrix spike recovery or %RPD outside of QC limits.

9.6 Laboratory Duplicate

Duplicate analyses (matrix duplicate) must be performed once per 20 samples (5% frequency).

Acceptable relative percent difference (RPD) of duplicates is $\leq$ 25%. Acceptance criterion is not applicable to sample concentrations less than 5X the reporting limit. Calculate RPD as follows:

$$\%RPD = \frac{R1 - R2}{\frac{[R1 + R2]}{2}} \times 100$$

where:

R1 = highest result

R2 = lower result

Corrective Action: Repeat analysis once to see if an analytical error has occurred. If the % RPD still exceeds the control limits; include a project narrative with the results to the client noting that there may be potential matrix effects on the precision of the TOC/Soot results as evidenced by the matrix duplicate RPD exceedance.

9.7 Method-specific Quality Control Samples

9.7.1 High Level Initial Calibration Verification (HICV) or Linearity Check

This sample consists of 4% carbon solution prepared at the high calibration point.

Aliquot 50 µL using a syringe into a tin boat, record weight and analyze. (Equivalent to a 5 mg sample containing 40% TOC).

The acceptable recovery QC range for the HICV is 80-120%.

Corrective Action: Repeat analysis once to see if an analytical error occurred. If the HICV still exceeds the control limits, re-calibrate. If any sample exceeds the HICV the sample is re-injected with a smaller sample weight that renders a measurement within range. Alternate HICV may be used to test the higher levels of quantification when needed as typically found in soot analysis.

9.7.2 Standard Reference Material (SRM)

If an SRM is required per project plan, 5.0mg of NIST 1944 with a true value of 4.4% TOC (Section 8.6) is used. It is treated with the same lot of HCl as the associated samples and dried at 75° C. The SRM must recover within 75-125% of the true value.

9.8 Method Sequence

- Calibration Curve generation
- ICV
- ICB
- HICV
- Method Blank
- LCS
- SRM (Optional)
- Laboratory Duplicate
- Matrix Spike
- Samples 1 – 5
- CCV
- CCB
- Samples 6 – 15

- CCV
- CCB

10. Procedure

10.1 Equipment Set-up

Samples are prioritized by the Department Manager for analysis based on hold time and client due date. Section 15.5 outlines the steps for final TOC reporting that will contain the sample analysis final results.

The analyst initials, date, sample ID #'s, sample weights (solids), or volumes (aqueous), of all Standards, QC samples and field samples, are entered into the TOC logbook. Include the standard, LCS and MS IDs and concentrations. Use TOC weight spreadsheet to record all information.

10.1.1 Pretreatment for Total Organic Carbon:

This procedure is used to remove the inorganic compounds in the sample prior to the determination of Total Organic Carbon. If Total Carbon is to be determined, addition of the acid solution is omitted.

10.1.1.1 Aliquot 10-15g of wet solid sample into a weighing vessel. Remove shells, rocks and other large non-representative materials with tweezers. Record weight of vessel and vessel + wet sample.

10.1.1.2 Heat for at least 4 hours at 75 +/- 2 °C. Return sample to oven if additional drying time is required(in ½ hour increments) Record time in and out of oven. Cool sample to room temperature in desiccator. Record weight of vessel + dry sample.

10.1.1.3 Add 10% HCl (Section 8.2) dropwise to dry sample to induce effervescence (removal of inorganic carbon). Do not use excessive acid. Only add enough acid to exhaust the effervescence. In lieu of additional acid, use DI water to wet the sample in order to form a slurry, if necessary, to distribute the acid throughout the sample. Record the volume of HCl used for each sample.

10.1.1.4 Return the sample to the oven. Heat for 4 hours at 75 +/- 2 °C. Return sample to oven if additional drying time is required(in ½ hour increments) Record time in and out of oven. Cool to room temperature in desiccator. Record final weight of dry sample + vessel. The change in weight of dry sample after acidification is used to adjust the weight of the aliquots used for analysis.

Note: If the % change post acidification is a negative value this indicates the initial drying step prior to acidification was incomplete. The sample prep must be repeated.

10.1.1.5 Take a sub-aliquot of approximately 2-3 g of dry sample and perform moisture content using SM2540G. The corrected sample weight (CSW, see Section 11.4) is corrected using this result (see Section 11.4 for calculations)

10.1.1.6 Grind sample with mortar and pestle to homogenize. Place ground sample into a 4 mL vial.

10.1.1.6.1 If Soot analysis is required, take a portion of the dried sample aliquot(prior to acidification), approximately 5-10 gm, and place into a muffle furnace at 375°C for 24 hours. Once the sample aliquot is cooled to room temperature, proceed to the acid addition step, Section 10.1.1.3. Samples for Soot are read analytically in the same way as Routine TOC.

10.1.1.7 Weigh (and record weight of) each replicate into a tin capsule (see Section 10.3 for appropriate weight range). Record the weight and use the TOC weight spreadsheet to calculate the corrected sample weight (CSW) which is entered into the analyzer.

10.2 Initial Calibration

On the accompanying computer, or "PC", Perkin Elmer software has been loaded to collect and save sample and standard analysis data. Create a file to store the data from each day's analytical sequence by clicking on the PE 2400 icon, go to "new" and "save" the file "as" the name of the date of the analysis (*i.e.,* 060503.chn). Data from each standard or sample analysis will be automatically stored in this file for future reference

10.2.1 Follow the instrument Manufacturers User's Manual for instrument set up and see Sections below for an outline of the initial, daily calibration procedure.

See the Manufacturers User's Manual, page 5-69, for the default instrument conditions when in the CHN operating mode.

10.2.2 *Lloyd Kahn* Calibration Method: **Note:** The *Routine* instrument calibration procedure (Section 10.2.2) must be established before the Linear Regression method is used, otherwise, the raw data will not reflect results that approximate the results from the curve.

Prepare a curve for *Lloyd Kahn* calibration as outlined below. Additionally, see page 5-82 and 5-83 of the Manufacturers User's Manual for proper set-up of this calibration method.

Analyze each point of the calibration curve following Section 10.3. On an Excel spreadsheet, plot *Carbon (mg*) vs. *Instrument Response*, using five standards and a blank covering the analytical range of interest. See Section 8.3 for Calibration Standard preparation. Standards are solutions of KHP.

Blank = 10 ul of DI water from Barnstead

Point 1 = 0.1% (1,000mg/Kg) Standard

Point 2 = 0.5% (5,000mg/Kg) Standard Point 3 = 1.0% (10,000mg/Kg) Standard Point 4 = 2.0% (20,000mg/Kg) Standard

Point 5 = 4.0% (40,000 mg/Kg) Standard

This is the calibration curve for Linear Regression analysis. The corrected response (Y-axis) is calculated by subtracting the nitrogen response from the carbon response. This is plotted against concentration.

The correlation coefficient (r) of the initial calibration curve must be ≥0.995. For any calibration other than average response the laboratory shall evaluate Relative Error. The

evaluation will be done on a mid-level calibration point and the lowest calibration point. The lowest point must recover within 50% and the mid-level within 30%. This information will be noted on the calibration check spreadsheet and scanned with the raw data stored at the laboratory.

For Lloyd Kahn analysis, one sample per 20 (or per batch) must be analyzed in quadruplicate.

Corrective Action: If the correlation coefficient criteria is not met, the standard curve must be re-prepared and re-analyzed until the correlation coefficient is acceptable.

10.3 Equipment Operation and Sample Processing

Prepare a Calibration Curve per Section 10.2.

An ICV/ICV and LCS/Method Blank are analyzed immediately following the third K-Factor of the Routine Calibration, provided the instrument is “in tolerance”. The LCS and method blank must be run with the same number of replicates that are required for the samples in the run.

Remove all pretreated samples from the oven, and allow to cool in a desiccator.

Weigh approximately 5-10mg (to an accuracy of 0.01 mg) (masses less than 5 mg must be avoided since balance is not calibrated below 5 mg) of sample into a tin boat using the Perkin-Elmer Microbalance, Model AD-6, . Samples that are known to be high in TOC may use a lesser sample aliquot. Samples where it is observed that the TOC may be low, weight as much as 20-25mg. Record the weights in the TOC/Soot logbook. Matrix Duplicate and Matrix Spike samples are weighed as separate samples, with their own replicates. As noted in Section 10.1.1.6, use the TOC weight spreadsheet to correct the sample weights for all replicates and re-analyses. Any observations regarding the sample composition must be noted in the TOC/Soot logbook such as: sandy, contains shells or rocks, contains grass or roots, is light/white in coloration, etc.

Analyze a CCV/CCB (Section 9.4 and 9.1.3) after every 10 “burns”, and at the end of the analytical sequence.

If following the *Lloyd Kahn* Calibration method, analyze the first set of batch QC samples, the ICV/LCS and Method Blank, followed by field samples with a CCV/CCB analyzed every 10 “burns”, and at the end of the analytical sequence.

10.3.1 Multiple Sample Instrument Loading:

10.3.1.1 Each tin boat is carefully folded with anti-magnetic tweezers, and placed into its respective holding tray position. (See the Manufacturer User’s Manual SOP, Section 1.2, page 4, for tin boat folding details.) Weights, sample tray position and Autosampler location are recorded in the TOC/Soot logbook. The analyst must be very careful when transferring samples. Documentation of sample consistency is very important. See Figure 1 for an example logbook page.

10.3.1.2 Adjust the Autosampler tray to begin sampling at auto-slot #1 and carefully transfer the folded tins containing the samples to the correct auto-slot, using tweezers. All Instrument Calibration Standards (Blanks, K-Factors and/or *Lloyd Kahn* Calibration Standards) are loaded first. Immediately following will be the first batch QC samples, the ICV/LCS and Method Blank for the run, followed by field samples. Rotate the Autosampler in a counterclockwise manner.

10.3.1.3 A maximum of 60 tins can be held on the Autosampler at one time. The instrument only allows for 100 total samples (including calibration/blank samples) to be programmed for any given run.

10.3.2 Sample Analysis

10.3.2.1 Select the *Auto Run* option on the keypad on the instrument. The following menu appears:

AUTO RUN NO. 1
1B 2K 3S
where: B = Blank
K = K-Factor
S = Sample

Assuming the *Instrument Standardization* occupies auto-slots 1-6, Enter "3" for Sample, then select "Enter". Enter the weight of the "Primer" Standard (position 1) and select "Enter".

10.3.2.2 You will be prompted for AUTO RUN NO. 2 as in Section 10.3.3.1, except now there is a function "4NP" which allows the analyst to stop/erase everything by selecting this feature.

- Enter "1" for the Blank. The weight will not be asked for.
- Enter "2" for the first K-Factor, then select "Enter". Enter the weight of your K-Factor (the standard weight should be ~ 10mg). Select "Enter".
- Enter "1" for the second Blank, then "Enter".
- Enter "2" for the second K-Factor, then "Enter". Enter the weight, then "Enter".
- Enter "2" for the third K-Factor, then "Enter". Enter the weight, then "Enter".

10.3.2.3 All remaining sample weights are added by entering "3" for Sample, and then by entering the sample weight. Continue adding all information to the run sequence, using the keypad. When entering sample identifications, and letters are necessary, refer to the Manufacturer's User's Manual, page 3-4, for instructions for converting numbers to letters.

10.3.2.4 When all sequence information is entered and complete, select "Start". The analyses will begin. The computer will continue to display the "AUTO RUN" menu during sample analysis.

10.3.2.5 The Standard and Sample data prints out as it is collected on the line printer, and is also collected on the linked computer using the PE 2400 software. The data on the computer is saved in a file named as the date of the analysis (*i.e.,* 060503.chn) for future reference, as noted in 10.2.

10.3.2.6 When all analyses are complete, refer to the Manufacturers User's Manual SOP, Section 3.1, page 7, for instructions to "Shut Down" the instrument.

10.3.2.7 Evaluate all batch QC samples first, to confirm or exclude the need for re-analyses. The ICV/LCS/HICV, Method Blank, CCB/CCV samples must meet the acceptance criteria in Section 9. Follow the corrective action for any QC failures.

10.3.2.8 Evaluate the field samples, LCS/SRM, and MS/Duplicates next. If replicate "burns" vary by more than 30% and are greater than 5 times the RL, re-weigh two additional aliquots similar to the initial weights used and re-analyze the sample. For a 2-replicate analysis, this provides a total of four burns/samples. If the second set of replicates also has RPD > 30%, report the first set. If the re-analysis has an RPD which passes, report the re-analysis. Three and four replicate analyses may be requested, and the average of the multiple burns is reported, unless otherwise indicated by the client. Analysts should use judgement in cases where there may have been an error with the autosampler or other factor (incorrect loading, etc.) which may have led to RPD failure. The Lloyd Kahn method requires that quadruplicate analysis be performed on one sample per batch of up to 20 samples and that the RSD must meet precision criteria of 3 times the standard deviation of laboratory generated sample data. One sample per batch of up to 20 must be analyzed in quadruplicate. RSD criteria is 40%. This value was generated using 30 data points of quadruplicate analysis of various concentrations and multiple instruments. The RSD was calculated for each sample in quadruplicate, and the RSD limit is based on 3 times the average standard deviation. Failure to meet these criteria requires that the entire batch be re-analyzed.

10.4 Continuing Calibration

A CCV must be analyzed as outlined in Section 10.3.3 at a minimum of every 10 burns and at the close of an analytical sequence.

10.5 Preventive Maintenance

The reduction column is changed after every 100-200 runs or as needed. The combustion column is replaced as needed.

11. Data Evaluation, Calculations and Reporting

11.1 Procedures for data and record management for TOC/Soot analysis must adhere to the Quality Systems Manual, other subordinate documents covering record keeping, and the *Document Control* SOP, 08-01. All records are stored in such a manner as to be safe and accessible for at least 10 years.

11.2 The relevant TOC/Soot laboratory notebooks must follow the specifications in the *Laboratory Notebook Usage* Work Instruction 1556, and all record keeping and document control practices.

11.3 Sample results for TOC/Soot are directly reported from the instrument printout.

11.4 Calculations: The following calculations are applied by the instrument when determining the percentage of Total Organic Carbon or Soot: Note: sample weights are corrected for sample mass changes due to acidification prior to analysis and entered into the instrument software as the corrected weights, therefore, no correction is required after analysis.

Corrected Sample Weight(CSW): Weight that is entered at the instrument, corrected for sample mass change after acidification.

CSW = aliquot wt (mg) * total sample dry wt (g) (prior to acidification) * dry wt.@ (75°C)
Total sample dry wt (g) (post acidification) *dry wt.@(105°C)

Carbon Blank (CB): Used to make all other determinations, namely, K-Factor and weight percent calculations. When run alternately with samples, the instrument averages the blank values.

Carbon Blank (CB) = Carbon Read – Nitrogen Read

Carbon K-Factor (C KF): K-Factor, or detector calibration factor, is determined when a known standard is analyzed to calibrate the analyzer in terms of micrograms of carbon. This calibration factor is then used to determine unknowns.

C KF = ((CR – NR) – CB x 100) / (CSW x C Theory Wt. %)

where: CR = Carbon Read
NR = Nitrogen Read
CB = Carbon Blank
CSW = Corrected Sample Weight (mg) (using corrected weight recorded in Section 10.3)
C Theory Wt % = 1% (based on the concentration of the K-Factor standard)

Carbon (mg/Kg):

Carbon (mg/Kg) = (((CR – NR) – CB) x 1000) / (SW x C KF)

where: CR = Carbon Read
NR = Nitrogen Read
CB = Carbon Blank
CSW = Corrected Sample Weight (mg)
C KF = Carbon K-Factor

To convert to % C divide result in mg/Kg by 10000

Example Calculation of Percent TOC/Soot from the instrument, converted to mg of TOC/Soot:

If the result from the instrument is 1.083% and the sample weight is 10.76mg:

1.083% = 100 x [**X** / 10.76mg] (to solve for **X,** divide each side by 100)
0.01083 = **X** / 10.76mg (multiply each side by 10.76mg)
0.116mg = **X**

11.5 Reporting Results

The following procedures must be followed for reporting of TOC/Soot results:

Go to the next page in the bound TOC/Soot logbook.

Retain all computer printouts for the standard and sample analyses of this sequence.

Print the sequence from the computer and review it to make sure that no weight or sample ID transcription errors were made.

Calculate the recoveries for the QC samples (IVC/LCS, CCV, MS and RPD between the duplicate samples) and ensure the Method Blank and CCB meet the criteria. Remember any "QC Failures" require corrective action. See Section 9.0 or 12.0.

After reviewing the final logbook entries, make a copy of the logbook page for secondary review.

Go to the LIMS and "batch" and "associate" the set of QC samples and field samples that were just analyzed.

Enter the Final Results into the LIMS report sheet and check for transcription errors.

Transfer all associated sample paperwork to the Department Manager for the second level review and approval.

All TOC/Soot results are reportable without qualification if analytical holding times are met, preservation (including pH and cooler temperatures) are met, all QC criteria defined in the table below are met, and matrix interference was not suspected during analysis of the TOC/Soot samples. If any of the below QC parameters are not met, all associated samples must be evaluated for re-analysis.

QC Parameter	Acceptance Criteria
Initial Calibration Curve, *Lloyd Kahn*	r $\geq$ 0.995
Initial Calibration Verification	80-120% of True Value
High-Level ICV (Linearity Check)	80-120% of True Value
Relative Error Calibration Verification	Low Standard: 50-150% Mid Standard: 70-130%
Method Blank	< reporting limit for matrix
Laboratory Control Sample/SRM	80-120% LCS 75-125% SRM
Matrix Duplicate	25%RPD for results >5x reporting limit
Replicate burns (each sample)	30% RPD for results >5x reporting limit
Quadruplicate analysis(1 per 20 samples)	< 40 % RSD
Matrix Spike	75-125% R
Matrix Spike Duplicate if requested	75-125%R/25%RPD
Continuing Calibration Verification	80-120% of True Value
Continuing Calibration Blank	< reporting limit for matrix

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Section 9 outlines sample batch QC acceptance criteria. If non-compliant TOC/Soot results are to be reported, the Department Manager and/or the Laboratory Director, and the QA Manager must approve the reporting of these results. The laboratory Project Manager is notified, and may chose to relay the non-compliance to the client, for approval, or other corrective action, such as re-sampling and re-analysis. The analyst or Department Manager performing the secondary review initiates the project narrative, and the narrative must clearly document the non-compliance and provide a reason for acceptance of these results.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

The *Hazardous Waste and Sample Disposal* SOP (1797) must be referenced for disposal of used standards, solvents, acids, reagents or other chemicals.

Once satisfactory TOC results have been generated, the sample containers must be scanned back into LIMS and returned to the appropriate sample refrigerator and held for 30 days.

All waste generated must be transferred to the waste disposal area. Dispose all solid samples in solid waste container. Aqueous samples must be poured into a 55-gallon drum marked acidic/non-chlorinated waste.

Refer to the *Chemical Hygiene Plan* and the *Hazardous Waste & Sample Disposal SOP* (1797) for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

PE 2400 Series II CHNS/O Analyzer User's Manual

Sample Receipt & Login SOP (1559)

Perkin-Elmer, Model AD-6 Manufacturer User's Manual

Document Control SOP 1729

Laboratory Notebook Usage Work Instruction (WI 1556)

SOP/1732 MDL Generation

SOP/1739 IDC Generation

Hazardous Waste & Sample Disposal SOP (1797)

16. Attachments

None.

Total and Amenable Cyanide

Reference: **Method 9010C / 9012B,** SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Revision 2 and Revision 3 2004

SM 4500CN-CEG. Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

Method 10-204-00-1-A, Lachat Instruments, 6645 West Mill Road, Milwaukee, WI 53218, 1994.

Method 9014 (Modified). SW-846, Test Methods for Evaluating Solid Waste: Physical / Chemical Methods, EPA SW-846, Update III, 1997

1. Scope and Application

Matrices: This method is applicable to waters, liquids, solids, soils and sludges

Definitions: Refer to Alpha Analytical Quality Manual.

The following SOP is a reflux-distillation procedure used to extract soluble cyanide salts and many insoluble cyanide complexes from wastes and leachates. It is based on the decomposition of nearly all cyanides by a reflux distillation procedure using a strong acid and a magnesium catalyst. Cyanide, in the form of hydrocyanic acid (HCN) is purged from the sample and captured into an alkaline scrubber solution. The concentration of cyanide in the scrubber solution is then determined by flow injection analysis on a Lachat Analyzer.

This method was designed to address the problem of “trace” analyses (<1000ppm). The method may also be used for “minor” (1000ppm – 10,000ppm) and “major” (>10,000ppm) analyses by adapting the appropriate sample dilution. However, the amount of sodium hydroxide in the standards and the sample analyzed must be the same.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Lachat and in the interpretation of Lachat data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The cyanide, as hydrocyanic acid (HCN), is released from samples containing cyanide by means of a reflux-distillation operation under acidic conditions and absorbed in a scrubber containing sodium hydroxide solution. The cyanide concentration in the absorbing solution is then determined colorimetrically by Lachat flow injection analysis.

2.1 Method Modifications from Reference

The sample size used is 50mL. The Midi distillation unit has demonstrated the ability to achieve the same RDL using 50mL instead of 500mL sample volume. Refer to EPA Method 335.4.

Modification for Method 9014: An automated determination of cyanide using the Lachat instrument is used instead of manual spectrophotometric determination.

Modification for amenable cyanide: Analysis is not prepped under amber light.

3. Reporting Limits

The Reported Limit for aqueous samples is 0.005mg/L; soil and solid samples is 1mg/Kg

4. Interferences

4.1 Interferences are eliminated or reduced by using the distillation procedure. However, chlorine and sulfide are interferences. Refer to Section 10.1.1.

4.2 High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose under test conditions to generate HCN. The possibility of interference of nitrate and nitrite is eliminated by pretreatment with sulfamic acid just before distillation. Nitrate and nitrite are interferences when present at levels higher than 10mg/L and in conjunction with certain organic compounds.

4.3 Thiocyanate is reported to be an interference when present at very high levels. Levels of 10mg/L were not found to interfere.

4.4 Fatty acids, detergents, surfactants, and other compounds may cause foaming during the distillation when they are present in high concentrations. Add anti-foaming agent to the sample during the distillation procedure (Section 10.3.1).

4.5 Carbonates and aldehydes are possible interferences.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The following chemicals have the potential to be highly toxic or hazardous. For detailed explanations consult the MSDS:

- Cyanide
- Sulfuric acid
- Pyridine
- Chloramine-T

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in plastic or glass containers. All containers must be thoroughly cleaned and rinsed.

Oxidizing agents such as chlorine decompose most cyanides. Testing for chlorine must be done in the field prior to sample preservation.

6.2 Sample Preservation

Prior to preservation, samples must be tested for chlorine (Section 6.1).

Aqueous samples are preserved with 50% sodium hydroxide in the field to a pH $\geq$ 12 at the time of collection.

Samples and distillates are stored in the refrigerator at 4 ±2 °C.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

When properly preserved, cyanide samples are stored for up to 14 days prior to sample preparation steps.

Distillates must be analyzed within 14 days of distillation. Samples must be analyzed within 14 days of receipt.

Note: for MCP-TCN samples must be analyzed within 24h after distillation

7. Equipment and Supplies

7.1 Cyanide Midi Distillation Unit: Lab Crest, BGL or comparable midi distillation unit. With reaction vessels, collection vessels, cold fingers and impingers.

7.2 pH paper: Range 1-14

7.3 Lead Acetate Paper

7.4 Vacuum source

7.5 50mL centrifuge tubes: New, plastic, with caps.

7.6 KI starch paper: Residual Chlorine sensitivity

7.7 Class A volumetric flasks: 25, 50, 100, 500 and 1000mL

7.8 Graduated cylinders: 50mL glass or plastic

7.9 Eppendorf pipettor or pipets: 0.5, 1, 2, and 5mL

7.10 Lachat 8500 Flow Analyzer: Including Quick Chem software, autosampler, pump and accessories.

7.11 Balance: Capable of weighing to 0.0001gram

7.12 Beakers: 100mL

7.13 Chiller

7.14 Stir plate

7.15 Stir bars

7.16 Dilution vials

8. Reagents and Standards

Reagent grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

8.1 Standards and Reagents for Distillation

8.1.1 Reagent Water: All references to water in this method refer to Deionized Water (DI) from Alpha's water treatment system.

8.1.2 Ascorbic Acid, $C_6H_8O_6$: Powder. Store at room temperature. Expires upon manufacturer's specified date.

8.1.3 Sodium hydroxide solution (1N), NaOH: In a 1L volumetric flask, dissolve 40g of NaOH. Bring to volume with DI water. Store at room temperature. Expires one month from date of preparation.

8.1.4 Sulfamic acid (0.4N), H_2NSO_3H: In a 1L volumetric flask, dissolve 40g H_2NSO_3H. Bring to volume with DI water. Store at room temperature. Expires 6 months from date of preparation.

8.1.5 Sulfuric acid (1:1), H_2SO_4: To a 1L volumetric flask, add 500mL DI water. Slowly and carefully add 500mL of concentrated H_2SO_4. Store at room temperature. Expires one month from date of preparation.

8.1.6 Magnesium chloride solution (2.5M), $MgCl_2 \cdot 6H_2O$: In a 1L volumetric flask, dissolve 510g of $MgCl_2 \cdot 6H_2O$. Bring to volume with DI water. Store at room temperature. Expires 6 months from date of preparation.

8.1.7 LCS, 1000ppm cyanide stock solution: Commercially available standard with a certificate of analysis and from a different source than the Lachat calibration standards. Purchased from Ricca, Catalog # 2543-32. Store refrigerated at 4 ± 2 °C. Expires upon manufacturer's specified date.

8.1.8 LCS 10ppm cyanide working solution: Pipet 1mL of 1000ppm cyanide stock solution (Section 8.1.7) into a 100mL volumetric flask. Add 10mL of 1N NaOH (Section 8.1.3). Bring to volume with DI water. Prepare each day of use.

8.1.8.1 This can also be made by taking 5mL of 10ppm (Section 8.1.8) up to 500mL with 0.1N NaOH. This would be distilled as a whole volume (50mL). Prepare daily.

8.1.9 1000ppm Stock Spiking Solution: 1000ppm cyanide standard available commercially with a certificate of analysis. This is from a different source than the LCS (8.1.7). Purchased from LabChem Inc., Catalog # LC13545. Store refrigerated at 4 ± 2 °C. Expires upon manufacturer's specified date.

8.1.10 10ppm Working Cyanide Spiking Solution: Pipet 1mL of the 1000ppm Stock Spiking Solution (Section 8.1.9) into a 100mL volumetric flask. Add 10mL 1N NaOH (Section 8.1.3). Bring to volume with DI water. Prepare fresh each day of use.**pH 4 Acetate Buffer solution:** In a 500mL volumetric flask, dissolve 410g of sodium acetate trihydrate. Bring to volume with DI water. Adjust to pH of 4.5 with acetic acid (Section 8.1.13). Store at room temperature. Expires 6 months from date of preparation.

8.1.11 Lead Carbonate Powder, [Pb (CO3)]

8.1.12 LCS 0.5 ppm Cyanide Working Solution: Pipet 5mL of the 10ppm Working Cyanide Spiking Solution (Section 8.1.10) into a 100mL volumetric flask . Add 1mL of 10N NaOH (Section 8.1.16). Bring to volume with DI water. Prepare each day of use.

8.1.13 Concentrated Acetic Acid: Store at room temperature. Expires upon manufacturer's specified date.

8.1.14 Ottawa Sand or Boiling chips

8.1.15 Sodium hydroxide solution (10N), NaOH: In a 1L volumetric flask, dissolve 400g of NaOH. Bring to volume with DI water. Store at room temperature. Expires 6 months from date of preparation.

8.1.16 Sodium hydroxide solution (0.1N), NaOH: In a 10L carboy mix 100mL of 10N NaOH. Bring to volume with DI water. Store at room temperature. Expires daily.

8.1.17 Total Cyanide SRM: ERA catalog # 541. Store in room temperature. Expires upon manufacturer's specified date.

8.1.18 Calcium Hypochlorite Solution: Dissolve 5g Ca(OCl)2 in 100mL Deionized water. Store in an amber colored bottle in the dark. Expires monthly.

8.2 Standards and Reagents for Lachat Analysis

8.2.1 Helium gas: To prevent bubble formation, degas all solutions except the standards with helium. Use He at 140kPa (20 lb/in^2) through a helium degassing tube (Lachat part number 50100). Bubble He vigorously through the solution for one minute.

8.2.2 Reagent 1. Carrier, 0.1N Sodium Hydroxide: In a 1L plastic container add 10mL of 10N NaOH (Section 8.1.16). Bring to 1L volume with DI. Store at room temperature. Prepare fresh bi-weekly.

8.2.3 Reagent 2. Acetate Buffer, 2.68M: In a 1L volumetric flask, dissolve 163g spdium acetate trihydrate (acetic acid, sodium salt trihydrate, $CH_3CO_2NA•H_2O$) in approximately 800mL of water. Add 40mL of acetic acid to solution. Dilute to the mark and invert to mix. Store at room temperature. Prepare fresh monthly.

8.2.4 Reagent 3. Chloramine-T: Dissolve 2.0g chloramine-T hydrate in 500mL DI. Prepare fresh daily.

8.2.5 Reagent 4. Pyridine-Barbituric Acid Reagent: Under a fume hood, place 15g barbituric acid in a 1L beaker and add 100mL water, rinsing down the sides of the beaker to wet the barbituric acid. Add 75mL pyridine (C_5H_5N) while stirring and mix

until the barbituric acid dissolves. Add the 15mL concentrated hydrochloric acid (12M HCl) and mix. Store at room temperature. Prepare fresh weekly.

8.2.6 **0.5ppm Calibration standard:** Pipet 5mL of the 10ppm working cyanide spiking solution (Section 8.1.10) into a 100mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.7 **0.2ppm Calibration standard:** Pipet 2mL of the 10ppm working cyanide spiking solution (Section 8.1.10) into a 100mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.8 **0.1ppm Calibration standard:** Pipet 1mL of the 10ppm working cyanide spiking solution (Section 8.1.10) into a 100mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use. This calibration standard is also used as the Continuing Calibration Verification sample.

8.2.9 **0.04ppm Calibration standard:** Pipet 5mL of the 0.2ppm calibration standard (Section 8.2.7) into a 25mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.10 **0.02ppm Calibration standard:** Pipet 1mL of the 0.5ppm calibration standard (Section 8.2.6) into a 25mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.11 **0.01ppm Calibration standard:** Pipet 10mL of the 0.02ppm calibration standard (Section 8.2.10) and 10mL of 0.1N NaOH into a container and mix. Prepare each day of use.

8.2.12 **0.004ppm Calibration standard:** Pipet 5mL of 0.04ppm calibration standard (Section 8.2.9) into a 50mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

8.2.13 **0.1ppm ICV standard:** Pipet 1mL of the 10ppm LCS cyanide working solution (8.1.8) into a 100mL volumetric flask. Bring to volume with 0.1N NaOH. Prepare each day of use.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank

A minimum of one method blank is distilled and analyzed per batch of 20 or less samples. The Method Blank is utilized to determine if contamination or any memory effects are occurring. (Section 10.3.1.1) The blank result must be less than the reporting limit of 0.005 mg/L for liquids and 1 mg/kg for soils. If the blank result is outside of acceptance criteria, it is injected another time. If failure continues, sample analysis is terminated and the source of the problem is found and corrected. All samples analyzed since the last acceptable blank analysis must be reanalyzed.

9.2 Laboratory Control Sample (LCS)

Distill and analyze two LCSs per batch of 20 samples. A Low LCS is analyzed at 0.1mg/L and a high LCS is analyzed at 0.2mg/L. (Section 10.3.1.2)

LCS measurements for Method SM 4500 CN-CE must be within ±10%. For Method 9010C/9012B, the LCS measurements must be within ± 15% for liquids and +/- 20% for soils.

Samples for Method 9010C/9012B: LCSDs are distilled and analyzed along with the LCSs, as described above. The RPD between LCS and LCSD must be ≤ 20% for liquids and <35% for soils.

For soil samples: LCS and LCSD recovery must be within vendor specified acceptance criteria (it will be different for different lots of SRM)

If any LCS fails acceptance criteria for either % Recovery or RPD, analysis is terminated and samples are redigested and analyzed

9.3 Initial Calibration Verification (ICV)

The Initial Calibration Verification Standard (ICV) (Section 8.2.13) is analyzed immediately following the calibration to verify the curve. If the measurements exceeds ±10% of 0.1ppm, the analysis is terminated and recalibration must occur. An acceptable result for the ICV must be obtained prior to any sample analysis.

9.4 Continuing Calibration Verification (CCV)

The Continuing Calibration Verification Standard (CCV) (Section 8.2.8) is analyzed after every 10 injections. The CCV measurements cannot exceed ±10% of the 0.1ppm. If the CCV is not within acceptance criteria, the standard is injected again. If failure continues, sample analysis is terminated and the source of the problem is found and corrected. All samples analyzed since the last acceptable calibration verification must be reanalyzed.

9.5 Matrix Spike

Distill and analyze one spike per batch of 20 samples. For Method 9010C/9012B distill and analyze one spike per batch of 10 samples.

For Method 9010C/9012B, the % Recovery must be within ±20% for liquids and ±35% for solids. For SM 4500CN-CE, the % Recovery must be within ±10%. (Section 10.3.1.4).

Samples for Method 9010C/9012B: A Matrix Spike Duplicate (MSD) is distilled and analyzed along with the MS, as described above. The RPD between MS and MCSD must be ≤ 20% for liquids and <35% for soils

9.6 Laboratory Duplicate

Analyze one duplicate sample for every 20 samples. A duplicate sample is a sample brought through the entire sample preparation and analytical process. (Section 10.3.1.5)

The RPD must be 20% or less for liquids and 35% or less for soils and solids. See Section 12 for Corrective Action if these criteria are not met.

9.7 Method-specific Quality Control Samples

None.

9.8 Method Sequence

The analytical sequence is:

- Screening of samples for chlorine and sulfide
- Prep of amenable cyanide, if needed
- Distillation:
    - Samples

- LCS Low
- LCS High
- Blank
- Matrix Spike
- Duplicate

- Analysis:
  - Calibration and Standardization of Lachat Instrument
  - CCV
  - CCB
  - ICV
  - ICB
  - 10 samples
  - CCV
  - CCB
  - 10 samples
  - CCV
  - CCB
  - Calculation of sample cyanide concentration

10. Procedure

10.1 Equipment Set-up

10.1.1 Screening for Chlorine and Sulfide Interference

10.1.1.1 Chlorine Interference

Oxidizing agents, such as chlorine, decompose most cyanides. Test by placing a drop of sample on a strip of potassium iodide (KI) - starch paper previously moistened with acetate buffer solution, pH 4. If positive indication is noted, then treat an aliquot of sample with Ascorbic Acid (Section 8.1.2). Repeat this test until the KI paper is negative. Immediately inform the Department Supervisor of this interference.

Manganese dioxide, nitrosyl chloride, etc., if present also may cause discoloration of the test paper

10.1.1.2 Sulfide Interference

Oxidized products of sulfide convert CN- to SCN- rapidly, especially at high pH. Test for S^{-2} by placing a drop of sample on lead acetate test paper previously moistened with acetic acid buffer solution, pH 4 (Section 8.1.11). Darkening of the paper indicates presence of S^{-2}. Add powdered lead carbonate [Pb (CO3)] in 1g increments to the whole sample volume. Re-test with acetate paper. Repeat test until a drop of treated sample no longer darkens the acidified lead acetate test paper. Record in the sample prep logbook the amount of lead carbonate added to the sample.

10.1.2 Amenable Cyanide Prep

10.1.2.1 Add 25mL, or 1g and 25mL of DI (8.1.1), to a 100mL beaker with a stir bar.

10.1.2.2 Add 1mL of 10ppm LCS cyanide working solution (8.1.8) to beaker.

10.1.2.3 Prep one sample in duplicate.

10.1.2.4 Add 1-2mL of calcium hypochlorite solution (8.1.18) to all samples and QC under the hood.

10.1.2.5 Check for the presence of chlorine by placing a drop of the sample on a KI-starch paper (7.6). It should turn blue if there is sufficient chlorine.

10.1.2.6 Check samples every 15 minutes for one hour for the presence of chlorine and add more calcium hypochlorite if needed.

10.1.2.7 After one hour of digesting, add ascorbic acid (8.1.2) until KI strip no longer turns blue.

10.1.2.8 Bring sample volume up to 50mL and distill following steps 10.3.1.6 thru 10.3.1.15.

10.2 Initial Calibration

10.2.1 Allow 15 minutes for heating unit to warm up to 60 °C.

10.2.2 Prepare a series of 7 calibration standards (Sections 8.2.6 – 8.2.12) and a 0.1N NaOH blank. Alternatively, calibration standards may be prepared by auto-diluting a 0.5ppm calibration standard (Section 8.2.6). Perform this function per the Lachat manufacturer's instructions for the Quick Chem 8500.

10.2.3 Set up manifold as shown in Table 1.

10.2.4 Input data system parameters as shown in Table 2.

10.2.5 Place standards and blank in the autosampler, per the manufacturer's instructions. Input the information required by the data system, such as concentration, replicates and QC scheme.

10.2.6 Inject the standards, per the manufacturer's instructions.

10.2.7 Prepare a standard curve by plotting instrument response against standard concentration values. A calibration curve is fitted to the calibration solution concentration/response data using the computer. The calibration coefficient of the curve must be greater than or equal to 0.995 before sample analysis can begin.

Calibration coefficient will be calculated using Lachat software. All calibration points are back calculated by Lachat software and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse than 0.995.

10.3 Equipment Operation and Sample Processing

10.3.1 Distillation

10.3.1.1 Add 50mL of shaken liquid sample, or 1gram of a well-homogenized solid sample and 50mL of DI, to the 50mL reaction vessel.

10.3.1.2 For the Liquid High LCS, fill one 50mL reaction vessel with 50mL DI. For the soil High LCS, add 1g Ottawa Sand (Section 8.1.15) and 50mL of DI. After the system has been charged with air, add 1mL of 10ppm LCS cyanide working solution (8.1.8) to the closed system. (Final concentration equals 0.2mg/L.)

For the Liquid Low LCS, fill one 50mL reaction vessel with 50mL of DI. For a soil Low LCS, add 0.2-0.3 g (depending on actual SRM stock concentration) of SRM (sec 8.1.17) and 50mL of DI. Record exact SRM weight. For liquid samples: After

the system has been charged with air, add 0.5mL of 10ppm LCS cyanide working solution (Section 8.1.8) to the closed system. (Final concentration equals 0.1mg/L.) **Don't add liquid Cyanide Standard for soil samples!** Final LCS soil concentration will change based on SRM lot
Samples for Method 9010C/9012B: Prepare a LCS Duplicate along with the LCSs described above.

10.3.1.3 For the method blank for liquid samples, fill one 50mL reaction vessel with 50mL of DI. For the method blank for soil samples, fill a 50mL reaction vessel with 1g of Ottawa Sand (Section 8.1.15) and 50mL DI.

10.3.1.4 For the matrix spike, fill a 50mL reaction vessel with 50mL of sample that has been chosen to be spiked. For soil samples, use 1g of soil and add 50mL of DI water. After the system has been charged with air, add 1mL of 10ppm working cyanide spiking solution (8.1.10) to the closed system.

Samples for Method 9010C/9012B: Prepare a Matrix Spike Duplicate (MSD) in the same manner as the MS, as described above.

10.3.1.5 For the duplicate, fill a 50mL reaction vessel with a duplicate aliquot of 50mL, or 1g soil and 50mL DI water of a sample that has been chosen to be duplicated.

10.3.1.6 Into the receiver or scrubber tube add 5mL of a 1N NaOH solution and add 40mL of DI water.

10.3.1.7 Arrange tubes in the distillation unit noting in the logbook which sample is in which glassware. The glassware is numbered and consistently placed in the same position in the distillation unit.

10.3.1.8 Assemble the unit completely. Turn on the pump. There must be gas bubbling in each tube. Check to make sure all connections are tight and bubbles are flowing at an equal rate in each sample tube. If not, adjust flow rate with the knobs in front of each receiver tube and/or check lines to ensure they are not obstructed.

10.3.1.9 Add 5mL of 0.4N sulfamic acid (8.1.4) to each sample tube and rinse the closed 50mL reaction vessel with a squirt of DI. No residue is to be left of the vessel wall.

10.3.1.10 Add 5mL of 1:1 H_2SO_4 (8.1.5) to each sample tube and rinse the closed 50mL reaction vessel with a squirt of DI. No residue is to be left on the vessel wall. Turn on the heat. Samples are to come to a boil on all of the midi-still units.

10.3.1.11 After 2 minutes of heating, add 2mL of 2.5M $MgCl_2$ Solution (8.1.6) to each sample tube, followed by a rinse with DI. If foaming occurs, an additional 2mL of $MgCl_2$ Solution may be added. If foaming continues, stop the distillation for that sample and reduce the sample size by 2 – 5x (as determined by the severity of the foaming). Contact the Inorganics Supervisor for guidance.

10.3.1.12 Turn on the chiller.

10.3.1.13 Set the timer-dial on the distillation unit to "110".

10.3.1.14 After 110 minutes the unit will shut off; leave the chiller running for an additional 30 minutes while the tubes cool down.

10.3.1.15 Pour contents of the scrubber tube into a new, labeled, centrifuge tube (Section 7.5). Carefully rinse the scrubber tube with DI water and add rinseate to the centrifuge tube to bring to 50mL volume. Cap and refrigerate for later analysis by the Lachat Instrument.

10.3.2 Lachat Analysis

10.3.2.1 Following initial calibration and standardization, (Section 10.2 and 10.4), place the samples in the autosampler, per the manufacturer's instructions. Input the information required by the data system, such as concentration, replicates and QC scheme.

10.3.2.2 Inject the samples, per the manufacturer's instructions.

10.3.2.3 The data system calculates sample concentration using the regression equation. Results are mg/L for Aqueous samples and mg/Kg for soil and solid samples.

10.3.2.4 If sample concentrations are greater than the highest calibration standard, the distilled sample is diluted with 0.1N sodium hydroxide (NaOH) diluent (Section 8.2.2), and reanalyzed. When the automated diluter is used, 0.1N NaOH is also used. **Do not dilute distilled samples or standards with DI water.**

10.4 Continuing Calibration

10.4.1 After the calibration has been established, it must be verified by the analysis of an Initial Calibration Verification Standard (ICV) (Section 8.2.13). The ICV of 0.1ppm must be made from a different source than the calibration standards. If the measurements exceed ±10% of 0.1ppm, the analysis is terminated. See Section 9.3 for Corrective Actions.

10.4.2 A Blank and a Continuing Calibration Verification (CCV) sample (Section 8.2.8) are analyzed after every 10 injections. The CCV measurements cannot exceed ±10% of the CCV value of 0.1ppm and the blank result must be less than the reporting limit of 0.005 mg/L. See Section 9.4 for CCV Corrective Actions and Section 9.1 for Blank corrective actions.

10.5 Preventive Maintenance

Preventative maintenance is recorded in the instrument maintenance logbook and is performed on the Lachat instrument as follows:

Daily:

1) Clean the autosampler
2) Clean the surfaces on the auto-dilutor
3) Prime the dilutor with fresh DI water
4) Clean the pump surfaces
5) Clean the detector with DI and dry with Kim-Wipes
6) Clean the instrument surfaces with DI, wipe clean with a paper towel

Bi-weekly:

1) Clean the injection ports with DI. Take apart the injection valve and inspect it for corrosion. Make sure that the valve connectors are tight and the o-rings are not worn. If the O-rings look worn replace with new ones.

Monthly:

1) Using DI water, clean the unions and the tees that are associated with the manifold.
2) Delete Temporary files on the computer

Every 6 months:

1) Replace the o-rings in the injection valve.
2) Replace the o-rings in the manifold

3) Back up the files on the computer

11. Data Evaluation, Calculations and Reporting

11.1 The Lachat data system calculates sample concentration using the regression equation.

11.2 Report only those values that fall between the lowest and the highest calibration standards.

11.3 Report results in mg CN/L for liquids and in mg CN/kg for soils.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and improper preservation are noted on the nonconformance report form. The analyst narrates the nonconformance when the project is turned in for review. The narration must state what the nonconformance was and any corrective action taken.

Perform routine preventative maintenance according to Section 10.5. Record all maintenance in the instrument logbook. Notify the Department Manager if the instrument problems are not routine in nature. The Department Manager determines whether the problem can be corrected with in-house technical staff or if the instrument vendor should be contacted to schedule service. All service calls are documented in the Instrument logbook, and a copy of the service report is given to the Department Manager.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. If any part of batch quality control does not meet acceptance criteria, the Department Manager is notified. If enough sample remains and holding time has not expired, then the batch is redistilled and reanalyzed. If there is not sufficient sample remaining to allow redistillation, then that analysis is repeated and both sets of data are reported with the nonconformance narrated on the final report.

If either the ICV, ICB, Method Blank, LCS, LCSD, CCV, or CCB recovery falls outside the designated acceptance range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form.

Immediate corrective action for a failing CCV/CCB includes reanalyzing the failing standard. If the standard passes the second time then the analysis may be continued. The raw data is noted. If the standard fails again, the problem must be found and corrected. The CCV/CCB standard is remade and reanalyzed. If the standard passes, all samples analyzed since the previous passing standard are reanalyzed. The raw data is noted and all data associated with the failing standard must have one line drawn through the data, indicating it's unable.

If the standard fails after instrument maintenance, the instrument is recalibrated. A new ICV/ICB is performed, and all samples analyzed since the previous passing CCV/CCB are reanalyzed.

If following reanalysis of the LCS, it is found to still be outside acceptance criteria, the entire sample batch must be redistilled and reanalyzed. If the %RPD between the LCS/LCSD fails after reinjection, then the entire sample batch must be redistilled and reanalyzed.

If the Method Blank fails it is re-poured and reinjected. If failure continues, the associated sample data is evaluated as follows: Sample results below the detection limit may be reported with a narrative included. If samples have positive results, and the results are greater than 10x the concentration found in the method blank, the data may be reported with a narrative included. Any positive samples with results less than 10x the concentration found in the method blank must be redistilled and reanalyzed.

If the Matrix Spike recovery does not meet acceptance criteria, and the LCS recovery is acceptable, matrix interference may be assumed. The associated data may be reported with a narrative included.

If sample Duplicates are outside of the acceptance criteria, the analyst examines the sample for homogeneity. If the sample is not homogenous, this is narrated on the final report. Clean, homogenous samples are redistilled and reanalyzed within holding time.

Sample nonconformance regarding a Matrix Spike recovery or a duplicate %RSD is narrated on the final report along with the corrective action(s) taken.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

The quarterly method reporting limit (MRL) verification requirement is satisfied by the quarterly LOQ analysis, as per SOP/1732.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

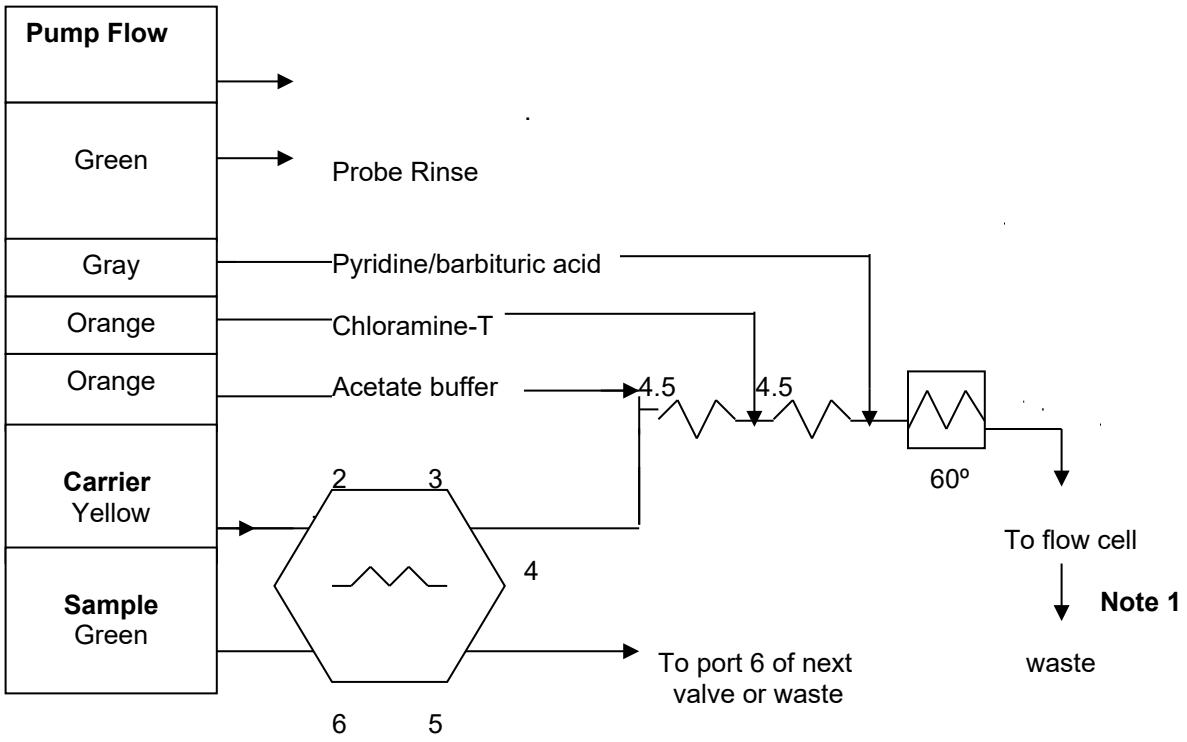
1728 Hazardous Waste Management and Disposal SOP

16. Attachments

TABLE 1: Cyanide Manifold Diagram

TABLE 2: Data System Parameters for QC 8500

TABLE 1
Cyanide Manifold Diagram



Sample Loop = 150cm x 0.8mm i.d. Interference Filter = 570nm
QC8000 Sample loop = 150cm x 0.8mm i.d.

CARRIER is 0.1 N sodium hydroxide solution.

All manifold tubing is 0.8mm (0.030 in) i.d. This is 5.2µL/cm.

4.5 is 70.0cm of tubing on a 4.5cm coil support

APPARATUS: An injection valve, flow cell, a 10mm path length flow cell, and a colorimetric detector module are required.

The box shows 650cm of tubing wrapped around the heater block at the specified temperature.

Note 1: 2 meter back pressure loop, 0.52mm i.d.

TABLE 2
Data system Parameters for QC 8500

The timing values listed below are approximate and will need to be optimized using graphical events programming.

Sample Throughput: 65 samples/hour, 55 s/sample
Pump Speed: 35
Cycle Period: 55

Analyte Data:

Concentration Units: mg CN^-/L
Peak Base Width: 60.5 s
% Width Tolerance: 100
Threshold: none
Inject to Peak Start: 55 s
Chemistry: Direct

Calibration Data:

Levels	1	2	3	4	5	6	7	8
Concentration ug/50mL	25	10	5	2	1	0.5	0.2	0

Calibration Fit Type: 1st Order Polynomial
Calibration Rep. Handling: Replace
Weighting Method: 1/X
Concentration Scaling: None
Force Through Zero: No

Sampler Timing:

Min. Probe in Wash Period: 14 s
Probe in Sample Period: 13 s

Valve Timing:

Load Time: 0.0 s
Load Period: 9 s
Inject Period: 20 s

Total Organic Carbon (TOC)
Dissolved Organic Carbon (DOC)
Total Inorganic Carbon (TIC)

Persulfate – Ultraviolet Oxidation Method

References: **SM Method 5310 C,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

EPA Method 9060 A, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III B, 2004.

1. Scope and Application

Matrices: This method may be applied to water and wastewater samples containing minimum amounts of particulates and phase-separated organics.

Definitions: See Alpha Laboratories Quality Manual Appendix A

The organic carbon in water and wastewater is composed of a variety of organic compounds in various oxidation states. Some of these carbon compounds can be oxidized further by biological or chemical processes, and the bio-chemical oxygen demand (BOD) and chemical oxygen demand (COD) may be used to characterize these fractions. The presence of organic carbon that does not respond to either the BOD or COD test makes these methods unsuitable for the measurement of total organic carbon. Total organic carbon (TOC) is a more convenient and direct expression of total organic content than either BOD or COD, but does not provide the same kind of information. If a repeatable empirical relationship is established between TOC and BOD or COD, then TOC can be used to estimate the accompanying BOD or COD. This relationship must be established independently for each set of matrix conditions, such as various points in a treatment process. Unlike BOD or COD, TOC is independent of the oxidation state of the organic matter and does not measure other organically bound elements, such as nitrogen and hydrogen, and inorganics that can contribute to the oxygen demand measured by BOD and COD. TOC measurement does not replace BOD and COD testing.

To determine the quantity of organically bound carbon, the organic molecules must be broken down to single carbon units and converted to a single molecular form that can be measured quantitatively. TOC methods utilize heat and oxygen, ultraviolet irradiation, chemical oxidants, or combinations of these oxidants to convert organic carbon to carbon dioxide (CO_2). Within this method, the CO_2 is measured directly by a nondispersive infrared analyzer.

The methods and instruments used in measuring TOC analyze fractions of total carbon (TC) and measure TOC by two or more determinations. These fractions of total carbon are defined as:

- Inorganic carbon (IC) - the carbonate, bicarbonate, and dissolved CO_2 total organic carbon (TOC) - all carbon atoms covalently bonded in organic molecules.
- Dissolved organic carbon (DOC) - the fraction of TOC that passes through a 0.45µm filter.
- Particulate organic carbon (POC) – also referred to as nondissolved organic carbon, the fraction of TOC retained by a glass fiber filter.
- Volatile organic carbon (VOC) also referred to as purgeable organic carbon, the fraction of TOC removed from an aqueous solution by gas stripping under specified conditions.

- Nonpurgeable organic carbon (NPOC) the fraction of TOC not removed by gas stripping.

In most water samples, the IC fraction is many times greater than the TOC fraction. Eliminating or compensating for IC interferences requires multiple determinations to measure true TOC. IC interference can be eliminated by acidifying samples to pH 2 or less to convert IC species to CO_2. Subsequently, purging the sample with a purified gas removes the CO_2 by volatilization. Sample purging also removes POC so that the organic carbon measurement made after eliminating IC interferences is actually an NPOC determination; determine VOC to measure true TOC. In many surface and ground waters, the VOC contribution to TOC is negligible. Therefore, in practice, the NPOC determination is substituted for TOC.

Alternatively, IC interferences may be compensated for by separately measuring total carbon (TC) and inorganic carbon. The difference between TC and IC is TOC.

The purgeable fraction of TOC is a function of the specific conditions and equipment employed. Sample temperature and salinity, gas-flow rate, type of gas diffuser, purging-vessel dimensions, volume purged, and purging time affect the division of TOC into purgeable and nonpurgeable fractions. When separately measuring VOC and NPOC on the same sample, use identical conditions for purging during the VOC measurement as in purging to prepare the NPOC portion for analysis. Consider the conditions of purging when comparing VOC or NPOC data from different laboratories or different instruments.

Many instruments utilizing persulfate oxidation of organic carbon are available. They depend on ultraviolet irradiation activation of the reagents. The persulfate-ultraviolet oxidation method is a rapid, precise method for the measurement of trace levels of organic carbon in water and is of particular interest to the electronic, pharmaceutical, and steam-power generation industries where even trace concentrations of organic compounds may degrade ion-exchange capacity, serve as a nutrient source for biological growth, or be detrimental to the process for which the water is being utilized.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the TOC Analyzer and in the interpretation of TOC data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Organic carbon is oxidized to carbon dioxide, CO_2 by persulfate in the presence of ultraviolet light. The CO_2 produced may be measured by a nondispersive infrared analyzer.

The TOC Analyzer utilizes an ultraviolet lamp submerged in a continuously gas-purged reactor that is filled with a constant-feed persulfate solution. The samples are introduced serially into the reactor by an autosampler. The CO_2 produced is sparged continuously from the solution and is carried in the gas stream to an infrared analyzer that is specifically tuned to the absorptive wavelength of CO_2. The instrument's microprocessor calculates the area of the peaks produced by the analyzer, compares them to the peak area of the calibration standard stored in its memory, and prints out a calibrated organic carbon value in milligrams per liter.

2.1 Method Modifications from Reference

2.1.1 Method 9060 requires that samples be analyzed in quadruplicate (4 replicates). Alpha analyzes samples in quadruplicate with a % RSD required to be ≤15%.

3. Detection Limits

The Reported Detection Limit for TOC is 0.5mg / L and for DOC is 1.0mg/L.

4. Interferences

4.1 Instrumental

The intensity of the ultraviolet light reaching the sample matrix may be reduced by highly turbid samples or with aging of the ultraviolet source, resulting in sluggish or incomplete oxidation. Large organic particles or very large or complex organic molecules such as tannins, lignins, and humic aid may be oxidized slowly because persulfate oxidation is rate-limited.

4.2 Parameters

Excessive acidification of sample, producing a reduction in pH of the persulfate solution to 1 or less, can result in sluggish and incomplete oxidation of organic carbon.

Persulfate oxidation of organic molecules is slowed in samples containing significant concentrations of chloride by the preferential oxidation of chloride; at a concentration of 0.1% chloride, oxidation of organic matter may be inhibited completely. If chloride interference is suspected, then the sample is diluted.

With any organic carbon measurement, contamination during sample handling and treatment is a likely source of interference. This is especially true of trace analysis. Take extreme care in sampling, handling, and analysis of samples with TOC below 1mg/L.

Removal of carbonate and bicarbonate by acidification and purging with purified gas results in the loss of volatile organic substances. The volatiles also can be lost during sample blending, particularly if the temperature is allowed to rise. Another important loss can occur if large carbon-containing particles fail to enter the needle used for injection. Filtration, although necessary to eliminate particulate organic matter when only DOC is to be determined, can result in loss or gain of DOC, depending on the physical properties of the carbon-containing compounds and the adsorption of carbonaceous material on the filter, or its desorption from it. Check filters for their contribution to DOC by analyzing a filtered blank. Note that any contact with organic material may contaminate a sample. Avoid contaminated glassware, plastic containers, and rubber tubing. Method Blanks are analyzed with each batch to verify a clean system.
The presence of large amounts of particulate material will cause problems with the determination of carbon. The TOC analyzers are not equipped for the analysis of samples with large amounts of particulate matter. Small amounts may be compensated for by dilution (refer to Section 10.3.2).

5. Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by

whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Collect samples in two (for method -5310) or three (for method -9060) 40mL amber vials with or without Teflon septa. Minimal headspace is desirable.

6.2 Sample Preservation

6.2.1 If samples are to be analyzed for Dissolved Organic Carbon (DOC), they must be filtered within 24 hours of collection, and prior to preservation. Refer to Section 10.1.1 for filtration procedure.

6.2.2 Within two hours of sampling, the samples are preserved with 1:1 H_2SO_4 to a pH $\leq$2 and refrigerated at 4 $\pm$ 2 ºC until analysis.

6.3 Sample Shipping

No special shipping requirements other than the samples should be on ice.

6.4 Sample Handling

Samples to be filtered by the laboratory for DOC analyses, must be filtered upon receipt at the laboratory (within 24 hours from collection time).

Preserved samples must be analyzed within a 28-day holding time.

7. Equipment and Supplies

7.1 Filtering Apparatus.

7.2 0.45 µm Filters.

7.3 40mL Amber Glass Vials: with or without Teflon septa.

7.4 Glass Syringes: 100µL and 200µL volumes.

7.5 Pipettor: **1-10ml**, with disposable tips.

7.6 Volumetric Flasks: Class A 1 L and 250 mL sidearm filtering flask

7.7 AAL: "TOC-3": Shimadzu TOC-V WP Total Organic Carbon Analyzer with autosampler.

7.7.1 Computer and related accessories capable of running associated software: TOC-Control V ver. 1.07.00. c2000-2004 by Shimadzu Corp.

7.8 AAL: "TOC-4", "TOC-5", "TOC-6": Shimadzu TOC-V WP Total Organic Carbon Analyzer with autosampler.

7.8.1 Computer and related accessories capable of running associated software: TOC-Control V ver. 2.30

7.9 10ml disposable syringes:
BD 10ml syringe with Luer-Lok Tip, P/N 309604

7.10 0.45u acrodisc for use with disposable syringe:
Pall LIfe Sciences Acrodisc 25mm syrringe filter with 0.45u Nylon membrane, P/N 4438 T

8. Standards and Reagents

8.1 Reagent Water: Deionized water (DI).

8.2 10% Persulfate + 5% Phosphoric Acid Reagent Mixture: Measure 50g 98^{+}% sodium persulfate ($Na_2S_2O_8$) into a rinsed bottle. Add 18mL of 85% phosphoric acid (H_3PO_4). Add 426mL of reagent water (DI). Mix well. This mixture must be prepared monthly. Label bottle with date made, date of expiration, reagent lot number, and store at room temperature. (Use AAL unique ID: example: TOC-030917-R2).

8.3 21% Acid Reagent: Measure 74mL of 85% phosphoric acid (H_3PO_4) into a rinsed bottle. Add 376mL of reagent water (DI). Mix well. This mixture must be prepared monthly. Label bottle with date made, date of expiration, and reagent lot number and store at room temperature. (Use AAL unique ID: example: TOC-030917-R1)..

8.4 2000ppm Organic Carbon Stock Calibration Standard: In a 1L volumetric flask, add 4.25g of Potassium Hydrogen Phthalate (KHP) and 1mL H_3PO_4. Bring to volume with DI and agitate to mix well. Transfer to a 1L Amber glass bottle. Prepare every six months. Label bottle with standard lot number, date made, and date of expiration. Store at 4 $\pm$ 2 °C. (Use AAL unique ID: example: TOC-030917-C).

8.4.1 4.0ppm CCV: To a 100mL volumetric flask, add 200µL of 2000ppm stock calibration standard (Section 8.4) and bring to volume with DI. Prepare fresh on each day of use. (Use AAL unique ID: example: TOC-030917-CCV).

8.4.2 50ppm Calibration Standard: To a 100mL volumetric flask, add 2.5mL of 2000ppm stock standard (Section 8.4) and bring to volume with DI. Prepare fresh for each calibration curve generated.

8.4.3 10ppm Calibration Standard: To a 100mL volumetric flask, add 0.5mL of 2000ppm stock standard (Section 8.4) and bring to volume with DI. Prepare fresh for each calibration curve generated.

8.4.4 5.0ppm Calibration Standard: To a 100mL volumetric flask, add 10mL of 50ppm calibration standard (Section 8.4.2) and bring to volume with DI. Prepare fresh for each calibration curve generated.

8.4.5 1.0ppm Calibration Standard: To a 100mL volumetric flask, add 2mL of 50ppm calibration standard (Section 8.4.2) and bring to volume with DI. Prepare fresh for each calibration curve generated.

8.4.6 0.5ppm Calibration Standard: To a 100mL volumetric flask, add 1mL of 50ppm calibration standard (Section 8.4.2) and bring to volume with DI. Prepare fresh for each calibration curve generated.

8.5 2000ppm Organic Carbon Stock Check Standard: In a 1L volumetric flask, add 4.25g of Potassium Hydrogen Phthalate (KHP) and 1mL H_3PO_4. Bring to volume with DI and agitate to mix well. Transfer to a 1L Amber glass bottle. Prepare every six months. Label bottle with standard lot number, date made, and date of expiration. Store at 4 ± 2 °C. (Use AAL unique ID: example: TOC-030917-W).

The KHP used for this solution must be from a different source and lot number than that used in Section 8.4.

8.5.1 Initial Calibration Verification Standard (ICV)

4.0ppm ICV: To a 100mL volumetric flask, add 200µL of 2000ppm stock check standard (Section 8.5) and bring to volume with DI. This is analyzed at the start of each daily run. Prepare fresh on each day of use. (Use AAL unique ID: example: TOC-030917-ICV).

8.5.2 Calibration Verification Blank (ICB)

One ICB is analyzed at the start of each daily run. The ICB consists of 40mL of DI. The DI must be from the same source as was used to prepare the Calibration Verification samples. Results must be < 0.5mg/L, but preferably ≤ 0.2mg/L. If this criterion is not met, the instrument must be internally cleaned again (Refer to Section 10.1.4) to ensure that the system is clean prior to sample analysis.

8.6 400ppm TOC IC Stock Standard:

To a 100mL volumetric flask, add 0.3545g sodium carbonate (Na2CO3) anhydrous powder. Bring to volume with DI water. Store at room temperature. Prepare annually or as needed. (Use AAL unique ID: example: TOC-030917-IC400).

8.6.1.1 10ppm TOC IC Working Check Standard (Negative LCS): Into a 100mL volumetric flask, add 2.5mL of 400ppm TOC IC Stock Standard. Bring to volume with DI water. Store at room temperature. Prepare weekly or as needed. Also referred to as the 10ppm IC CK STD (Inorganic Check Standard). (Use AAL unique ID: example: TOC-030917-IC10).

8.7 Purging / Carrier Gas: Nitrogen, ultra-high purity.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank (MB)

9.1.1 Method Blank (MB)

One MB is analyzed per batch of 20 samples or less. The MB consists of 40mL of DI. The DI must be from the same source as was used to prepare the Calibration Verification samples. Results must be < 0.5mg/L, but preferably ≤ 0.2mg/L. If this criterion is not met, the instrument must be internally cleaned again (Refer to Section 10.1.4) in order to ensure that the system is clean prior to sample analysis.

In the case of DOC, the MB (or also referred to as the Filter Blank) consists of DI that was filtered through a 0.45µm filter (Section 7.2). Results must be <1.0mg/L. If this criterion is not met, first ensure as stated above, that the instrument system is clean.

Next: there are two options regarding the sample:

1. The sample and associated Filter Blank are refiltered. This must depend on sample holdtime, and available volume.
2. If the sample result is greater than 10x the result of the Filter Blank, then the reported data can be accompanied by a narrative stating this.

9.2 Laboratory Control Sample (LCS)

9.2.1 **4.0ppm LCS:** To a 100mL volumetric flask, add 200µL of 2000ppm stock check standard (Section 8.5) and bring to volume with DI. The 2.0ppm LCS is analyzed for each batch of 20 or less samples (per instrument and per day). Therefore, a new LCS should be analyzed for each additional batch of 20 or less samples, when multiple batches are run on the same instrument and daily analytical sequence. Refer to Section 9.3 (Use AAL unique ID: example: TOC-030917-LCS).

9.2.2 **Negative LCS Standard (LCSN):** A negative LCS (LCSN) is analyzed before the Calibration Blank (ICB) to demonstrate that there is no Inorganic Carbon contribution to the Organic Carbon analysis. The analysis of the 10ppm LCSN (also referred to as the 10ppm IC CK STD) (8.6.1) must yield a result of < 0.5mg/L

(Use AAL unique ID: example: TOC-030917-IC).

If the LCSN fails these criteria, the data is inspected to determine the cause of the failure. The standard is reanalyzed. If failure continues, the standard is remade and reanalyzed. If failure continues, there is a system malfunction and sample analysis cannot take place. The following instrument parameters are investigated: UV Lamp function, sparger function and acid delivery to the sparger. If the problem persists, contact the Department Supervisor. Analysis cannot begin until the 10ppm LCSN is within acceptance criteria.

Note: Also referred to as the 10ppm “IC CK STD” (Inorganic Check Standard).

9.3 Initial Calibration Verification (ICV)

An ICV is analyzed at the beginning of the analytical sequence, at a concentration of 4.0ppm. (For preparation instructions, refer to Section 8.5.1.)

The ICV must be recovered within ± 10% of the true value. If this criterion is not met, the system must be cleaned again, and the ICV must be re-analyzed. If failure continues, remake

the ICV (Section 8.5.1) and/or generate a new calibration curve (Section 10.1.6). Sample analysis cannot proceed until an acceptable ICV is obtained.

9.4 Continuing Calibration Verification (CCV)

A Continuing Calibration Verification standard is analyzed after every 10 or less samples (inclusive of the method blank, lcs, duplicate and matrix spike samples).

To a 100mL volumetric flask, add 200µL of 2000ppm stock calibration standard (Section 8.4) and bring to volume with DI. Prepare fresh on each day of use.

9.4.1 This CCV is at a concentration of 4.0ppm TOC (Section 8.4.1) and must be recovered within ±10% of the true value. If the CCV fails this criterion, it is re-made and re-analyzed.

If failure continues, all samples analyzed since the last valid CCV, are considered invalid and must be re-analyzed. However, prior to re-analysis, a new calibration curve must be generated. (Refer to Section 10.1.6.)

9.4.2 Continuing Calibration Blank (CCB)

A Continuing Calibration Blank is analyzed after every 10 samples (inclusive of the method blank, lcs, duplicate and matrix spike samples). This CCB consists of 40mL DI water and must be recovered below the detection limit. If the CCB fails this criterion, it is re-poured and re-analyzed.

If failure continues, all samples analyzed since the last valid CCB, are considered invalid and must be re-analyzed. However, prior to re-analysis, a new calibration curve must be generated. (Refer to Section 10.1.6.)

9.5 Matrix Spike

One Matrix Spike (MS) is analyzed per batch of 10 samples or less. Measure 40mL of the well-homogenized sample to be spiked into a 40mL amber glass vial with Teflon cap. Using a glass syringe, add 80µL of the 2000ppm Stock Check Standard (Section 8.5). Invert to mix. The true value of the spike prepared accordingly, is 4ppm. The matrix spike must be recovered within 80 – 120% for method 9060 and 85-115% for method 5310.

If the matrix spike recovery is outside of acceptance criteria, the sample and its MS are reanalyzed upon dilution with DI water for TOC analysis. If failure repeats, the data is reported, and a narrative submitted for inclusion on the Client report.

9.6 Laboratory Duplicate

One Duplicate sample is analyzed per batch of 10 samples or less. Samples are routinely collected in two 40mL amber glass vials; therefore, the second vial may be used as the sample duplicate. It must, however, be analyzed as if it were a separate sample. The %RPD between the sample and its duplicate must be <20% for method 9060 and <15% for method 5310.

If the % RPD is outside of acceptance criteria, the duplicate is reanalyzed. If failure repeats, the data is reported, and a narrative submitted for inclusion on the Client report.

9.7 Method-specific Quality Control Samples

Refer to Section 9.2.2.

9.8 Method Sequence

- TOC-3, TOC- 4, TOC-5 and TOC-6: DI (Rinse water)

- TOC-3 & TOC-4, TOC-5 and TOC-6:
- Calibration curve generation (if necessary)
- DI water (to check system stability)
- IC CK STD (10ppm Negative LCS)
- ICV 4.0ppm
- ICB
- MB (per 20 samples or less)
- LCS 4ppm (per 20 samples or less)
- Samples 1-8
- CCV 4.0ppm
- CCB

If Method 5310, then proceed with samples 9-18 (dup/spk required per 10 samples)

- CCV 4.0ppm
- CCB
- Duplicate
- Matrix Spike 4ppm
- CCV 4.0ppm
- CCB
- Shut down

If Method 9060, then proceed with dup and spk (dup/spk required per 10 samples)

- Duplicate
- Matrix Spike 4ppm
- CCV 4.0ppm
- CCB
- Shut down

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation for Dissolved Organic Carbon (DOC) Analysis

Prior to preservation, samples to be analyzed for dissolved organic carbon (DOC) are filtered through a 0.45µm filter (Section 7.2) as follows:

- Prior to filtration of the sample, filter 1L of DI through a 0.45µm filter and discard.
- Filter sample through the same 0.45µm filter:
  - For method -5310: 100ml of sample for two vials
  - For method -9060: 150ml of sample for three vials
- Transfer filtered sample to two 40mL amber vials and preserve accordingly.
- Prepare a DOC method blank following the same procedure listed above. Filter enough to fill two to three 40mL amber vials, dependent on which method is being filtered for.

Another method of filtration of DOC samples can be the following: Use 0.45 µm filtering discs with a 10 or 20mL disposable syringe. Each syringe and disc must be rinsed thoroughly first by filling with DI and discarding PRIOR to any Filter Blanks or samples.

Record all necessary information in the filtration logbook: sample ID, date and time of filtration, analyst's initials, the lot number of the vials and syringe discs used, and the preservative used.

10.1.2 Instrument Set-Up

Follow the manufacturer's instructions for proper instrument set-up procedures. For TOC-3, TOC- 4, TOC-5, and TOC-6 instruments, refer to Table A.

10.1.3 Reagent Preparation

10.1.3.1 Verify that the reagents (Sections 8.2 and 8.3) have not expired and that there is adequate Nitrogen gas supply. If necessary, prepare reagents and/or alert Tech Support if gas manifold has a malfunction.

10.1.3.2 Ensure that the DI supply to the instrument is fresh for each day of analysis.

10.1.3.3 Prepare the 4.0ppm ICV (Section 8.5.1) and CCV standard (Section 8.4.1) each day of analysis.

10.1.4 Instrument Preparation

10.1.4.1 (TOC-3, -4, -5, -6)

Set instrument to analysis 1 vial of Rinse water, entitled DI, to ensure system is stable and clean before analyses.

Proceed to section 10.1.5

10.1.5 If a valid calibration curve is already in place, proceed to Section 10.2.

10.1.6 Calibration Curve Generation

A new calibration curve is generated every six months. However, if there has been a major change to the system, a new calibration curve must be generated following the change and prior to sample analysis.

NOTE: For TOC/DOC Method 5310 with samples originating from Massachusetts, a new calibration curve must be generated DAILY prior to the analysis of these samples.

A calibration curve of 5 points will be used for this: Blank (0ppm, DI), 0.5ppm, 1.0ppm, 5.0ppm, and 10.0ppm (refer to Section 8.4 for instructions).

Prepare five calibration standards per Section 8.4.2 through Section 8.4.6 and two DI water blanks.

Analyze the five calibration standards and blanks:

(TOC-3, -4, -5, -6) with the TOC Shimadzu TOC-V WP software program, Set the range at 0.1 – 10ppm for TOC.

After analysis, the software program for TOC-3, 4, 5, 6 will allow the user to select the calibration standards and blank desired to include in the calibration curve calculation.

All of the standards must be chosen, and also the second blank (the first blank is considered a rinse). The correlation coefficient (r) must be $\geq$ 0.995. If this criterion is not met, the calibration standards are re-made, and the calibration curve is re-analyzed.

All calibration points are back calculated by software and should be within 10% from true concentration, except lowest points of calibration curve. % Recoveries for low range will be wider,but shouldn't exceed 100% and correlation coefficient will not be worse than 0.995.

Samples and QC samples may not be analyzed until a valid Calibration Curve is obtained.

10.2 Initial Calibration

10.2.1 Before any analyses, verify that the system is clean.

10.2.2 Run one DI (Rinse water) sample to check for system stability. (Note: Only at initial instrument startup. This also acts as a TOC water screen to ensure the lab water being used is acceptable (resulting value is below the reporting limit of 0.5ppm for TOC and 1.0ppm for DOC) prior to use).

Per the manufacturer's instructions, set up the autosampler to analyze the following:

TOC-3, TOC-4, TOC-5, TOC-6 = DI (Rinse water)

10.2.3 Negative LCS Standard (LCSN) (also referred to as the IC CK STD): Prior to sample analysis and following the ICV and ICB, a negative LCS (LCSN) is analyzed to demonstrate that there is no Inorganic Carbon contribution to the Organic Carbon analysis. The analysis of the 10ppm LCSN (8.6.1) must yield a result of < 0.5mg/L.

If the LCSN fails this criteria, the data is inspected to determine the cause of the failure. The standard is reanalyzed. If failure continues, the standard is remade and reanalyzed. If failure continues, there is a system malfunction and sample analysis cannot take place. The following instrument parameters are investigated: UV Lamp function, sparger function and acid delivery to the sparger. If the problem persists, contact the Department Supervisor. Analysis cannot begin until the 10ppm LCSN is within acceptance criteria.

10.2.4 Initial Calibration Verification (ICV) and Initial Calibration Blank (ICB)

Prior to sample analysis, the calibration curve must be verified.

Analyze an ICV and an ICB to confirm stability and cleanliness.

4.0ppm ICV (Section 8.5.1)

ICB (DI water)

The ICV must have a % Recovery ± 10% of the true value. The results for the ICB must be less than 0.5mg/L, but preferably less than or equal to 0.2mg/L. If these criteria are not met, the system must be cleaned again (Section 10.1.4) and the ICV and ICB must be re-analyzed. If failure continues, remake the ICV (Section 8.5.1) and/or generate a new calibration curve (Section 10.1.6).

10.3 Equipment Operation and Sample Processing

10.3.1 Samples and QC samples are analyzed according to the manufacturer's instructions for TOC Shimadzu TOC-V WP software program (TOC-3, 4, 5, 6) instructions, using

the valid calibration curve in the TOC Shimadzu TOC-V WP software program range of "0.1 – 10ppm TOC". (As generated in Section 10.1.6).

10.3.1.1 Dissolved Organic Carbon: If samples are to be analyzed for DOC, the same procedure is followed as for TOC.

10.3.2 The samples to be analyzed must have a minimum amount of sediment. Initial dilution of samples with sediment may be necessary to facilitate analysis by either instrument. Dilutions are prepared volumetrically (or with a graduated cylinder and calibrated auto-pipettor) with DI water. However, prior to dilution, consider the history of the samples and the detection limit requirements of the client. If questions arise due to sample matrix, consult with the Department Supervisor and/or the Laboratory Director before proceeding with analysis.

10.3.3 Samples are to be analyzed with 3 Reps each. The TOC Software will calculate the standard deviation, the %RSD, and the mean. The %RSD must be $\leq$ 15% for method 9060 and <10% for method 5310. If this criterion is not met, the sample must be re-analyzed with 2 Reps. If failure continues, consult with the Department Supervisor and/or the Laboratory Director before proceeding.

TOC Method 9060: Samples (including all batch QC) must be analyzed with 4 Reps each.

10.3.4 Similarly, a duplicate sample and a matrix spike sample are to be analyzed in replicate. Prepare the duplicate and matrix spike samples according to Sections 9.6 and 9.5.

10.3.4.1 For Method 5310, a duplicate and matrix spike is required for every **10** samples or less in a batch.

10.3.4.2 For Method 9060, a duplicate and matrix spike is required for every **10** samples or less in a batch.

10.3.5 If the sample results are above 10mg/L TOC, they are above the high end of the calibration curve. In this instance, the sample must be manually diluted volumetrically (or with a graduated cylinder and calibrated auto-pipettor) with DI water and re-analyzed. A timeout message or baseline drift might also necessitate a dilution of the sample even if the sample is < 10mg/L.

While setting up the diluted sample in the batch, the analyst must include the dilution factor in the Sample ID field.

10.4 Continuing Calibration

10.4.1 Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB)

A Continuing Calibration Verification standard and a Continuing Calibration Blank pair are analyzed after every 10 samples (inclusive of the duplicate and matrix spike samples).

10.4.1.1 The CCV is at a concentration of 4.0ppm TOC (Section 8.4.1) and must be recovered within ±10% of the true value. If the CCV fails this criterion, it is re-made and re-analyzed.

If failure continues, all samples analyzed since the last valid CCV, are considered invalid and must be re-analyzed. However, prior to re-analysis, a new calibration curve must be generated. (Refer to Section 10.1.6.)

10.4.1.2 The results for the CCB must be less than 0.5mg/L, but preferably less than or equal to 0.2mg/L. If the CCB fails this criterion, it is reanalyzed.

If failure continues, all samples analyzed since the last valid CCB, are considered invalid and must be re-analyzed. However, prior to re-analysis, a new calibration curve must be generated. (Refer to Section 10.1.6.)

10.5 Preventive Maintenance

The instrument is primed and cleaned internally prior to analysis. Tubing, copper/tin scrubber, and autosampler syringe are inspected routinely to ensure they are in proper working order. The external autosampler is kept clean and free of dust. Extra parts are kept on hand in the event they are necessary. All instrument maintenance is recorded in the Instrument Maintenance Logbook.

If continual bubbling in the gas/liquid separator tube occurs during analysis, lift the back, right cover of the instrument body (carefully so as not to disturb any connected fan wiring), and gently tap the line from the separator tube until all the bubbles are released. Any sample currently running may need reanalysis due to failed detection. (i.e. instrument message “no sample detected”.)

11. Data Evaluation, Calculations and Reporting

The TOC Shimadzu TOC-V WP software program calculates the area of the peaks produced by the analyzer, compares them to the peak area of the calibration standard stored in its memory, and calculates a mean TOC value in mg/L.

If the sample was manually diluted prior to analysis, the calculated mean must be manually multiplied by the dilution factor. The Reported Detection Limit is also multiplied by the same dilution factor.

If the sample result is <0.5mg/L for TOC, it is reported as ND (Not Detected). Likewise, if the sample result is <1.0mg/L for DOC, it is reported as ND (Not Detected).

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Refer to Section 9 for corrective actions.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

The quarterly method reporting limit (MRL) verification requirement is satisfied by the quarterly LOQ analysis, as per SOP/1732.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

Table A: TOC-VW (TOC-3 , TOC-4, TOC-5, TOC-6 INSTRUMENTS)

Table A:

TOC-VW (TOC 3, 4, 5, 6 INSTRUMENT)

STARTING THE INSTRUMENT:

Turn on nitrogen ultra-pure nitrogen gas tank.

Push the on/off button on the bottom right front of the instrument to on (green light will be visible).

With the computer on, use the mouse to double click on the TOC-CONTROL V icon on the desktop.

Double click on “Sample Table Editor”

Enter user’s initials, hit OK

At Sample Table page:

Go to File “New”

Double click on “Sample Run”

Select System “TOC-VW”, hit OK

At Save As page:

For File name, overwrite existing to say current date as month/day/year (00/00/0000), hit SAVE

At Dated Run page:

Click on yellow lightning bolt icon @ top to connect to system

At Parameter Confirmation Dialog page:

Click on “Use Settings on PC”

At Sequence Box page:

Wait for system to initialize (page box will disappear once initialized)

Once initialized, click on white square box @ top (just left of yellow lightning bolt), to see Background Monitor

Wait for NDIR: baseline pos., baseline fluc., and baseline noise -> all 3 items should have a green checkmark before instrument can run analyses.

When all 3 items are green, close this box by clicking on the “X” on the top right of the box.

At the Dated Run page again:

Adjust columns to include: Analysis//Sample Name//Result//Status//Date/Time//Vial

NOTE: Prior to analyzing samples, generate a new Calibration Curve according to the Instrument's User Manual,

TO SET UP AN ANALYSIS RUN:

At top of Dated Run page:

Go to Insert, Autogenerate

Sample group wizard (page 1): sample source:

Bullet calibration curve, push rectangular button w/ dots within it

Next to file name, type toc3curve120211a (for example)

Select current dated curve from choices below

Hit OPEN

Then hit NEXT

Sample group wizard (page 2):

Number of samples = enter number of samples to analyze

Start vial = vial number to begin analyzing

Erase sample name/leave blank, hit NEXT

Page 3: hit NEXT

Page 4: hit NEXT

Page 5: hit FINISH

Sparging/acid addition with autosampler pie display, hit OK

Back at Dated Run page:

Type in information for each sample name in the appropriate column

TO BEGIN ANALYSIS:

Click on the green stoplight icon at top of screen:

Standby box should have "keep running" bullet

Hit Standby button

Then hit OK on sparging/acid addition autosampler pie display page

Then @ start ASI measurement box, UNCHECK the external acid addition box, hit START

TO TURN OFF INSTRUMENT AFTER ANALYSIS IS COMPLETED:

At the end of the run, hit the round clock icon at the top of the screen

Mare sure “shut down instrument” is bulleted

Hit “standby”

Turn off instrument power button so that the green light is now off.

Turn off gas tank.

TO PRINT ANALYSIS PAPERWORK:

File, Print, sample report

Close all pages on screen by hitting the “X” at the top right of each page.

Total Phosphorous

Dissolved Phosphorus

Colorimetric, Combined Reagent

References: **SM 4500P-E,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

SM4500P-B, Section 5 (Persulfate Digestion), Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

AQ2 method**: EPA-119-A** Rev. 7, equivalent to EPA 365.1, version 2(1993) **SM4500-P-B, F(**18-20**)**

1. Scope and Application

Matrices: Water and wastewater samples and soils.

Definitions: See Alpha Laboratories Quality Manual Appendix A

Phosphorus occurs in natural waters and in wastewaters almost solely as phosphates. These are classified as orthophosphates, condensed phosphates (pyro-, meta-, and other polyphosphates), and organically bound phosphates. They occur in solution, in particles or detritus, or in the bodies of aquatic organisms.

These forms of phosphate arise from a variety of sources. Small amounts of certain condensed phosphates are added to some water supplies during treatment. Larger quantities of the same compounds may be added when the water is used for laundering or other cleaning, because these materials are major constituents of many commercial cleaning preparations. Phosphates are used extensively in the treatment of boiler waters. Orthophosphates applied to agricultural or residential cultivated land as fertilizers are carried into surface waters with storm run-off and to a lesser extent with melting snow. Organic phosphates are formed primarily by biological processes. They are contributed to sewage by body wastes and food residues, and also may be formed from orthophosphates in biological treatment processes or by receiving water biota.

Phosphorus is essential to the growth of organisms and can be the nutrient that limits the primary productivity of a body of water. In instances where phosphate is a growth-limiting nutrient, the discharge of raw or treated wastewater, agricultural drainage, or certain industrial wastes to that water may stimulate the growth of photosynthetic aquatic micro- and macro-organisms in nuisance quantities.

Phosphates also occur in bottom sediments and in biological sludges, both as precipitated inorganic forms and incorporated into organic compounds.

Phosphorus analyses embody two general procedural steps: (a) conversion of the phosphorus form of interest to dissolved orthophosphate, and (b) colorimetric determination of dissolved orthophosphate. The separation of phosphorus into its various forms is defined analytically but the analytical differentiations have been selected so that they may be used for interpretive purposes.

Filtration through a 0.45-µm-pore-diameter membrane filter separates dissolved from suspended forms of phosphorus. No claim is made that filtration through 0.45-µm filters is a true separation of suspended and dissolved forms of phosphorus; it is merely a convenient and replicable analytical technique designed to make a gross separation.

Phosphates that respond to colorimetric tests without preliminary hydrolysis or oxidative digestion of the sample are termed “reactive phosphorus.” While reactive phosphorus is largely a measure of orthophosphate, a small fraction of any condensed phosphate present usually is hydrolyzed unavoidably in the procedure. Reactive phosphorus occurs in both dissolved and suspended forms.

Acid hydrolysis at boiling-water temperature converts dissolved and particulate condensed phosphates to dissolved orthophosphates. The hydrolysis unavoidably releases some phosphate from organic compounds, but this may be reduced to a minimum by judicious selection of acid strength and hydrolysis time and temperature. The term “acid-hydrolyzable phosphorus” is preferred over “condensed phosphate” for this fraction.

The phosphate fractions that are converted to orthophosphate only by oxidation destruction of the organic matter present are considered “organic” or “organically bound” phosphorous. The severity of the oxidation required for this conversion depends on the form of, and to some extent on the amount of, the organic phosphorus present. Like reactive phosphorus and acid hydrolyzable phosphorus, organic phosphorus occurs both in the dissolved and suspended fractions.

The total phosphorus as well as the dissolved and suspended phosphorus fractions each may be divided analytically into the three chemical types that have been described: reactive, acid hydrolyzable, and organic phosphorus. Determinations usually are conducted only on the unfiltered and filtered samples. Suspended fractions generally are determined by difference.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Digestion Method: Because phosphorus may occur in combination with organic matter, a digestion method to determine total phosphorus must be able to oxidize organic matter effectively to release phosphorus as orthophosphate. This digestion is performed by using the persulfate oxidation technique.

Colorimetric Method: The ascorbic acid method is used for the determination of orthophosphate in environmental samples. An extraction step is recommended for the lower levels and when interferences must be overcome. Ammonium molybdate and potassium antimonyl tartrates react in acid medium with orthophosphate to form a heteropoly acid-phosphomolybdic acid that is reduced to intensely colored molybdenum blue by ascorbic acid. The absorbance of this complex is measured photometrically at 880nm.

2.1 Method Modifications from Reference

Glassware is acid rinsed with room temperature 1:1 HCl, instead of hot dilute HCl.

Initial testing of samples with phenolphthalein has been eliminated since samples are received already preserved with H_2SO_4 and are pH checked by the Login Department upon receipt.
Soil samples are analyzed using the same digestive procedure.

3. Reporting Limits

The Reported Detection Limit is 0.01mg/L for waters and 5.0mg/kg for soils

4. Interferences

Correction for Turbidity or Interfering Color: The natural color of water generally does not interfere at the high wavelength used. For highly colored or turbid waters, prepare a blank by adding all reagents except ascorbic acid and potassium antimonyl tartrate to the digested sample aliquot. Subtract the blank absorbance from the absorbance of each sample.

Arsenates react with the molybdate reagent to produce a blue color similar to that formed with phosphate. Concentrations as low as 0.1mg As/L interfere with the phosphate determination.

Hexavalent chromium and NO_2 interfere to give results about 3% low at concentrations of 1mg/L and 10 to 15% low at 10mg/L.

Sulfide (Na_2S) and silicate do not interfere at concentrations of 1.0 and 10mg/L.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Water samples are collected in 500mL plastic bottles, soil samples may be collected in plastic or glass jars.

6.2 Sample Preservation

If samples are for Dissolved Phosphorus analysis, filtration must take place prior to preservation with H_2SO_4 to a pH < 2.

All samples are preserved with H_2SO_4.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are stored under refrigeration at 4 ± 2 °C. Analysis must be performed within 28 days of collection. All samples should be analyzed as soon as possible after digestion. If a prolonged period passes in between, sample extracts are refrigerated at 4 ± 2 °C.

7. Equipment and Supplies

7.1 Spectrophotometer, with infrared phototube for use at 880nm, providing a light path of 2.5cm.

7.2 Acid-washed Glassware: Use acid-washed glassware for determining low concentration of phosphorus. Phosphate contamination is common because of its absorption on glass surfaces. Avoid using commercial detergents containing phosphate. Clean all glassware with 1:1 HCl two times followed by two DI water rinses. Preferably, reserve the glassware only for phosphate determination. Only disposable syringes and filters are to be used for filtering samples for Dissolved Phosphorus analysis.

7.3 Centrifuge Tubes: 50mL volume. (Must be new and disposable.)

7.4 Hot Plate: A 30cm x 50cm heating surface is adequate.

7.5 Scoop, 0.5gm To hold required amounts of persulfate crystals.

7.6 Erlenmeyer Flasks: 125mL volume.

7.7 0.45µm membrane filters: For Dissolved Phosphorus sample preparation.

7.8 Borosilicate Glass beads

7.9 SEAL AQ2 Discrete Analyzer, with all associated reagent wedges, sample tubes, and reaction segments. The SEAL has a light and filter capable of maintaining a 880nm wavelength.

7.10 Boiling Chips ultra-pure, non-reactive.

7.11 Syringes to use with membrane filters.

7.12 Pipettes Class A glass or automated.

8. Reagents and Standards

8.1 Calibration Curve and Spike, Stock Complex Phosphate Standard: 1000 mgP/L This stock solution is certified and purchased commercially prepared. Stored at room temperature per manufacturer's specifications. Expires upon manufacturer's specified date.

8.2 Calibration Curve and Spike, Intermediate Complex Phosphate Standard: 50 mgP/L Dilute 5.0mL stock complex phosphate solution to 100mL with DI water. Store at 4 ± 2°C. Expires 6 months after date of preparation.

8.3 Calibration Curve, Working Standard: 1.0 mgP/L Add 2mL of 50 mgP/L intermediate standard (Section 8.2) to 100mL volumetric flask and dilute to volume with DI water. Prepare fresh on each day of use.

8.4 Calibration Standards: Follow table below. Prepare fresh on each day of use.

Volume of 1.0 mg/L Working Standard (Section 8.3)	Final Volume (mL)	Calibration Standard Final Concentration (mgP/L)
0 mL	50	0
0.5 mL	50	0.010
2 mL	50	0.040
5 mL	50	0.100
25mL	50	0.500
50mL	50	1.000

8.5 ICV-LCS-CCV Stock Complex Phosphate Standard: 1000 mgP/L Second, independent, source standard. Stored at room temperature per manufacturer's specifications. Expires upon manufacturer's specified date.

8.6 ICV-LCS-CCV Intermediate Complex Phosphate Standard: 50 mgP/L Add 5mL of 1000 mgP/L stock standard (Section 8.5) to 100mL volumetric flask and dilute to volume with DI water. Store at 4 ± 2°C. Expires 6 months after date of preparation.

8.7 ICV-LCS-CCV Working Standard: Prepare fresh each day of use.

8.7.1 0.5 mgP/L: Add 0.5mL of 50 mg/L intermediate standard (Section 8.6) to 50mL centrifuge tube and dilute to the 50mL mark with DI water.

8.8 Matrix Spike: Intermediate Phosphate Standard (Section 8.2) is utilized for matrix spike solution. Pipet 0.5mL of the 50 mgP/L standard into 50mL of sample to result in a 0.5mg/L spike concentration.

8.9 Sulfuric Acid, H_2SO_4, 5N: Dilute 140mL concentrated sulfuric acid to 1L with DI. Store at room temperature. Expires 6 months from date of preparation.

8.10 Potassium Antimonyl Tartrate Solution: Dissolve 1.3715g $K(SbO)\ C_4H_4O_6 \cdot ½H_2O$ in 400mL DI water in a 500mL volumetric flask and dilute to volume. Store at 4 ± 2°C. Expires one month from date of preparation.

8.11 Ammonium Molybdate Solution: Dissolve 10g $(NH_4)_6Mo_7O_{24} \cdot 4H_2O$ in 250mL distilled water. Store at 4 ± 2°C. Expires one month from date of preparation.

8.12 Ascorbic Acid, 0.1M: Dissolve 3.52g ascorbic acid in 200mL DI water. The solution is stable for about 1 week at 4 ± 2°C.

8.13 Orthophosphate 1000ppm solution Independent, source standard. Store at 4 ± 2°C. Expires upon manufacturer's specified date.

8.14 Orthophosphate 25ppm spike solution Add 2.5mLs 1000ppm Orthophosphate solution to a clean, glass 100mL volumetric and dilute to volume with DI. Store at 4 ± 2°C. Expires 6 months from date of preparation.

8.15 SEAL Working Ascorbic Acid, 15g/L (with orthophosphate spike): Dissolve 1.5g of ascorbic acid in about 80mL DI water. Spike with .15mL 25ppm Orthophosphate standard to produce a spike level of .025mg P/L. Dilute to 100mL and mix well. The solution is stable for one week if stored at 4 ± 2°C. Discard if the solution becomes yellowed.

8.16 Combined Reagent: Mix 8.9, 8.10, 8.11, and 8.12 in the following proportions for 100mL of the combined reagent: 50mL 5N H_2SO_4 (Section 8.9), 5mL potassium antimonyl tartrate solution (Section 8.10), 15mL ammonium molybdate solution (Section 8.11), and 30mL ascorbic acid solution (section 8.12). Mix after addition of each reagent. Let all reagents reach room temperature before they are mixed and mix in the order given. If turbidity forms in the combined reagent, shake and let stand for a few minutes until turbidity disappears before proceeding. The reagent is stable for 4 hours. Discard reagent if it turns blue or black in color.

8.17 SEAL Working Coloring Reagent: To a clean 100mL volumetric flask, add 40mL sulfuric acid (8.9), followed by 6.5 mL antimony potassium tartrate (8.10) and swirl to mix. Then, add 20 mL ammonium molybdate (8.11). Swirl the contents, fill the flask up to the mark with DI water and mix well. Expires three weeks from day of preparation if stored at 4 ± 2°C. Discard if the reagent turns blue or becomes turbid.

8.18 Sodium Hydroxide, 6N: Dissolve 240 grams of NaOH pellets in 1000mL of DI water. Store at room temperature. Expires one month from date of preparation.

8.19 Phenolphthalein Indicator: Aqueous solution, commercially available. Store at room temperature. Expires upon manufacturer's specified date.

8.20 11N Sulfuric Acid Solution: Dilute 308mL concentrated sulfuric acid to 1000mL with DI. Store at room temperature. Expires 6 months from date of preparation.

8.21 Potassium Persulfate ($K_2S_2O_8$): Commercially available. Store at room temperature. Expires upon manufacturer's specified date.

8.22 Deionized Water

8.23 Soil LCS/SRM ERA Standard Reference Material for Nutrients in soil, catalog no. 542

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Method Blank/Calibration Blank - One method blank, which consists of DI water brought through the entire method, must be analyzed per batch of 20 samples or less. The CCB is analyzed after every 10 samples and at the end of the sequence.

Results of the Blanks must be less than the reporting limit. Otherwise the entire batch of samples must be re-prepared and reanalyzed. Exceptions are samples with results of more than 10 times the positive blank value.

Soil blanks are made with 0.1gm boiling chips and 50mLs of DI water and are analyzed like water blanks.

9.2 Laboratory Control Sample (LCS)

Analyze one per batch of 20 samples or less. The calibration curve must be verified by a second source standard prior to performing any sample analysis. For Total and Dissolved Phosphorus, the LCS is the ICV.

The ICV/LCS for aqueous samples must be recovered within 90-110% of the true value. If the ICV/LCS fails, re-analyze. If failure continues, stop analysis, correct problem and re-calibrate.

The Soil LCS made with approximately 0.15g of Standard Reference Material (SRM) brought up to 50mL with DI water. The soil LCS recovery criteria will vary per Lot of SRM, according to the SRM manufacturer's criteria listed on the certificate of analysis.

9.3 Initial Calibration Verification (ICV)

The calibration curve must be verified by a second source standard prior to performing any sample analysis. For Total and Dissolved Phosphorus, the ICV is the LCS.

The ICV/LCS must be recovered within 90-110% of the true value for water samples and be within vendor criteria for SRM. If the ICV/LCS fails, re-analyze. If failure continues, stop analysis, correct problem and re-calibrate.

9.4 Continuing Calibration Verification (CCV)

The calibration curve must be verified by a second source standard. The CCV is analyzed after every 10 samples and at the end of the sequence to verify the curve.

The CCV must be recovered within 90-110% of the true value. If the CCV fails, re-analyze. If failure continues, stop analysis, correct problem and re-calibrate.

9.5 Matrix Spike

Analyze one per batch of 20 samples or less. Concentration is 0.5 mgP/L. The matrix spike must be recovered within 75 – 125% of the true value. If the matrix spike recovery is outside acceptance criteria, and the LCS is acceptable, a narrative is submitted with the data for inclusion on the client report.

9.6 Laboratory Duplicate

Analyze one sample in duplicate per batch of 20 samples or less. The RPD must be ≤ 20%. If this criterion is not met, a narrative is submitted with the data for inclusion on the client report.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

9.8.1 Using spectrophotometer:

- Acid-rinse all glassware
- Calibration curve generation.
- Filter samples if analysis is for Dissolved Phosphorus, then preserve with H_2SO_4.
- Add 50mL of water sample or 0.1g of soil sample and 50mL DI water to an Erlenmeyer flask.
- Add 1mL H_2SO_4 solution and scoop solid $K_2S_2O_8$ and glass beads
- Boil down to 10mL or less.
- Cool and dilute to 30mL with DI.
- Add 1 drop phenolphthalein indicator solution.
- Neutralize to faint pink color with NaOH.
- Add sample aliquot to a new centrifuge tube and bring up to 50mL with DI.
- Add 4 mL combined reagent to a 25mL aliquot of sample.
- Read sample absorbance after 10-30 minutes.
- Analyze CCV and CCB after every 10 samples to verify curve.
- End sequence with CCV and CCB.
- Calculate results.

9.8.2 Using SEAL AQ2 analyzer:

- Acid-rinse all glassware
- Filter samples if analysis is for Dissolved Phosphorus, then preserve with H_2SO_4.
- Add 50mL of water sample (soils are not done on the SEAL) to an Erlenmeyer flask, along with a 1.0ppm calibration standard for the calibration curve.
- Add 1mL H_2SO_4 solution and scoop solid $K_2S_2O_8$ and glass beads
- Boil down to 10mL or less.
- Cool and dilute to 50mL with DI.
- Turn on the SEAL AQ2 analyzer.
- Fill out a run sequence and save it.
- Fill cups, including a cup of 1.0ppm digested standard for the curve
- Start the analysis.
- Change names of blank and LCS to be what the LIMS will recognize (ie, what is on the batch sheet).
- Export the data to LIMS by dropping it into the “SEAL on bowzer” folder.

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Preparation for Dissolved Phosphorus Analysis

Prior to preservation, samples to be analyzed for dissolved phosphorus are filtered using new disposable syringes and new 0.45um filter discs. 100mL of sample is filtered, poured into two new centrifuge tubes and preserved with H_2SO_4.

10.2 Initial Calibration

10.2.1:

Preparation of calibration curve, with spectrophotometer: Prepare individual calibration curve from a series of six digested standards (0 mgP/L to 1.0 mgP/L) on each day of analysis. The curve must be digested. Use DI water without the combined reagent to zero the Spectrophotometer. Plot absorbance vs. phosphate concentration to give a straight line. The correlation coefficient must be 0.995 or greater for the curve to be considered valid. Analyze at least one phosphate standard with each batch of 20 samples or less.

All calibration points are back calculated (on excel) and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse then 0.995.

10.2.2:

Preparation of calibration curve, with SEAL AQ2 analyzer: Prepare and digest a 1.0 mgP/L standard and put it into the first slot in the auto-sampler. When prompted, click on "Auto-calibrate" to start calibration. Once the curve is finished, it may be checked in the "calibration" section. The correlation coefficient must be 0.995 or greater for the curve to be considered valid.

. All calibration points are back calculated by SEAL software and should be within 10% from true concentration, except 2 lowest points of calibration curve. %recoveries for low range will be wider, but shouldn't exceed 100% and correlation coefficient will not be worse then 0.995.

10.3 Equipment Operation and Sample Processing, with spectrophotometer

10.3.1 Add 50mL or a suitable portion of thoroughly mixed sample to a prepared (Section 7.2) 125mL Erlenmeyer flask. Use 0.1g of soil sample with 50 ml of DI for soil samples.

10.3.2 Add 1mL H_2SO_4 solution, one scoop solid $K_2S_2O_8$ and 3 to 5 glass beads.

10.3.3 Boil gently on a preheated hot plate until a final volume of 10mL or less is reached. Organophosphorus compounds such as AMP may require as much as 1-1/2 to 2 hours for complete digestion.

10.3.4 Cool, dilute to 30mL with DI water.

10.3.5 Add 0.05mL (1 drop) phenolphthalein indicator solution.

10.3.6 Neutralize to a faint pink color with NaOH.

10.3.7 Pour pink liquid sample into a new (unused) centrifuge tube and bring volume to 50mL with DI. Pour back into a 125mL Erlenmeyer flask.

10.3.8 Swirl the sample to mix and pour off 25mL digested sample into centrifuge tube.

10.3.9 Add 4.0mL combined reagent to all 25mL sample and QC sample aliquots and mix thoroughly.

10.3.10 After at least 10 minutes but no more than 30 minutes, use DI as the reference solution to zero the spectrophotometer at 880nm. Measure absorbance of each sample and record in the electronic laboratory notebook. If samples seem to have high background color before the addition of the coloring reagent, a background color may be checked for (see section 4).

If the sample concentration is greater than the highest concentration of the calibration curve (1.0mg/L), the digested sample is diluted with DI water to a concentration within the range of the calibration curve.

10.4 Equipment Operation and Sample Processing, with SEAL AQ2 Analyzer

10.4.1 Acid rinse all glassware twice with 1:1 hydrochloric acid and then twice with DI water.

10.4.2 Pour out 50mLs of mixed samples and QC samples, including a 1.0ppm calibration standard.

10.4.3 Add 1mL H_2SO_4 solution, one scoop solid $K_2S_2O_8$ and 3 to 5 glass beads.

10.4.4 Boil gently on a preheated hot plate until a final volume of 10mL or less is reached. Organophosphorus compounds such as AMP may require as much as 1-1/2 to 2 hours for complete digestion.

10.4.5 Cool and dilute to 50mLs.

10.4.6 Turn on SEAL AQ2 analyzer by flipping first the small, and then the large switch in the back.

10.4.7 Give the instrument at least half an hour to warm up.

10.4.8 If it has not yet been done that day, go through daily start-up, check voltages, and test aspiration.

10.4.9 Double click on scheduling to open the schedule form, and insert samples, Method Blanks, LCSs, Duplicates, and Matrix Spikes (the CCVs and CCBs populate automatically) NB: leave the first spot for the calibration curve.

10.4.10 Pour a small (approximately 1 mL) aliquot into small tubes and put them into the instrument. Put the 1.0ppm standard into the first spot.

10.4.11 Check to see that all the reagent wedges are in the correct spots and that there are sufficient reaction segments.

10.4.12 Save the sequence and double click on “run”; check the boxes for curve analysis.

10.4.13 Once the instrument is done analyzing the run, check and approve the results in the Data Review section. Then, change the Blank and LCS names to be whatever they are on the batch sheet (ex: WG123456-1) and save the run in the “out” folder.

10.4.14 Open the “SEAL on bowzer” folder and drop the run into it from the “out” folder. This saves the data to LIMS.

10.5 Continuing Calibration

The method blank and LCS are used as the CCB/CCV and should be read after every ten samples and at the end of the batch. Recovery for the CCV must be between 85-115% of the true value. Recovery for the CCB must be between the RL and its negative, (i.e: within -.01mg/L and .01mg/L for waters).

10.6 Preventative Maintenance

The Spectrophotometers are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file.

11. Data Evaluation, Calculations and Reporting

Calculate the concentration value of the sample directly from the standard curve. (Section 10.2).

$$\text{mg } P_{Total}/L = \frac{\text{absorbance} - \text{y-intercept}}{\text{slope}} \times \text{Dilution factor}$$

If samples were filtered prior to preservation, report as mg $P_{Dissolved}$ / L.

For soil samples, convert results to mg/kg, by multiplying result in mg/l by extraction volume and dividing by weight. All results must by reported based on dry weight.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedances or improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CCV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

The quarterly method reporting limit (MRL) verification requirement is satisfied by the quarterly LOQ analysis, as per SOP/1732.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

Total Solids in Solid and Semisolid Samples

(Percent Solids)

Reference Method: **SM 2540 G,** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

1. Scope and Application

Matrices: Soils, solids and sludges.

Definitions: See Alpha Analytical Quality Manual

This method is applicable to the determination of total solids in such solid and semisolid samples as river and lake sediments, sludges separated from water and wastewater treatment processes, and sludge cakes from vacuum filtration, centrifugation, or other sludge dewatering processes.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results by completing an initial demonstration of capability

2. Summary of Method

A homogenized aliquot of sample is weighed in a tared dish and set in a 103º - 105ºC oven until dry. The sample and dish are cooled and re-weighed, thus the percent of solids in the original sample can be calculated.

2.1 Method Modifications from Reference

Aluminum pans are used instead of porcelain dishes. However, if the sample is corrosive, then the porcelain dishes are used.

5-10 grams of sample are used for soil and solid matricies.

One duplicate is analyzed per batch of 20 samples or less.

3. Reporting Limits

The Reported Detection Limit is 0.1%.

4. Interferences

4.1 Humidity: Humidity in the laboratory may cause samples to pick up moisture. When not being weighed, samples should be kept tightly capped or in a dessicator.

4.2 Large rocks / debris: Large rocks or debris may cause false high results and therefore should not be included in the sample aliquot.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents. Personal protective equipment is to be worn at all times within the laboratory areas. At a minimum, a labcoat, gloves and safety glasses are worn.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples are collected in glass or plastic containers with minimal headspace. Containers are covered immediately to minimize the loss of sample moisture.

6.2 Sample Preservation

Samples are refrigerated at 4 ºC.

6.3 Sample Shipping

No special shipping requirements.

6.4 Sample Handling

Samples are kept refrigerated at 4 ºC until the time of analysis.

For samples received, marked and commented as **Foreign Soils** reference SOP 2296 Treatment of Foreign Soils.

For samples received, marked and commented as **Containing or May contain Asbestos** reference WI 2535 Asbestos Handling Procedures.

7. Equipment and Supplies

7.1 Analytical Balance: Capable of weighing to 0.01g

7.2 Aluminum Weighing Dishes or Pans

7.3 Porcelain Evaporation Dishes

7.4 Dessicator: With a color-indicator dessicant.

7.5 Drying Oven: Capable of maintaining 103 – 105 ºC.

7.6 Oven Trays

7.7 Computer: with connection to LIMS and the Analytical Balance (Sect. 7.1)

7.8 Baked Sand: created by and available in the extractions department

8. Reagents and Standards

None.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

One blank is analyzed per batch of 20 samples or less from the state of Maine. Blank recovery must be between 95%-105%. If this criterion is not met, the sample and it's duplicate are reanalyzed. Sand, baked to dryness in the extractions laboratory, is used for the blank sample.

9.2 Laboratory Control Sample (LCS)

Not applicable.

9.3 Initial Calibration Verification (ICV)

Not applicable.

9.4 Continuing Calibration Verification (CCV)

Not applicable.

9.5 Matrix Spike

Not applicable.

9.6 Laboratory Duplicate

One duplicate is analyzed per batch of 20 samples or less. Duplicate determinations must agree within 20%. If this criterion is not met, the sample and it's duplicate are reanalyzed.

If sample, used for batch duplicate, is non-homogeneous, then data may be reported with a narrative.

9.7 Method-specific Quality Control Samples

Not applicable.

9.8 Method Sequence

- Create a Workgroup in LIMS
- Locate Samples and take possession of them in LIMS
- Create an ELN for the Workgroup
- Homogenize Sample
- Record the Tare weight of the evaporation dish, then the Gross weight for each sample one at a time
- Dry samples in the oven 3+ hours.
- Cool samples in the dessicator
- Record the Net Weight (1)
- Dry samples again for 1+ hour(s), unless samples were originally dried overnight. Due to running of analysis 24 hours a day overnight is interpreted to mean a minimum of 5 hours.

Samples from the State of Maine logged for ME-TS-2540 must always be dried again regardless of the length of time dried.

- Cool samples in the dessicator
- Record the Net Weight (2)
- Save and Send Data to LIMS

10. Procedure

10.1 Equipment Set-up

10.1.1 LIMS Knowledge: Prior to utilizing this SOP, the analyst must first be familiar with the operation of the Laboratory Information Management System (LIMS) and the generation of a sample batch or workgroup.

10.1.2 Porcelain Dish Preparation: Porcelain evaporation dishes are used only if a sample is corrosive to aluminum. To prepare the porcelain dishes, bake them in the 103 – 105 ºC drying oven for a minimum of 1 hour before placing them in the dessicator. Cool in the dessicator for a minimum of one hour.

10.1.3 Matrix and Volume Evaluation: Total Solids analysis should not be performed on plastic, caulking or oil. If logged for total solids analysis see login staff for assistance in altering the login in LIMS.

When taking an aliquot of sample for analysis, it is important that the sample volume is not exhausted to perform total solids before all other analysis has been performed. If the container has analysis printed on the alpha applied label other than total solids, ensure those tests have been performed prior to utilizing the remainder of the sample for total solids analysis.

10.2 Initial Calibration

Not applicable.

10.3 Equipment Operation and Sample Processing

10.3.1 Prioritizing Work and Selecting Samples to Analyze

Using the Pivot Table located in the Login folder on the network drive (Login_TS_#.#) identify the next samples to be analyzed. Rushes must be prioritized by due date and priority level (P1, P2, P3 or null), followed by non-rush samples by due date and priority level. Gather the samples to be run from sample custody prior to batching into workgroups.

10.3.2 Generating a LIMS Batch

Utilizing a computer (Sect. 7.7), generate a LIMS batch of samples and assign a Workgroup (WG) number to the batch. When generating the batch, choose a sample that will be duplicated. Print out a copy of the LIMS batchsheet. For samples from the state of Maine, a blank must be analyzed. Use the “Scan to Batch” function in LIMS to scan the barcodes of samples you are batching to ensure no typographical errors are made. When selecting the DUP (and Blank for samples from Maine) also use the barcode scanner to ensure accuracy.

10.3.2 Creating a Gravimetric Laboratory Notebook

From the menu bar in LIMS select: “Sample Data”, “Data”, “Logbooks”, “ELN-Wet Chem” and enter the workgroup number in the space provided. If this is the first time you have attempted to open a lab notebook for this workgroup a window will pop up confirming you wish to create a new gravimetric laboratory notebook.

Once you create the laboratory notebook the samples will populate in the spreadsheet with the Duplicate at the top. Above the list of samples. The reference sample for the Duplicate can be confirmed by double clicking the Sample Number of the Duplicate.

10.3.3 Taking the Tare Weight

10.3.3.1 With the cursor on the tare weight field in the ELN, weigh the corresponding empty dish on a tared balance. When the weight is stable, push the “Print” button on the balance. This will transfer the weight of the empty dish into the ELN.

10.3.4 Taking the Gross Weight

10.3.4.1 Homogenize the sample by mixing with a spatula or spoon.

10.3.4.2 Remove a 5 – 10g aliquot of soil sample or 20 – 25g of a sludge sample and place it in the weighing dish.

10.3.4.3 With the cursor on the cell for the “Gross Weight” corresponding to the sample to be weighed, zero the balance and weigh the dish plus the sample. When the weight is stable, push the “Print” button on the balance. This will transfer the weight into the ELN.

10.3.4.4 Place the dish onto an oven tray.

10.3.5.5 Once the samples have all been weighed and the weights recorded in the ELN, click save.

10.3.6 Drying the Samples : Phase I

10.3.6.1 Place the oven tray in the 103 – 105 ºC drying oven. In the ELN double click the Time In (1) field to populate the current time, edit as needed to reflect the time the samples were placed in the oven. Enter the temperature reading on the oven into the Temperature field.

10.3.6.2 After a minimum of three hours (samples with state of origin ME must be dried at least five hours), if samples appear dry, move the oven tray of dried samples to a dessicator. Allow to cool completely.

10.3.7 Taking the First Net Weight

10.3.7.1 In the ELN with the cursor on the “Net Weight (1)” field corresponding to the sample to be weighed, zero the balance and weigh the dish plus the sample. When the weight is stable, push the “Print” button on the balance. This will transfer the weight into the ELN.

10.3.7.2 Repeat Section 10.3.7.1 until the Net Weight (1) for all samples has been recorded.

10.3.7.3 Click on the “Save” button to save the weights.

10.3.8 Drying the samples : Phase II

10.3.8.1 If the samples were dried in the oven for less than five (but at least three) hours, the tray of samples must be placed back in the 103 – 105 ºC drying oven for a minimum of one hour. If the samples are from the state of Maine the samples must always be placed back in the oven for a minimum of one hour.

10.3.8.2 After drying, move the oven tray of samples to a dessicator. Allow to cool completely.

10.3.9 Taking the Second Net Weight

10.3.9.1 In the ELN with the "Net Weight(2)" field selected corresponding to the sample to be weighed, zero the balance and weigh the dish plus the sample. When the weight is stable, push the "Print" button on the balance. This will transfer the weight into the ELN.

10.3.9.2 Repeat Sections 10.3.9.1 until the Net Weight (2) for all samples has been recorded.

10.3.9.3 Click on the "Save" button to save the weights.

10.3.9.4 If Net Weight (1) and Net Weight (2) are within 4% or 50mg, the ELN will highlight the values light pink and display. If this is the case for all samples in the batch, proceed to Section 10.3.10.

10.3.9.5 If Net Weight (1) and Net Weight (2) are not within 4% or 50mg the ELN will highlight the values red and the words "Not Acceptable" will be displayed, repeat Sections 10.3.8 and 10.3.9 for those samples. This will allow the chemist to record a Net Weight (3), (4) or (5), until the values are within the acceptable range.

If the samples have been dried ≥ 24 hours, and the weights are still not within 4% or 50mg, consult the Department Supervisor as to how to proceed.

10.3.10 Saving the Batch

10.3.10.1 Click on the "Save" button to save the weights in the ELN.

10.3.10.2 Click on the "Send to LIMS" button in the ELN.

10.4 Continuing Calibration

Not applicable.

10.5 Preventative Maintenance

The temperature of the laboratory ovens is recorded constantly on the data logger. The chart recorder and the laboratory ovens are calibrated on an annual basis by an instrument service company. Certificates are kept on file.

Analytical balances are calibrated on a semi-annual basis by an instrument service company. Certificates are kept on file. The calibration of the balances is verified on a daily basis and records are kept in a Logbook.

11. Data Evaluation, Calculations and Reporting

The Excel Spreadsheet is programmed to calculate the Percent Solids results. This is the formula that is used for calculation:

$$\% \text{ Total Solids} = \frac{(A - B)}{} \times 100$$

(C - B)

Where: A = Final Net Weight (weight of dried residue + dish, g)
B = Tare weight (weight of dish, g)
C = Initial Gross Weight (weight of wet sample + dish, g)

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Improper preservation is noted on the Sample Delivery Group form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial **(IDC)**

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Unless the containers are labeled as hazardous material (i.e. low flashpoint, ignitable, containing asbestos or high levels of toxic materials), the dried samples are disposed of into the trash.

If sample containers are labeled as hazardous, refer to the Chemical Hygiene Plan for waste handling and disposal instructions.

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

WI 2535 Asbestos Handling Procedures

SOP 2296 Treatment of Foreign Soils

16. Attachments

None.

Total Sulfide, Methylene Blue Method

References: **SM 4500S2-D:** Standard Methods for the Examination of Water and Wastewater. APHA-AWWA-WEF. Standard Methods Online.

EPA 9030B: SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, 1997.

1. Scope and Application

Matrices: This method is applicable to water, wastewater and seawater samples.

Definitions: Refer to Alpha Analytical Quality Manual.

Sulfide often is present in groundwater, especially in hot springs. Its common presence in wastewaters comes partly from the decomposition of organic matter, sometimes from industrial wastes, but mostly from the bacterial reduction of sulfate. Hydrogen sulfide escaping into the air from sulfide-containing wastewater causes odor nuisances. The threshold odor concentration of H_2S in clean water is between 0.025 and 0.25µg/L. H_2S is very toxic and has claimed the lives of numerous workers in sewers. It attacks metals directly and has indirectly caused serious corrosion of concrete sewers because it is oxidized biologically to H_2SO_4 on the pipe wall.

From an analytical standpoint, three categories of sulfide in water and wastewater are distinguished:

- Total sulfide includes dissolved H_2S and HS-, as well as acid-soluble metallic sulfides present in suspended matter. The S_{-2} is negligible, amounting to less than 0.5% of the dissolved sulfide at pH 12, less than 0.05% at pH 11, etc. Copper and silver sulfides are so insoluble that they do not respond in ordinary sulfide determinations; they can be ignored for practical purposes.
- Dissolved sulfide is that remaining after suspended solids have been removed by flocculation and settling.

Un-ionized hydrogen sulfide may be calculated from the concentration of dissolved sulfide, the sample pH, and the practical ionization constant of H_2S.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

Sulfide is separated from the sample matrix by suspending the sample in a hydrochloric acid solution and vigorous agitation. Hydrogen sulfide gas is released from the sample and collected in a gas scrubbing bottle containing sodium hydroxide. The methylene blue method is based on the reaction of sulfide, mild oxidizing agent potassium dichromate, and dimethyl-p-phenylenediamine to produce methylene blue.

2.1 Method Modifications from Reference

Soil samples don't require preservation and have hold time of 7 days

For water samples: whole sample is filtered and filter with sediment is being analyzed.

Lab is purchasing commercially available coloring reagents from HACH.

Lab is using DI instead of dagassed reagent water.

3. Reporting Limits

The reported detection limit is 0.1 mg/L for liquid samples and 0.5 mg/kg for soils samples

4. Interferences

4.1 Strong reducing agents interfere in the methylene blue test by preventing formation of the blue color. Thiosulfate at concentrations about 10mg/L may retard color formation or completely prevent it. Sulfide itself prevents the reaction if its concentration is very high, in the range of several hundred milligrams per liter. To avoid the possibility of false negative results, use the antimony method to obtain a qualitative result in industrial wastes likely to contain sulfide but showing no color by the methylene blue method. Iodide, which is likely to be present in oil-field wastewaters, may diminish color formation if its concentration exceeds 2mg/L. Ferrocyanide produces a blue color; distillation will minimize interference.

4.2 Eliminate interferences due to sulfite, thiosulfate, iodide, and many other soluble substances (but not ferrocyanide), by precipitating ZnS.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Take samples with minimum aeration in either a glass or plastic bottle. Fill the bottle completely and cover. Aqueous samples are collected in a 250mL plastic bottle, soil/solid samples are collected in an amber glass container.

6.2 Sample Preservation

All samples must be preserved with 2N zinc acetate (Section 8.5) and 6N sodium hydroxide (Section 8.6).

Use four drops of 2N zinc acetate solution per 100mL aqueous sample. Adjust the pH to greater than 9.0 with 6N NaOH.

Soils require no preservative.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Samples are stored refrigerated at 4 ± 2° C. Aqueous and soil samples are analyzed within a 7-day holding time.

7. Equipment and Supplies

7.1 Spectrophotometer, Genesys 10vis (or equivalent): For use at a wavelength of 664nm with cells providing light paths of 1cm.

7.2 Test Tubes (centrifuge tubes): 25mL capacity

7.3 Pipets: Glass, volumetric, various volumes or Auto-pippets.

7.4 Volumetric Flasks: 100mL and 1L volumes, 250 ml ,200 ml and 50 ml

7.5 Erlenmeyer Flasks: 125mL volume

7.6 Three-neck Flask: 500mL volume, 24/40 standard taper joints.

7.7 Dropping funnel: 250mL volume, 24/40 joint.

7.8 Purge gas inlet tube: 24/40 joint with coarse frit.

7.9 Purge gas outlet: 24/40 joint reduced to ¼ in. tube.

7.10 Gas scrubbing bottles: 125mL volume, with ¼ inch o.d. inlet and outlet tubes.

7.11 Tubing: ¼ inch o.d. polytetrafluoroethylene (PTFE) or polypropylene.

7.12 Magnetic Stirrer

7.13 Nitrogen gas: 5.0 Ultra pure.

7.14 Nitrogen regulator

7.15 Flowmeter

7.16 Teflon-coated stirring bar

7.17 Analytical Balance: Capable of weighing 0.1g.

7.18 Glass fiber filter disks: 90mm

7.19 Filtration Apparatus: With membrane filter funnel.

7.20 Suction flask

7.21 Vacuum pump

7.22 Graduated cylinders

8. Reagents and Standard

8.1 2 different sources. Stock Sulfide Solution, approximately 1097 mg/L: In 200 ml volumetric flask weigh 1.5779 grams of $Na_2S \cdot 9H_2O$ (Section 8.8) and bring volume up to 200 ml of DI Store in the refrigerator. Final concentration is 1097 mg/L. Stable for 2 weeks.. Standard has to be re-made, if degradation of LCS and ICV is noticeable.

8.1.1 LCS/ICV/CCV/ Spike Working Solution (65.82 mg/l): In 50 ml volumetric flask add 10 ml of 1N NAOH (8.6) solution, then adds 3 ml of Stock Sulfide Solution (8.1), bring volume up to 50 ml with DI. Gently mix; can not be mixed vigorously. Different stocks are used to prep ICV/ LCS solution and CCV / Spike solution. Solution is stable for one week.

8.1.2 Intermediate calibration standard 10.97 mg/l: Pipet 1 ml of Stock Sulfide Calibration Solution (8.1) into 100ml volumetric flask. Bring volume up to 100ml with DI. Make daily.

8.2 Sulfide 1 Reagent: Available commercially from HACH Company. Store at 4 ± 2°. Reagent expires upon manufacturer's specified date.

8.3 Sulfide 2 Reagent: Available commercially from HACH Company. Store at 4 ± 2°. Reagent expires upon manufacturer's specified date.

8.4 Sulfide Calibration Solutions: These solutions are used to generate the calibration curve in Section 10.1. Prepare a series of sulfide solutions by diluting the Intermediate Calibration Sulfide Standard second source (Section 8.1.2). These solutions are prepared fresh prior to generating a calibration curve.

Into seven 100mL volumetric flasks containing 20ml of 1N NaOH pipet 0mL, 0.5 ml 1mL, 2.0mL, 4mL, and 8mL,12 ml and 16 ml of Intermediate Calibration Sulfide Solution (Section 8.1.2). Bring each to volume with DI.

8.5 Zinc Acetate Solution: Dissolve 220g $Zn(C_2H_3O_2)_2 \cdot 2H_2O$ in 500mL water. This solution is used as the preservative during sampling. Store at room temperature. Solution is stable for one year.

8.6 Sodium Hydroxide Solution, NaOH, 1N, 6N and 0.2N: For 1N NaOH, dissolve 40g NaOH in 1L of DI water. For 6N, dissolve 240g NaOH in 1L of DI water. This solution is used as a preservative during sampling. Store at room temperature. Solution is stable for 6 month. For 0.2N NaOH, add 200 ml of 1.0 N NaOH in 1L of DI water. Stable for 1 month.

8.7 HCL Solution, 2N: Add 175mL concentrated HCL (Section 8.10) to 500mL of DI water. Dilute to 1L with DI water. Store at room temperature. Make daily.

8.8 $Na_2S \cdot 9H_2O$: Store at 4 +/- 2 degrees C, out of direct light.

8.9 Concentrated HCL: Store at room temperature.

8.10 Ottawa sand or equivalent.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blanks

9.1.1 **Method Blank:** Distill and analyze one Method Blank of DI water (for liquid samples) or 10g of Ottawa sand (for soil samples) per batch of 20 samples or less. The Method Blank results must be less than the Reporting Limit. Refer to Section 12 for any corrective actions.

9.1.2 **ICB/CCB:** The ICB/CCB is not distilled. It consists 25 ml of 0.02N NaOH and is analyzed following each ICV/CCV. Results must be less than the Reporting Limit. Refer to Section 12 for any corrective actions.

9.2 Laboratory Control Sample (LCS)

Distill and analyze one LCS (Section 8.1.1) per batch of 20 samples or less.

For waters: in 250 ml volumetric flask add approximately 50 ml of DI, 6 drops on 2N Zn Acetate, swirl and add 1 ml of LCS working Solution (8.1.1), adjust pH with 6N NaOH solution (@ 6 drops) to pH greater then 9. Bring volume up to 250 ml with DI. Let solution to sit for several minutes prior to preparation (filtration).

For soils: weigh 10g of Ottawa sand and add 1ml of LCS working solution (8.1.1) directly to reaction flask.

Follow distillation and colorimetric process.

% Recovery must be within 75 – 125%. Refer to Section 12 for any corrective actions.

9.3 Initial Calibration Verification (ICV)

The ICV is not distilled. In 100 ml volumetric flask add 20 ml of 1N NAOH solution, 1 ml of LCS working solution; bring volume up to 100 ml with DI. Done fresh prior to analysis

ICV is analyzed at the beginning of analytical batch.

% Recovery must be within 75 – 125%. Refer to Section 12 for any corrective actions.

9.4 Continuing Calibration Verification (CCV)

The CCV is not distilled. In 100 ml volumetric flask add 20 ml of 1N NAOH solution, 1 ml of CCV working solution; bring volume up to 100 ml with DI. Done fresh prior to analysis. It consists of the CCV Standard (Section 8.1.1) and is analyzed after every 10 samples and at the end of the batch.

% Recovery must be within 75 – 125%. Refer to Section 12 for any corrective actions.

9.5 Matrix Spike

Prepare and analyze one matrix spike per batch of 20 samples or less. The matrix spike consists of the 2ml of Working spike solution (8.1.1) added directly to reaction flask prior to distillation. % Recovery must be within in-house control limits. Refer to Section 12 for any corrective actions.

9.6 Laboratory Duplicate

Prepare and analyze one sample in duplicate per batch of 20 samples or less. %RPD must be $\leq$ 25%. Refer to Section 12 for any corrective actions.

9.7 Method-specific Quality Control Samples

Upon client specification

9.8 Control Limits

The laboratory maintains performance records to document the quality of data that is generated. Method accuracy for samples is assessed and records maintained. After the analysis of 20 spiked samples, and 20 laboratory control samples, calculate the average percent recovery (R) and the standard deviation of the percent recovery (S).

Control limits for the method parameters are generated. The control limits are calculated based on in-house performance data. The limits are compared to the control limits found in the reference method.

9.9 Method Sequence

- Generate a calibration curve.(done once a year)
- Filter water samples or weigh out soil samples
- Add turned filters (for water samples) or aliquot of soil to reaction flask
- Assemble distillation system and distill for at least an hour.
- Disassemble distillation unit and bring distillate up to final volume.
- Start colorimetric procedure.
- Add 1mL Sulfide 1 Reagent to 25mL of sample and swirl gently to mix.
- Add 1mL of Sulfide 2 Reagent.
- Wait 5 – 15 minutes.
- Read the absorbance of samples and QC samples.
- Calculate results.

10. Procedure

10.1 Initial Calibration

Prepare a calibration curve using the selected standards from Section 8.4

Analyze the standards as in Section 10.2.2 and 10.2.3 below, and plot concentration vs. absorbance. Determine the slope and the y-intercept. The correlation coefficient must be $\geq$ 0.995. A calibration curve is generated once a year of when significant changes are noted.

10.2 Equipment Operation and Sample Processing

10.2.1 Sample Preparation

10.2.1.1 **Soil samples:** Weigh 10 grams of the sample add to the reaction flask with a clean stir bar. Record the weight on excel form/ prep book.

10.2.1.2 **Aqueous samples:** Filter entire sample volume through a 90mm glass fiber filter and rinse with DI water. Place filter into the reaction flask with a clean Teflon stir bar. Record the volume of sample filtered on excel form/ prep book.

10.2.1.3 Add 200mL of 2N HCL Solution (Section 8.7) into the thistle tube, making sure that the stopcock is closed.

10.2.1.4 Add 20mL of 1N NaOH (Section 8.6) and 50mL of DI to the 250mL collection vessel.

10.2.1.5 Assemble the distillation train.

10.2.1.6 Connect the thistle flask to the center position of the reaction flask

10.2.1.7 Connect the collection vessel to the left side of the reaction flask, and the impinger tube to the collection vessel. The impinger tube must be below the surface of the NaOH solution. Make sure that all ground glass joints are sealed.

10.2.1.8 Connect the Nitrogen gas to the right side of the collection vessel. The gas inlet must be below the surface of the reaction flask solution.

10.2.1.9 Making sure that the train is a closed system, purge the train with Nitrogen for 5 minutes. Open the valve on the Nitrogen tank (Section 7.13) and open up the regulator so that 45 psi of Nitrogen is flowing (this will effectively give a flow rate of about 150mL/min).

10.2.1.10 Once the system has been purged for 5 minutes, open the stopcock fully on the thistle tube and release the 2N HCL solution into the reaction vessel. At this point the sample should be stirring. Close the stopcock as soon as all of the 2N HCL solution has been delivered to the reaction flask. Let the reaction progress for 1 hour.

10.2.1.11 After one hour has passed, dissemble the train and bring the total volume of the collection vessel to 100mL with DI**.**

10.2.2 Color Development: Transfer 25mL sample to a test tube. Add 1.0mL Sulfide 1 reagent (Section 8.2) and swirl to mix. Add 1mL of Sulfide 2 reagent (Section 8.3). Let the reaction continue for 5 minutes. Excessive mixing causes low results by loss of H_2S as a gas before it has had time to react. The presence of sulfide will be indicated by the appearance of blue color. Color development usually is complete in about 1 minute, but a longer time is often required for fading out of the initial pink color. Wait 5 to 15 minutes and make color comparisons. If zinc acetate was used, wait at least 10 minutes before making a visual color comparison.

10.2.3 Absorbance Determination: Set the spectrophotometer to a wavelength of 664nm. Zero the instrument with a portion of the reagent blank sample using a 1 cm cell. Read the absorbance of the samples and QC samples and record this information on the excel format.

If the absorbance is greater than the highest standard in the calibration curve, the sample must be diluted with 0.2N NaOH and reanalyzed. Record on excel format any dilutions that are prepared.

10.3 Continuing Calibration

See Sections 9.4

10.4 Preventive Maintenance

An instrument service company calibrates the spectrophotometers on a semi-annual basis. Certificates are kept on file.

11. Calculations

Calculate sulfide concentration using the calibration curve generated in Section 10.1:

$$\text{Sulfide (mg/L)} = \frac{\text{Absorbance} - \text{y intercept}}{\text{Slope}} \times \text{Dilution factor}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance and improper preservation are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the ICV, CCV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

If the Method Blank result is greater than the reporting limit, and all associated sample results are non-detect, or greater than 10x the concentration of the Method Blank, the data may be reported and a narrative submitted for inclusion on the final report. If associated sample results are positive, but at a concentration less than 10x the concentration of the Method Blank, those samples are re-distilled and reanalyzed.

If the LCS fails acceptance criteria, the entire batch must be redigested and reanalyzed. If there is insufficient sample volume to redigest, then the Client Services Department is notified and the sample results are reported as estimated, with a narrative to be included on the final report.

If the Matrix Spike fails acceptance criteria, the non-conformance is narrated and included on the final report.

If %RPD for the Duplicate fails acceptance criteria, the non-conformance is narrated and included on the final report.

13. Method Performance

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/1732 These studies performed by the laboratory are maintained on file for review.

The quarterly method reporting limit (MRL) verification requirement is satisfied by the quarterly LOQ analysis, as per SOP/1732.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 MDL/LOD/LOQ Generation
SOP/1739 IDC/DOC Generation
SOP/1728 Waste Management and Disposal SOP

16. Attachments

None.

TPH- Diesel Range Organics

References: **Maine 4.1.25,** Maine Health and Environmental Testing Laboratory, 09/06/1995.

EPA 8015D (Modified), SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, June 2003.

1. Scope and Application

Matrices: Diesel Range Organics can be determined in water, soil, sludge and product samples.

Definitions: See Alpha Laboratories Quality Manual Appendix A

TPH-DRO is used to identify and determine the concentration of Diesel Range Organics. This corresponds to the hydrocarbon range of C10 – C28, inclusive (Alpha LIMS products Maine-DRO and TPH-DRO). Alpha also reports the hydrocarbon ranges of C10 – C36 as (Alpha LIMS modified product TPH-DRO-D) and C9 – C44 as (Alpha LIMS modified product TPH-DROD-C44) by this method. TPH-DRO-D can be also used to report Oil Range Organics (ORO), C20-C36, and Residual Range Organics (RRO), C24-C36, TPH C10-C20 and TPH C20-C34.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of trained analysts experienced in the operation of the Gas Chromatograph and in the interpretation of Gas Chromatograph data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

TPH-DRO provides GC conditions for the detection petroleum fractions. Prior to use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids may be analyzed by direct injection. A 1µL or 2µL aliquot of the extract is injected into a GC using the solvent flush technique, and compounds are detected by a FID.

2.1 Method Modifications from Reference, EPA 8015D

2.1.1 Alkane Standard Calibration is performed instead of Fuel Oil Calibration.

2.1.2 Quality control samples consist of a Blank, LCS and a Duplicate. MS/MSD is performed upon Client request only.

2.1.3 Alpha LIMS product TPH-DROD product includes C10-C36 and TPH-DROD-C44 product includes C9-C44.

3. Reporting Limits

The reported detection limit for this procedure as outlined is as follows:

- For the Maine DRO Method: 50µg/L for aqueous samples and 3.33mg/kg for soil/solid samples.
- For TPH-DRO and TPH-DROD-C44 Methods: 500µg/L for aqueous samples and 33.3mg/kg for soil/solid samples. For TPH-DROD: 200µg/L for aqueous samples and 33.3mg/kg for soil/solid samples.

4. Interferences

Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

Interferences co-extracted from samples will vary considerably from source to source, depending upon the waste being sampled.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound must be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material data handling sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Samples must be collected in glass containers with Teflon-lined caps. Plastic containers or lids may not be used for the storage of samples due to the possibility of sample contamination from phthalate esters and other hydrocarbons within the plastic.

Samples must be kept cool until receipt at the Laboratory.

6.2 Sample Preservation

Samples for Maine DRO are preserved with 1:1 HCl to a pH < 2. Samples for TPH-DRO, TPH-DRO-D and TPH-DROD-C44 are not preserved.

6.3 Sample Handling

Samples must be stored under refrigeration to 4 ± 2°C.

Liquid samples must be extracted within 7 days from collection, and soils must be extracted within 14 days from collection.

The extracts must be stored under refrigeration and must be analyzed within 40 days of extraction.

7. Equipment and Supplies

7.1 Gas Chromatograph, Hewlett Packard 6890 (or equivalent): An analytical system suitable for on-column injections. All required accessories, including: detectors, column supplies, recorder, gases and syringes. A data system for measuring peak area and/or peak heights.

7.2 GC Columns
Column 1: RTX-5 – 30m x 0.32mm ID, 0.25µm
Column 2: RTX-5MS – 30m x 0.32mm ID, 0.25µm

7.3 Class A Volumetric Flask: 10, 50, and 100mL with ground glass stopper.

7.4 Microsyringe: 10µL.

7.5 Helium: Compressed Helium, 5.0 ultra high purity.

7.6 Pipets: 9 inch glass pipets.

7.7 Analytical Balance: 0.0001g sensitivity

7.8 Pasteur Pipets: Borosilicate glass

7.9 Autosampler vials: 2mL volume with Teflon-lined septum crimp caps.

8. Reagents and Standards

8.1 Solvents, pesticide quality: Hexane, methylene chloride, acetone and pentane. Store in a flammables cabinet. Expires upon manufacturer's specified date.

8.2 o-Terphenyl, 2000µg/mL Stock Standard in methylene chloride: Vendor purchased stock solution used to prepare calibration standards. This solution is used only for Calibration Standards preparation. Expires upon manufacturer's specified date. Store at room temperature.

8.3 99.5% pure Nonane: Vendor purchased. Two sources are purchased. One source is used for the calibration standard and the other source is used for the continuing calibration standard. Store per vendor recommendations. Expires upon manufacturer's specified date.

8.4 Combination of CT-ETPH Alkane Standard and n-tetratetracontane, 1000ug/mL: Vendor purchased stock solution. This solution is used for LCS/MS spiking solution preparation. Expires upon manufacturer's specified date if unopened. Once opened, standard expires 6 months from the date of opening or the manufacturer's specified date, whichever is sooner. Store 20± 2°C.

8.5 Stock Calibration Standards

8.5.1 n-Nonane (c9), 2000µg/mL Stock Working Standard: To a 50mL volumetric flask, add 140µL of vendor purchased 99.5% pure Nonane (Section 8.3). Bring to volume with methylene chloride. Standard expires 6 months from the date of preparation. Store in the refrigerator at 4 ± 2°C.

Use one source for the calibration standards and the other source for the continuing calibration standards.

8.5.2 Calibration/Window defining Hydrocarbon Stock Standard, 1000µg/mL. Vendor purchased stock solution used to prepare calibration standards. Two sources are purchased: One is used for the Calibration Standards, and the other is used for the Continuing Calibration Standards. Contains the following alkanes in methylene chloride:

n-Octane	(c8)	n-Hexacosane	(c26)
n-Decane	(c10)	n-Octacosane	(c28)
n-Dodecane	(c12)	n-Triacontane	(c30)
n-Tetradecane	(c14)	n-Dotriacontane	(c32)
n-Hexadecane	(c16)	n-Tetratriacontane	(c34)
n-Octadecane	(c18)	n-Hexatriacontane	(c36)
n-Eicosane	(c20)	n-Octatriacontane	(c38)
n-Docosane	(c22)	n-Tetracontane	(c40)
n-Tetracosane	(c24)		

Expires upon manufacturer's specified date if unopened. Once opened, standard expires 6 months from the date of opening or the manufacturer's specified date, whichever is sooner. Store in the refrigerator at 20± 2°C .

NOTE: n-Octane (c8), and c38 through c40 are not used in the calculation of the DRO calibration standards, but are present in the solution.

The calibration/window defining hydrocarbon standard also serves: a) to demonstrate sufficient separation between the solvent and C8-alkane peak, b) to ensure that the last component, C36, is eluted within a reasonable amount of time (30minutes), and c) to serve as a retention time marker for oil identification.

8.5.3 n-Tetratetracontane Stock Working Standard: Vendor purchased stock solution used to prepare calibration standards. Two sources are purchased: One is used for the Calibration Standards, and the other is used for the Continuing Calibration Standards. Contains n-Tetratetracontane in carbon disulfide (c44).

Expires upon manufacturer's specified date if unopened. Once opened, standard expires 6 months from the date of opening or the manufacturer's specified date, whichever is sooner. Store in the refrigerator at 20± 2°C .

8.6 Working Calibration Standards: **(Also refer to Table 1)**

Working calibration standards are stored in the refrigerator at 4 ± 2°C and expire 6 months from the date of preparation.

8.6.1 Level 1 Calibration Standard for TPH-DRO/TPH-DRO-D (3µg/mL): To 994uL of methylene chloride add 6 uL of L6 calibration standard (Section 8.6.6)

8.6.2 Level 2 Calibration Standard for TPH-DRO/TPH-DRO-D (20µg/mL): To 960 uL of methylene chloride add 40 uL of L6 calibration standard (Section 8.6.6)

8.6.3 Level 3 Calibration Standard for TPH-DRO/TPH-DRO-D (50µg/mL): To 900 uL of methylene chloride add 100 uL of L6 calibration standard (Section 8.6.6)

8.6.4 Level 4 Calibration Standard for TPH-DRO/TPH-DRO-D (100µg/mL): To 800 uL of methylene chloride add 200 uL of L6 calibration standard (Section 8.6.6)

8.6.5 Level 5 Calibration Standard for TPH-DRO/TPH-DRO-D (200µg/mL): To 600 uL of methylene chloride add 400 uL of L6 calibration standard (Section 8.6.6)

8.6.6 Level 6 Calibration Standard for TPH-DRO/TPH-DRO-D (500µg/mL): To a 5mL volumetric flask add 1.25mL of n-Nonane Stock Solution (Section 8.5.1) and 1.25mL o-Terphenyl I Stock Solution (Section 8.2) and 2.5mL of Calibration/Window Defining Hydrocarbon Solution (Section 8.5.2).

8.7 Working Calibration Standards: (Also refer to Table 2)

Working calibration standards are stored in the refrigerator at 4 ± 2°C and expire 6 months from the date of preparation.

8.7.1 Level 1 Calibration Standard for TPH-DROD-C44 (3µg/mL): To 988uL of methylene chloride add 12 uL of L5 calibration standard (Section 8.6.6)

8.7.2 Level 2 Calibration Standard for TPH-DROD-C44 (20µg/mL): To 920 uL of methylene chloride add 80 uL of L5 calibration standard (Section 8.6.6)

8.7.3 Level 3 Calibration Standard for TPH-DROD-C44 (50µg/mL): To 800 uL of methylene chloride add 200 uL of L5 calibration standard (Section 8.6.6)

8.7.4 Level 4 Calibration Standard for TPH-DROD-C44 (100µg/mL): To 600 uL of methylene chloride add 400 uL of L5 calibration standard (Section 8.6.6)

8.7.5 Level 5 Calibration Standard for TPH-DROD-C44 (250µg/mL): To a 5mL volumetric flask add 1.00mL of n-Nonane Stock Solution (Section 8.5.1) and 1.00mL o-Terphenyl Stock Solution (Section 8.2) and 2.00mL of Calibration/Window Defining Hydrocarbon Solution (Section 8.5.2) and 2.00mL of n-Tetratetracontane (Section 8.5.3).

8.7.6 Level 6 Calibration Standard for TPH-DROD-C44 (500µg/mL): To a 10mL volumetric flask add 2.50mL o-Terphenyl Stock Solution (Section 8.2).

8.8 o-Terphenyl: Vendor purchased. Used to prepare extraction surrogate. Expires upon manufacturer's specified date. Store in the refrigerator at 4 ± 2°C.

8.8.1 Surrogate Standard Solution, 330ug/L: To a 500mL volumetric flask, add 15mL of 10000ug/mL o-terphenyl solution. Bring to volume with acetone. The surrogate standard solution expires 6 months from the date of preparation. Store in the refrigerator at 4 ± 2°C.

8.9 LCS / MS spiking solution, 100µg/mL

To a 100mL volumetric flask, add 10mL of the CT-ETPH Alkane Standard (Section 8.4) and 10mL of second source of n-Tetratetracontane (Section 8.5.3) using a 1mL syringe. Bring to

volume with pentane. This solution is stored in a refrigerator at 4 ± 2°C and expires 6 months from the date of preparation.

8.10 Continuing Calibration Standards

8.10.1 n-Nonane (c9), 2000µg/mL Stock Working Standard: Refer to Section 8.5.1.

8.10.2 Continuing Calibration Standard, 100µg/mL: To a 50mL volumetric flask, add 5mL of second source, vendor purchased 1000ug/mL o-Terphenyl solution, 2.5mL Nonane solution (Section 8.5.1 alternate source) and 5mL of Calibration/Window defining Hydrocarbon Stock Standard (1000µg/mL, Section 8.5.2) and add 5mL of second source of n-tetratetracontane (1000ug/mL, Section 8.5.3). Bring to volume with methylene chloride. This standard is stored in a refrigerator at 4 ± 2°C and expires 6 months from the date of preparation.

8.11 Fuel Degradation Standard

To a 20mL volumetric flask of methylene chloride, add 500 uL of a 2000 ug/mL of a vendor prepared Fuel Oil Degradation Mix. Adjust to volume with methylene chloride.

9. Procedure

9.1 SET-UP

9.1.1 Extraction

In general, water samples are extracted at a neutral pH with methylene chloride, using Method 3510. Solid samples are extracted using either Method 3540, 3545 or 3546 to achieve maximum sensitivity. The extract must be concentrated to a final volume of 1mL.

9.1.2 Typical Gas Chromatography Conditions

Set helium carrier gas flow at 30mL min^{-1}, column temperature at 45°C for 2 minutes, then 12°C min^{-1} to 320°C, hold for 10.0 minutes. Injection volume can be either 1uL or 2uL depending on the instrument utilized. The same injection volume for all calibration standards and samples extracts must be used.

9.2 Initial Calibration

9.2.1 Calibration Curve Generation:

A curve consisting of the 6 different concentrations (Table 1), each containing C8, C9 and C10 through C40 even numbered alkanes (Section 8.5), is analyzed under the conditions listed in Section 9.1.2.

The %RSD for MEDRO and TPH-DRO (C10-C28) must be < 20% for the initial calibration. The %RSD for Alpha LIMS product TPH-DROD (C10-C36) and TPH-DROD-C44 (C9-C44 must be < 25% for the initial calibration. Once this is achieved, a continuing calibration standard (Section 9.3) is analyzed. If the Maine DRO method is to be analyzed at any point after Initial Calibration, a Fuel Degradation Standard is added after the continuing calibration verification.

9.3 Continuing Calibration

9.3.1 As a minimum requirement, the working calibration curve and retention times must be verified at the beginning of each 12-hour work shift, or every 20 samples, followed by an acceptable bracketing continuing calibration verification standard. Additional analyses of the verification standard(s) throughout a 12-hour work shift are strongly recommended, especially if the samples being analyzed contain visible concentrations of oily material.

9.3.2 Calculate the % Difference of the standard from the calibration curve.

9.3.2.1 If the response for the MEDRO or TPH-DRO C10-C28 range is within ± 20% of the response obtained during the initial calibration then the initial calibration is still considered valid, and the analyst may continue to use the mean CF or RF values from the initial calibration to quantitate the sample results. Individual alkanes may exceed %D of ± 20, as long as the range is ± 20%.

9.3.2.2 If the response for the Alpha LIMS product TPH-DROD (C10-C36) and TPH-DROD-C44 (C9-C44) range is within ± 25 of the response obtained during the initial calibration then the initial calibration is still considered valid, and the analyst may continue to use the mean CF or RF values from the initial calibration to quantitate the sample results. Individual alkanes may exceed %D of ± 25, as long as the range is ± 25%

9.3.2.3 If the % difference exceeds then preventative maintenance must be performed (Section 9.5).

Once maintenance is complete, another standard is analyzed. If the % difference still exceeds the method requirements, a new initial calibration must be performed (Section 9.2).

9.3.2.4 The alkanes for the opening and ending Containing calibration cannot be over 50% and 10% must be within the criteria.

9.3.3 **Maine DRO, Fuel Degradation Check Standard**

For Maine-DRO analysis, each analytical sequence ends with a demonstration that the resolution criteria have been met for all the samples run in that sequence. (i.e. The system must resolve pristane from C17 and phytane from C18 at 50ug/mL total commercial diesel fuel oil, or at the minimum reporting level. This is typically analyzed after an ICAL or analytical sequence containing samples. Peaks are considered to be resolved when 60% resolution has been achieved.

If any of the above criteria are not met, the problem must be corrected before further samples are analyzed. Any samples analyzed between the last acceptable QC samples must be reanalyzed. If reanalysis is not possible (e.g. due to holding time exceedences or limited volume, etc.) the data must be flagged and a narrative included on the final report.

9.4 Sample Analysis and Quantitation

9.4.1 The same GC operating conditions used for the initial calibration must be employed for sample analyses, including sample injection volume (Section 9.1.2).

9.4.2 Tentative identification occurs when a peak from a sample extract falls within the retention time window for the compound.

9.4.3 The sample ranges are quantitated according to the product requested by the Client (TPH-DRO, Maine-DRO, TPH-DRO-D or TPH-DROD-C44). Refer to Table 1 for carbon ranges.

9.4.4 The concentration reported for an extract is calculated using the Environquant data processing software. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined. See the Manual Integration SOP for integration guidelines.

9.4.5 If the sample concentration exceeds the highest concentration of the respective calibration curve, the sample extract must be diluted with methylene chloride and reanalyzed.

9.5 Preventative Maintenance

Preventive maintenance is performed at the discretion of the GC analyst.

9.5.1 Cool the oven to 25°C; turn off both the front and back inlet pressure and temperature.

9.5.2 Remove the septum retaining nut and the old septum. Use forceps to insert a new septum, pressing it firmly into the fitting. Replace the septum retaining nut, and tighten finger-tight.

9.5.3 Using a septum nut wrench, loosen the split/splitless insert nut located under the septum retaining nut. Remove and replace the pre-packed glass wool liner from injection ports using forceps.

9.5.4 To replace the inlet seal base, remove the column from the inlet using a ¼-inch wrench. Remove the insulation cup using either a Phillips-head or star-head screwdriver, or a ½ –inch wrench to loosen the reducing nut. The seal is placed in the nut first, followed by the seal, making sure the raised portion is facing downwards. Replace the reducing nut and tighten.

9.5.5 **Capillary Column Replacement**

9.5.5.1 To prepare the capillary column for installation, score the column using a glass or ceramic scribing tool. Break off the column end by supporting it against the column cutter opposite the scribe. Ensure there are no jagged edges. Position the column through the column nut and 0.5 mm graphite Restek ferrule, making sure it extends 4 to 6mm to the end of the ferrule. Insert the column in the inlet and slide the nut and ferrule up the column to the inlet base. Finger-tighten the column nut until it starts to grab the column. Adjust the column position accordingly. Tighten the column nut with a ¼-inch wrench until the column is firmly in place.

9.5.5.2 Reload the method either on the instrument or at the computer terminal. Set the oven temperature to 270 °C for approximately one-half hour.

9.5.5.3 A new calibration curve must be analyzed following capillary column replacement.

9.6 Calculations

Quantitation: The total area under all peaks and any baseline rise is determined for the sample and standard.

For liquids:

$$C = \frac{[(R_S)(V_E)(D)]}{[(V_S)(m)]}$$

Where:

C = Concentration of Standard, µg/L
D = Dilution Factor
Vs = Volume of water sample, L
V_E = Total Volume of sample extract, mL
R_S = GC Response in DRO window
m = Slope of the curve

For solids:

$$C = \frac{[(R_S)(V_E)(D)]}{[(W)(m)]}$$

Where:

C = Concentration of Standard, mg/Kg
D = Dilution Factor
W = Weight of water sample, Kg
V_E = Total Volume of sample extract, mL
R_S = GC Response in DRO window
m = Slope of the curve

10. Quality Control and Data Assessment

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

10.1 Demonstration of Capability

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. Each time a method modification is made, the analyst is required to repeat the procedure.

When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must locate and correct the source of the problem and repeat the test for failed parameters of the method.

Repeated failure confirms a general problem with the measurement system or analytical technique of the analyst. If the failure repeats, locate and correct the source of the problem and repeat the test for all parameters listed in the method.

10.2 Method Blanks

Method blanks are extracted and analyzed with each analytical batch of 20 or less samples. The result of the method blank must be less than the reporting limit.

If results are detected above the reporting limit and the associated samples are non-detect, a narrative is submitted for inclusion on the final report. If results are detected above the reporting limit and the associated samples also have positive results, all samples in the extraction batch must be reextracted and reanalyzed.

10.3 Laboratory Control Samples (LCS)

A Laboratory Control Sample is extracted with each sample batch. The LCS consists of an aliquot of clean (control) matrix similar to the sample matrix, and of the same weight or volume, and a known amount of analyte spike. The LCS must be recovered between 60 – 140% for Maine-DRO and TPH-DRO products. For the TPH-DRO-D and TPH-DROD-C44 product, the LCS must be recovered between 40 – 140%.

If the LCS recovery is outside of acceptance criteria, all samples in the extraction batch must be reextracted and reanalyzed.

10.4 Duplicates

A duplicate aliquot of a sample is extracted and analyzed to ensure the consistency of the extraction. The relative percent difference (%RPD) must be $\leq$ 20% for the Maine-DRO product. For TPH-DRO and TPH-DRO-D and TPH-DROD-C44 products, the relative percent difference (%RPD) must be $\leq$ 40%.

If the % RPD is outside of acceptance criteria, the Duplicate sample is reextracted and reanalyzed, unless there is obvious sample non-homogeneity. If there is obvious sample non-homogeneity, a narrative is submitted for inclusion on the final report.

10.5 Matrix Spike (MS)

For the Maine-DRO product, a MS is extracted and analyzed with each analytical batch. The MS consists of an aliquot of sample matrix and a known amount of analyte spike. The MS must be recovered between 60 – 140%.

If the MS recovery is outside of acceptance criteria and the LCS is within acceptance criteria, a narrative is submitted for inclusion on the final report. If the MS and the LCS are outside or acceptance criteria, all samples in the extraction batch must be reextracted and reanalyzed.

10.6 Control Limits

The laboratory maintains performance records to document the quality of data that is generated. Method accuracy for samples is assessed and records maintained.

Control limits for the method parameters are generated. The control limits are calculated based on in-house performance data. The limits are compared to the control limits found in the reference method.

10.7 Surrogate Standards

Surrogate recoveries are calculated for all samples and QC samples and must recover within 40 – 140%. If any surrogate recovers outside acceptance criteria, the sample is reextracted and reanalyzed for confirmation.

If any surrogate recovers above acceptance criteria and the sample is non-detect, a narrative is submitted for inclusion on the client report.

If the surrogate in a sample duplicate recovers outside of acceptance criteria and the surrogate in the sample recovers within acceptance criteria, the data may be reported with a narrative submitted for inclusion on the client report.

10.8 Analytical Sequence

A typical 12-hour analytical sequence:

- Continuing Calibration Standard
- Method Blank
- LCS
- Samples (up to 20)
- Bracketing Continuing Calibration Standard

11. Method Performance

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations were obtained using a diesel fuel mixture that is calculated against the DRO component standard. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

Method performance data is on file in the laboratory QC department. Comparison of method performance data for the laboratory to the reference method criteria occurs when laboratory in-house acceptance limits are generated. In-house generated data must be within the specifications of the reference method or the analysis is not continued until corrective action is completed.

12. Corrective Actions

Holding time exceedence, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CCV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the un-spiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Pollution Prevention

See Chemical Hygiene Plan for pollution prevention operations.

14. Waste Management

See Chemical Hygiene Plan for waste handling and disposal.

15. Attachments

Table 1: Calibration Concentrations for TPH-DRO and TPH-DRO-D

Table 2: Calibration Concentrations for TPH-DROD-C44

Table 1: Calibration Concentrations for TPH-DRO and TPH-DRO-D

	Level 1 (ug/mL)	Level 2 (ug/mL)	Level 3 (ug/mL)	Level 4 (ug/mL)	Level 5 (ug/mL)	Level 6 (ug/mL)
Dodecane	3	20	50	100	200	500
Tetradecane	3	20	50	100	200	500
Hexadecane	3	20	50	100	200	500
Octadecane	3	20	50	100	200	500
Eicosane	3	20	50	100	200	500
Docosane	3	20	50	100	200	500
Tetracosane	3	20	50	100	200	500
Hexacosane	3	20	50	100	200	500
Octacosane	3	20	50	100	200	500
Triacontane	3	20	50	100	200	500
Dotriacontane	3	20	50	100	200	500
Tetratriacontane	3	20	50	100	200	500
Hexatriacontane	3	20	50	100	200	500
C10 - C28 Total (Maine DRO, TPH-DRO)	30	200	500	1000	2000	5000
C10 - C36 Total (TPH-DRO-D)	42	280	700	1400	2800	7000

Table 2: Calibration Concentrations for TPH-DROD-C44

	Level 1 (ug/mL)	Level 2 (ug/mL)	Level 3 (ug/mL)	Level 4 (ug/mL)	Level 5 (ug/mL)
Nonane	3	20	50	100	250
Decane	3	20	50	100	250
Dodecane	3	20	50	100	250
Tetradecane	3	20	50	100	250
Hexadecane	3	20	50	100	250
Octadecane	3	20	50	100	250
Eicosane	3	20	50	100	250
Docosane	3	20	50	100	250
Tetracosane	3	20	50	100	250
Hexacosane	3	20	50	100	250
Octacosane	3	20	50	100	250
Triacontane	3	20	50	100	250
Dotriacontane	3	20	50	100	250
Tetratriacontane	3	20	50	100	250
Hexatriacontane	3	20	50	100	250
Octatriacontane	3	20	50	100	250
Tetracontane	3	20	50	100	250
Tetratetracontane	3	20	50	100	250
C9 - C44 Total (TPH-DROD-C44)	54	360	900	1800	4500

TPH-Gasoline Range Organics 8015D

Reference: Maine 4.2.17, Maine Health and Environmental Testing Laboratory, 09/06/1995.

EPA 8015D (Modified), SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, June 2003

1. Scope and Application

Matrices: This method is suitable for the analysis of waters, soils, or wastes.

Definitions: Refer to Alpha Analytical Quality Manual.

This method is designed to measure the concentration of gasoline range organics, from MTBE through naphthalene inclusive, in both water and soil. This corresponds to a boiling point range between approximately 60 ºC and 220 ºC.

The analytical procedure is based on a purge-and-trap, Gas Chromatography (GC) procedure.

With the optional PID/FID in series, the method can be extended for the specific determination of petroleum volatile organic compounds (PVOCs) as specified in the current edition of EPA Method 8020.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the Gas Chromatograph and in the interpretation of Gas Chromatograph data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

This method provides gas chromatographic conditions for the detection of volatile petroleum fractions such as gasoline, Stoddard solvent, or mineral spirits. Samples are analyzed utilizing purge-and-trap sample concentration. The gas chromatograph is temperature programmed and utilizes a capillary column to facilitate separation of organic compounds. Detection is achieved by a flame ionization detector (FID) or FID with photoionization detector (PID) in series (photoionization detector first in the series). Quantitation is based on FID detector response to a gasoline component standard.

This method is suitable for the analysis of waters, soils, or wastes. Water samples can be analyzed directly for gasoline range organics by purge-and-trap extraction and gas chromatography. Soil or waste samples are dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is then analyzed by purge-and-trap GC.

This method is based in part on (1) API's Petroleum Hydrocarbon Methods (Revised August 1993); (2) work by the Wisconsin Ad-Hoc Committee on LUST Program Analytical Requirements and Wisconsin State Laboratory or Hygiene; and (3) work by the Maine Laboratory Hydrocarbon Study Committee

2.1 Method Modifications from Reference

Soil samples do not have extraction surrogates or spikes added to them at the time of methanol preservation. Surrogates and spikes are added directly to the 5mL syringe containing water and the appropriate volume of the methanol extract.

GRO is defined as all chromatographic peaks eluting from methyl-tertiary-butyl ether through naphthalene.

3. Reporting Limits

The Reported Limit for water is 50 µg/L and for soil is 2.5 mg/Kg.

4. Interferences

4.1 Heavier petroleum products such as diesel fuel may contain some volatile components producing a response within the retention time range for GRO. Other compounds that respond to the FID, such as chlorinated and oxygenated hydrocarbons, are detected by this method and will be included in the concentration. Spills of known specific constituents are analyzed and quantified by a method specific for those constituents. If the chromatogram exhibits the characteristics of fuel oil, the GRO is reported but flagged with a recommendation for DRO analysis.

4.2 Samples can be contaminated by diffusion of volatile organics through the sample container septum during shipment and storage, or by dissolution of volatiles into the methanol used for preservation. Trip Blanks prepared from both reagent water and methanol should be carried through sampling and subsequent storage and handling to serve as a check on such contamination.

4.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe and/or purging device must be rinsed between samples with reagent water or solvent. For volatile samples containing high concentrations of water-soluble materials, suspended solids, high boiling compounds or organohalides, it may be necessary to wash the syringe or purging device with a detergent solution, rinse with distilled water, and then dry in a 105ºC oven between analyses. The trap and other parts of the system are also subject to contamination, therefore frequent bake-out and purging of the entire system may be required. A screening step is recommended to protect analytical instrumentation. Whenever an unusually concentrated sample is encountered, it should be followed by analysis of a water blank to check for cross-contamination.

4.4 The retention time window definition (methyl tert-butyl ether through naphthalene inclusive) introduces a negative bias, however, it improves comparability between laboratory data. Note that gasoline blends often contain 10% ethanol, which could be responsible for a portion of this negative bias.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in duplicate, without agitation and without headspace in VOA vials with Teflon-lined septa screw caps. The Teflon liner must contact the sample.

A reagent water trip blank accompanies each batch of water samples.

Methanol (purge and trap grade) must be added to soil sample vials before or immediately after sample collection. In lieu of the in-field preservation of samples with methanol, soil samples may be collected in specially designed, air tight sampling devices. Samples that have not been preserved in the field are preserved in the lab as soon as possible after arrival in the lab. Date and time of preservation is noted on the Chain of Custody or final report.

A methanol trip blank accompanies each batch of soil samples.

6.2 Sample Preservation

Aqueous samples are acidified to a pH of 2.0 or less at the time of collection. This is typically accomplished by adding 3 or 4 drops (0.1 to 0.2mL) of 1:1 HCL (one part reagent water and one part concentrated hydrochloric acid) to a 40mL sample. The laboratory must determine the pH of all water samples at the time of analysis and if the pH is found to be greater than 2.0, it must be noted.

Aqueous and soil samples must be cooled to 4 ± 2 ºC immediately after collection

6.3 Sample Shipping

No specific requirement.

6.4 Sample Handling

Water and soil samples must be analyzed within 14 days of collection.

7. Equipment and Supplies

7.1 Gas Chromatograph: Agilent 6890 or equivalent, Data system Enviroquant Software in conjunction with Nelson Intelligent 900 Series Interface.

7.1.1 Column: Restek RTX-502.2 Capillary Column, 105m X .53mm I.D., 3.0 micron film thickness or equivalent. The column must be capable of achieving a 60% resolution of all 10 components, with the exception of m/p-xylene. It must also be capable of separating methyl tert-butyl ether from the methanol solvent peak. The Resolution is defined as:

$$\frac{(\text{Height of smallest peak} - \text{Height of valley}) \times 100\%}{(\text{Height of smallest peak})}$$

7.1.2 Detector: FID (in series with a PID detector, optional)

7.1.3 Purge-and-trap Device: The purge and trap device consists of two separate pieces of equipment: a purging device coupled to a desorber (Autosamplers: Centurion. Concentrators: Encon evolution).

7.1.3.1 Purge/carrier gas = Helium, analytical grade (99.999% pure) or Nitrogen analytical grade (99.999%).

7.1.3.2 Traps are conditioned, desorbed, and baked according to manufacturer's guidelines. The trap may be vented to the analytical column during daily conditioning, however, the column must be run through the temperature program prior to analysis of the samples.

7.1.3.3 The desorber must be capable of rapidly heating the trap to 260ºC.

7.2 Analytical Balance: A top-loading balance capable of weighing to the nearest 0.01 g (for weighing soil samples).

7.3 Ultrasonic Bath

7.4 VOA Vials: 40mL amber VOC vials with Teflon/silicone septa.

7.5 Syringes: 5mL gas-tight syringe with shutoff valve.

7.6 Volumetric Flask (class A): 10mL, 50mL, 100mL, 500mL, 1000mL with a ground-glass or screw-top stopper.

7.7 Micro syringes: 1µL, 5µL, 10µL, 25µL, 100µL, 250µL, 500µL, and 1000µL.

7.8 Spatula: Stainless steel.

7.9 Ottawa Sand: SiO2.

8. Reagents and Standards

8.1 Reagent Water: Organic-free water; municipal tap water filtered through carbon.

8.2 Methanol: Purge-and-trap grade or equivalent. Store away from other solvents.

8.3 Gasoline Component Stock Standard: Purchased from Ultra Scientific UST-100, GRO PVOC/WISC mix. Refer to Table 1 for concentrations. Store with minimal headspace at -10°C to -20°C. Expires 6 months from the date of opening or upon signs of degradation or evaporation.

8.3.1 GRO Calibration Stock Standard: To a 10mL volumetric flask, add 1mL of Gasoline Component Stock Standard (Section 8.3), Add 500µL of 4-bromofluorobenzene (Section 8.5.2) and 500µL of a,a,a-trifluorotoluene (Section 8.5.1). Bring to volume with methanol. Store with minimal headspace at -10°C to -20°C. Expires 6 months from the date of preparation or upon signs of degradation or evaporation.

8.3.1.1 GRO Working Calibration Standards

Store standards with minimal headspace at -10°C to -20°C. Standards expire 6 months from the date of preparation or upon signs of degradation or evaporation.

8.3.1.1.1 Level 1, 50ng ranges, 5ng surrogates: To a 100mL volumetric flask, add 1µL of GRO Calibration Stock Standard (Section 8.3.1). Bring to volume with reagent water.

8.3.1.1.2 Level 2, 100ng ranges, 10ng surrogates: To a 100mL volumetric flask, add 2µL of GRO Calibration Stock Standard (Section 8.3.1). Bring to volume with reagent water.

8.3.1.1.3 Level 3, 500ng ranges, 50ng surrogates: To a 100mL volumetric flask, add 10µL of GRO Calibration Stock Standard (Section 8.3.1). Bring to volume with reagent water.

8.3.1.1.4 Level 4, 2000 ng ranges, 200ng surrogates: To a 5mL syringe containing reagent water, add 2µL of GRO Calibration Stock Standard (Section 8.3.1).

8.3.1.1.5 Level 5, 5000ng ranges, 500ng surrogates: To a 5mL syringe containing reagent water, add 5µL of GRO Calibration Stock Standard (Section 8.3.1).

8.3.1.1.6 Level 6, 10000ng ranges, 1000ng surrogates: To a 5mL syringe containing reagent water, add 10µL of GRO Calibration Stock Standard (Section 8.3.1).

8.3.1.1.7 Level 7, 20000ng ranges, 2000ng surrogates: To a 5mL syringe containing reagent water, add 20µL of GRO Calibration Stock Standard (Section 8.3.1).

8.4 Gasoline Component Stock Standard (second source for LCS, MS):

Purchased from Accustandard GRH-003S-Pak. Store with minimal headspace at -10°C to -20°C. Standard expires 6 months from the date of preparation or upon signs of degradation or evaporation.

8.4.1 Continuing Calibration Standard, water LCS, MS: To a 10mL volumetric flask, add 200µL of second source Gasoline Component Stock Standard (Section 8.4). Add 200µL 4-BFB (Section 8.5.2) and 200µL a,a,a-trifluorotoluene (Section 8.5.1). Bring to volume with methanol.

CCV/LCS for water: Spike 5µL of this solution into 5mL of reagent water.

MS: Spike 5µL of this solution into 5mL of a second aliquot of sample.

8.4.2 Continuing Calibration Standard, soil LCS:

CCV/LCS for soil: Weigh 15g of Ottawa sand into 15ml methanol. Spike with 15ul Gasoline Component Stock Standard (8.4). Take 100ul aliquot and add to 5ml reagent water, add 5ul working surrogate standard solution (8.6).

8.5 Surrogate Control Stock Standards

Standards expire 6 months from the date of opening or upon signs of degradation or evaporation. These standards are stored with minimal headspace at -10°C to -20°C.

8.5.1 a,a,a-trifluorotoluene: 2000µg/mL, purchased from Ultra Scientific, Cat # STS-220N

8.5.2 4-bromofluorobenzene: 2000µg/mL, purchased from Ultra Scientific, Cat # STS-110N.

8.6 Working Surrogate Standard Solution:

To a 10mL volumetric flask, add 200µL 4-BFB (Section 8.5.2) and 200µL a,a,a-trichfluorotoluene (Section 8.5.1). Bring to final volume with MeOH for a final concentration of 40ug/L. Standard expires 6 months from the date of preparation or upon signs of degradation or evaporation. Store with minimal headspace at -10°C to -20°C.

Spike 5µL of this solution into 5mL of each sample and Blank.

8.7 Gasoline Stock Standard (MDL): Purchased from Ultra Scientific, Cat # RGO-600-1. Standard expires 6 months from the date of opening or upon signs of degradation or evaporation. This standard is stored with minimal headspace at -10°C to -20°C.

8.8 Ohio Gasoline Component Stock Standard: Purchased from Absolute Standards Cat #96018, n-Hydrocarbon Mix C5-C8. Refer to Table 2 for concentrations. Store with minimal headspace at -10°C to -20°C. Expires 6 months from the date of opening or upon signs of degradation or evaporation.

8.8.1 Ohio Calibration Stock Standard: To a 10mL volumetric flask, add 1mL of Ohio Gasoline Component Stock Standard (Section 8.8), Add 500µL of 4-bromofluorobenzene (Section 8.5.2) and 500µL of a,a,a-trifluorotoluene (Section 8.5.1). Bring to volume with methanol. Store with minimal headspace at -10°C to -20°C. Expires 6 months from the date of preparation or upon signs of degradation or evaporation.

8.8.1.1 Ohio GRO Working Calibration Standards

8.8.1.1.1 Level 1, 40 ng ranges, 5ng surrogates: To a 100mL volumetric flask, add 1µL of Ohio Calibration Stock Standard (8.8.1). Bring to volume with reagent water.

8.8.1.1.2 Level 2, 80 ng ranges, 10ng surrogates: To a 100mL volumetric flask, add 2µL of Ohio Calibration Stock Standard (8.8.1). Bring to volume with reagent water.

8.8.1.1.3 Level 3, 400 ng ranges, 50ng surrogates: To a 100mL volumetric flask, add 10µL of Ohio Calibration Stock Standard (8.8.1). Bring to volume with reagent water.

8.8.1.1.4 Level 4, 1600 ng ranges, 200ng surrogates: To a VOA vial, add 5 mL of water and 2µL of Ohio Calibration Stock Standard (8.8.1).

8.8.1.1.5 Level 5, 4000 ng ranges, 500ng surrogates: To a VOA vial, add 5 mL of water and 5µL of Ohio Calibration Stock Standard (8.8.1).

8.8.1.1.6 Level 6, 8000 ng ranges, 1000ng surrogates: To a VOA vial, add 5 mL of water and 10µL of Ohio Calibration Stock Standard (8.8.1).

8.8.1.1.7 Level 7, 16000 ng ranges, 2000ng surrogates: To a VOA vial, add 5 mL of water and 20µL of Ohio Calibration Stock Standard (8.8.1).

8.9 Ohio Gasoline Component Stock Standard (second source for LCS, MS): Purchased from Sigma Aldrich Cat #, UST157, Aliphatics Mix (C5-C12). Store with minimal headspace at -10°C to -20°C. Standard expires 6 months from the date of preparation or upon signs of degradation or evaporation.

8.9.1 Ohio Continuing Calibration Standard, water LCS, MS: To a 10mL volumetric flask, add 200µL of second source Ohio Gasoline Component Stock Standard (Section 8.9). Add 200µL 4-BFB (Section 8.5.2) and 200µL a,a,a-trifluorotoluene (Section 8.5.1). Bring to volume with methanol.

CCV/LCS for water: Spike 5µL of this solution into 5mL of reagent water.

MS: Spike 5µL of this solution into 5mL of a second aliquot of sample.

8.9.2 Ohio Continuing Calibration Standard, soil LCS:

CCV/LCS for soil: Weigh 15g of Ottawa sand into 15ml methanol. Spike with 15ul Ohio Gasoline Component Stock Standard (8.9). Take 100ul aliquot and add to 5ml reagent water, add 5ul working surrogate standard solution (8.6).

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blanks

9.1.1 A water blank must be run with every analytical sequence containing water samples, utilizing the same water used to prepare standards and make dilutions. The concentration of the blank should not exceed the laboratory reporting level.

Water blanks are analyzed after samples suspected of being highly concentrated, to prevent carryover.

9.1.2 A reagent methanol blank must be run with each analytical sequence containing soil samples. The methanol blank is created by adding 15g of Ottawa sand to 15ml reagent methanol. Take 100ul aliquot and add to 5ml reagent water, add 5ul working surrogate standard solution (8.6) The amount of material in the blank should not exceed the laboratory reporting level.

9.2 Laboratory Control Sample (LCS)

The calibration curve must be verified with each analytical sequence by running a mid-level calibration standard in the beginning and end of the sequence.
See section 8.4.2 for LCS preparation.

NOTE: For soil batches the LCS is also injected with 100 µL methanol.

The response must fall within 20% of the expected response. If the response for the standard varies from the predicted response by more than 20%, the analytical system is examined to determine the cause and corrective action is performed and/or a new continuing calibration standard is prepared and analyzed. If the 20% criteria is still not met, the system must be recalibrated.

Any samples that were analyzed following the opening standard and before a closing standard that did not meet calibration criteria must be reanalyzed. If reanalysis is not possible, the data must be flagged and a narrative submitted for inclusion on the Client report

LCS Duplicates are run at a frequency of 5%. Alternatively, duplicate samples and spike samples can be substituted for the laboratory spiked duplicates at a frequency of 5%. Care must be taken to ensure that the samples are homogenous before analyzing duplicates and spikes.

9.3 Initial Calibration Verification (ICV)

9.4 Continuing Calibration Verification (CCV)

9.5 Matrix Spike

For every 20 samples analyzed, the lab will analyze a Gasoline Component Spike. The spike must be run through the method in the same manner as samples. The accuracy must be within

± 20% of the known concentration. If the accuracy is outside of acceptance criteria, and the LCS is acceptable, the data is reported and the MS failure is narrated.

A Matrix Spike Duplicate is performed only upon Client request.

If requested, for every 20 samples analyzed, the lab will analyze a set of Duplicate Gasoline Component Spikes. The duplicate spikes must be run through the method in the same manner as samples. The accuracy of the 2 spikes must be within 20% of the known concentration and the %RPD must be 20% or less. If these criteria are not met, and the LCS is acceptable, the data is reported and the MS failure is narrated

9.6 Laboratory Duplicate

A duplicate sample is analyzed once for every 20 samples. The duplicate aliquot is taken from the same sample container and analyzed separately under the same conditions. The %RPD between the sample and duplicate results is calculated and must be ≤ 20%. If this criterion cannot be met, the duplicate is reanalyzed. If failure continues, the data is reported and the nonconformance is narrated.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

If surrogate recovery is outside of the established limits of 70 – 130%, verify calculations, dilutions, and standard solutions. Verify instrument performance, including checking for leaks and purge problems if the recovery is low. Low recovery may be due to the sample matrix. The analysis is repeated. If the recovery is less than 70% or, if the analyses cannot be repeated, the data is flagged and a narrative submitted for inclusion on the Client report.

High recoveries may be due to coeluting matrix interference. Surrogate recoveries may be reported as “masked” in high-level samples exhibiting matrix interference.

9.1 Method Sequence

The analytical sequence is:

- Calibration Standards (initial) or mid-range Calibration Check Standard (LCS)
- LCS Duplicate (as needed)
- Laboratory Method Blank
- Samples
- Duplicate sample (as needed)
- Matrix Spike (as needed)
- Matrix Spike Duplicate (as requested)
- Closing mid-range Calibration Check Standard (LCS) – Note: This may be used as an opening CCAL (LCS) for the next sequence.

10. Procedure

10.1 Equipment Set-up

10.1.1 Gas Chromatography Conditions: Agilent 6890 or equivalent.

PID Temperature	250 ºC
FID Temperature	250 ºC

Column	12.5 mL/min
Carrier /Makeup Gas	Helium, 99.999 grade @ 12.5 mL/min
	Air @ 350 mL/min
	Hydrogen @ 30 mL/min
Injection Port	250 ºC

10.1.2 Oven Conditions: 45 ºC for 2 minutes to 100 ºC at 3 ºC/min, to 160 ºC at 8 ºC/min, to 230 ºC at 20 ºC/min for 11.6 minutes.

10.1.3 Typical Purge and Trap Conditions:

Purge Gas:	Helium
Purge Gas Flow Rate:	40 mL/min
Purge Temperature:	35 ºC or less
Purge Time:	11.0 ± 0.1minutes
Dry purge:	4 minutes
Desorb Preheat:	220 ºC
Desorb temp:	225 ºC
Desorb Time:	6 minutes
Bake temp:	260 ºC
Bake time:	15 minutes

10.2 Initial Calibration

10.2.1 Per Section 10.3 and under the conditions listed in Section 10.1, analyze the Gasoline Component Standard at the seven concentration levels (1 µg/L, 2µg/L, 10µg/L, 40µg/L, 100µg/L, 200µg/L and 400µg/L) listed in Section 8.3.1.1. Whenever a calibration curve is analyzed, an independent check standard (Section 8.4.1) is also analyzed to validate the curve (the LCS serves as a second source verification).

10.2.2 Relative Response Factors

The external standard calibration technique is used.

A collective calibration factor must be established for the GRO hydrocarbon range. The GRO marker compounds that determine the GRO range are MTBE through naphthalene, inclusive.

10.2.3 Initial Calibration Criteria

If the RSD for an individual analyte is ≤ 20%, then the response for this compound is considered linear over the range and the mean calibration factor is used to quantitate sample results. If the RSD criterion is not met for an analyte, linearity through the origin cannot be assumed. The calibration is repeated for any compounds that fail, or a calibration curve is generated for this compound and used for sample quantitation.

10.2.3.1 Percent Relative Error (%RE): For linear fit compounds, the %RE must be calculated for the standard at or near the midpoint and at the lowest level of the initial calibration. The %RE between the calculated and expected concentration of an analyte must be ≤ 30%, otherwise recalibration is necessary.

10.2.4 Retention Time Windows and Quantitation

10.2.4.1 All chromatographic peaks eluting from methyl tert-butyl ether through naphthalene, inclusive, are used to calculate the retention time window. Quantitation is based on a direct comparison of the area within this range to the

total area of the 10 components in the Gasoline Component Standard. (Using a "baseline to baseline" integration as opposed to a "valley to valley" integration.)

10.2.4.2 The retention time window is defined as beginning approximately 0.1 minutes before the retention time of methyl tert-butyl ether and ending 0.1 minutes after the retention time of naphthalene in the calibration run.

10.2.4.3 The laboratory must determine retention time windows for the first and last standard on each GC column and whenever a new GC column is installed. This data must be retained by the laboratory.

10.2.4.4 Quantify by summing all peak areas eluting from methyl tert-butyl ether through naphthalene, inclusive.

10.3 Equipment Operation and Sample Processing

10.3.1 Gas Chromatographic Analysis of Water Samples and Soil Samples

10.3.1.1 Retrieve sample VOA vials from the sample bank refrigerator just prior to loading onto the Purge and Trap. Soilsamples preserved with methanol and QC samples must be vortexed to extract the volatile components into the methanol. Let sample settle prior to taking methanol aliquot.

10.3.1.2 Rinse a 5mL Luerlock syringe with reagent water. For aqueous samples, add appropriate amount of sample (typically 5mL) to the syringe after pouring off approximately 2 – 3 mL of sample to waste. For soil samples, fill the syringe with 5mL of reagent water. Add the appropriate amount of the methanol extract (typically 100µL) to the syringe containing the reagent water. Less sample (water or methanol) may be used if sample history or screening information indicates high concentrations of target or non-target analytes. Add 5 µL of surrogate standard solution (Section 8.5) to the sample. Transfer the sample to a VOA vial and load on the autosampler.

10.3.1.3 If sample dilutions are necessary, they may be made in volumetric flasks (10mL to 100mL). Dilutions are made without delay. If aliquots of sample less than 1mL are required, use microliter syringes to transfer the sample aliquot to the flask. Dilute to the mark with reagent water.

Alternatively, the dilutions may be made directly in a gas tight syringe to avoid further loss of volatiles.

All dilutions must ensure that the response of the major constituents is within the linear range of the curve.

10.3.1.4 If the initial analysis of a sample or a dilution of the sample has a concentration of analytes that exceeds the initial calibration range, the sample must be reanalyzed at a higher dilution. When a sample is analyzed that has a saturated response from a compound, this analysis must be followed by a blank reagent water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of interferences at less than the reporting limit.

10.4 Continuing Calibration

The working calibration curve must be verified on each 12-hour period, by the injection of a midpoint Gasoline Component Standard (or an LCS) in the beginning of the analytical sequence. If the response for the Gasoline Component Standard varies from the predicted response by more than 20%, the analytical system is examined to determine the cause and corrective action is performed and/or a new continuing calibration standard is prepared and

analyzed. If the 20% criteria are still not met, the system must be recalibrated. Any samples that were analyzed following the opening standard that did not meet calibration criteria must be reanalyzed. If reanalysis is not possible, the data must be flagged and a narrative submitted for inclusion on the Client report.

10.5 Preventive Maintenance

If the response of the PID detector decreases significantly, perform a cleaning of the PID lamp per the manufacturer's instructions. If the response is still low, the PID lamp is replaced with a new one (PID lamp life is typically 30 – 90 days).

Additional maintenance is performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and clipping several inches off the injector end of the capillary column. The trap used in the Purge and Trap equipment will also need periodic replacement, when system performance degrades.

All maintenance and repair is recorded in the appropriate Instrument Maintenance Log.

11. Data Evaluation, Calculations and Reporting

11.1 Calculations

The concentration of Gasoline Range Organics in the sample is determined from a summation of the total peak area of all chromatographic peaks eluting from methyl tert-butyl ether through naphthalene, inclusive, and comparing to the calibration curve. (Using a "baseline to baseline" integration as opposed to a "valley to valley" integration.) Refer to Section 10.2.4**.**

Quantification is based on the average response from the calibration curve. The %RSD of the RF's must not exceed 20%.

Linear regression: From the calibration standards GC responses, R, and their known concentrations, C, the following linear equation is derived [R is plotted on the y-axis; C is plotted on the x-axis]:

$$R = mC + b; \text{ which can be rearranged to } C = (R - b) / (m)$$

Using the slope (m) and the intercept (b) from this equation, the concentration of sample can be calculated from the following equations:

Water Samples:

$$Cs = (Rs - b)(D) / (m)$$

Soil Samples:

$$Cs = [(Rs-b)(D)(Vt)(K)] / [(Vp)(W)(m)]$$

Where:

Cs = Concentration of sample in µg/L for waters and mg/Kg on a dry weight basis for soils. (Note: Wet weight basis can be used upon Client request.)

m = slope of the calibration curve

Rs = GC response of sample in the GRO retention time window

b = intercept of calibration curve

D = dilution factor if water sample or soil extract was diluted

Vp = volume of soil extract purged (units must be the same as those used for Vt)

Vt = total volume

K = 5 x 10^{-6} L mg/µg (this constant adjusts for both conversion from µg/Kg to mg/Kg and for the dilution of the volume of extract purged up to the 5mL used for purging).

W = total dry weight of soil sample in Kg. (This weight can be the wet weight if requested by the Client.)

11.2 Blank areas cannot be subtracted from the sample areas. Chromatographic baseline rises due to temperature programming are corrected for by using baseline correction. The baseline correction is performed by the most convenient method that the data handling system allows.

11.3 If the water blank concentration (this does not include trip blanks) exceeds 50µg/L, (or the minimum reporting level if lower) all samples associated with this blank (samples run since the last blank that was below 50 µg/L) with reportable hits must be rerun. If the associated samples are non-detect to the reporting limit, the data is reported and the nonconformance is narrated.

11.4 If a methanol blank concentration (this does not include trip blanks) exceeds 2.5mg/L, all soil samples associated with this blank (samples that have concentrations greater than 2.5mg/Kg since the last blank that was below 2.5mg/L) must be rerun. If the associated samples are non-detect to the reporting limit, the data is reported and the nonconformance is narrated.

NOTE: The blank level is equivalent to 50µg/L in the water sample prepared by spiking 100µL of the methanol blank into 5mL of water. This is also equivalent to 2.5mg/Kg in soil

11.5 To ensure that peaks outside the GRO window are not missed, run the chromatogram out 5 minutes past the last component in the GRO component standard. All significant peaks (and baseline rises) outside the window should be qualitatively assessed and reported.

11.6 Soil concentration is reported on a dry-weight basis unless specified by the Client. The report indicates whether results are on a wet or dry weight basis.

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedance, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CV or LCS recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate

nonconformance action form. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

16. Attachments

Table 1: Gasoline Component Standards and Concentrations
Table 2: Ohio Gasoline Component Standards and Concentrations

Table 1

Gasoline Component Standards and Concentrations

COMPONENT	CONCENTRATION, µg/mL
Methyl tert-butyl ether	1000
Benzene	1000
Toluene	1000
Ethylbenzene	1000
m-Xylene	1000
p-Xylene	1000
o-Xylene	1000
1,2,4-Trimethylbenzene	1000
1,3,5-Trimethylbenzene	1000
Naphthalene	1000
	TOTAL 10,000

Table 2

Ohio Gasoline Component Standards and Concentrations

COMPONENT	CONCENTRATION, µg/mL
n-Pentane	1000
n-Hexane	1000
n-Heptane	1000
n-Octane	1000
n-Nonane	1000
n-Decane	1000
n-Undecane	1000
n-Dodecane	1000
	TOTAL 8,000

Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)

References: **Method 8260D**, SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update VI (Phase II), June 2018.

Method 5035A, SW-846, Closed System Purge & Trap and Extraction for Volatile Organics in Soil and Waste Samples. Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, Draft Revision I, July 2002.

Method 5030B, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update III, December 1996.

Method 5030C, Purge & Trap for Aqueous Samples. SW-846, Test Methods for Evaluating Solid Waste: Physical/Chemical Methods, EPA SW-846, Update IV, May 2003.

1. Scope and Application

Matrices: Method 8260 is used to determine volatile organic compounds in a variety of solid waste matrices. This method is applicable to nearly all types of samples, regardless of water content, including various air sampling trapping media, ground and surface water, aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, mousses, tars, fibrous wastes, polymeric emulsions, filter cakes, spent carbons, spent catalysts, soils, and sediments.

Definitions: Refer to Alpha Analytical Quality Manual.

The compounds listed in Table 5 may be determined by this method.

There are various techniques by which these components may be introduced into the GC/MS system. Purge-and-trap, by Methods 5030C (aqueous samples) and 5035A (solid and waste oil samples), is the most commonly used technique for volatile organic analytes. However, other techniques are also appropriate and necessary for some analytes. One technique is direct injection of an aqueous sample (concentration permitting).

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Department Manager, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the gas chromatograph/mass spectrometers and in the interpretation of mass spectra and their use as a quantitative tool. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

2. Summary of Method

The volatile compounds are introduced into the gas chromatograph by the purge-and-trap method or by direct injection. The analytes are introduced to a narrow-bore capillary column for analysis. The Gas Chromatograph (GC) is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) interfaced to the GC.

Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact (or electron impact-like) spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard, comparing sample response to the calibration standards.

2.1 Method Modifications from Reference

None.

3. Reporting Limits

Table 1 lists our typical reporting limits.

4. Interferences

4.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be free from contamination under the conditions of the analysis. Running laboratory reagent blanks as described in Section 9.1 and 10.3 demonstrates the system is free of contamination. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system must be avoided.

4.2 Sample contamination occurs by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A trip blank or a field reagent blank prepared from reagent water and carried through the sampling and handling protocol serves as a check on such contamination.

4.2.1 Storage blanks shall be analyzed if contamination is suspect. If contamination is confirmed by positive detections in the sample storage blanks, all data from samples contained in the relative refrigerator or freezer shall be evaluated for possible contamination. If the samples contain suspected contamination, the Client Services department shall be notified in order to contact the necessary clients regarding the contamination. Samples shall be reanalyzed if so desired by the client. If suspected contamination is not confirmed by storage blanks, no further action shall be pursued concerning said blanks. It is recommended that further action be taken to determine the possible cause of suspected contamination.

4.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. Whenever a highly concentrated sample is being encountered, it should be followed by an analysis of reagent water (instrument blank) to check for potential contamination. If carry-over is suspected, then numerous instrument blanks may be required; additionally, all affected samples are rerun for confirmation. In case of severe contamination, preventive maintenance of the entire system may be required.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of safety data sheets (SDS) is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

The following method analytes have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride, 1,4-dichlorobenzene, 1,2-dichloroethane, hexachlorobutadiene, 1,1,2,2-tetrachloroethane, 1,1,2-trichloroethane, chloroform, 1,2-dibromoethane, tetrachloroethene, trichloroethene, and vinyl chloride. Pure standard materials and stock standard solutions of these compounds should be handled in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, standards, or solvents.

5.2 All stock solution standard preparation must be performed in the volatiles hood. Initial calibration, continuing calibration, laboratory control sample and client sample dilutions do not need to be performed in the hood.

5.3 All expired standards must be placed into the waste bucket in the lab for future disposal. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing Methanol must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Storage, Shipping and Handling

6.1 Sample Collection and Preservation

6.1.1 Aqueous Samples

Grab samples are collected in standard 40mL glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two or more VOA vials should be filled per sample location. EPA Method 8260 requires that samples be acidified to eliminate the possibility of biological degradation. Unless otherwise directed for project-specific reasons, all VOA vials are delivered to the client with approximately 2 – 4 drops of 1:1 HCl added to the vial, which is sufficient to adjust the pH of the sample to < 2. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Fill the sample vial to the point of overflowing so that no headspace is contained within. Samples must be introduced into the vials gently to reduce agitation, which might drive off volatile compounds or cause loss of the HCl preservative.

Seal the bottle so that no air bubbles are in the VOA vial. If preservative has been added, shake vigorously for one minute. Invert the bottle and tap to check for air bubbles. Recollect the samples if any air bubbles are present.

Maintain the hermetic seal on the VOA vial until time of analysis.

6.1.2 Soil Samples

The recommended sampling method for soil samples is EPA 5035A. Method 5035A provides for two distinct sampling procedures, depending on the required reporting limits and suspected or known concentration levels of target analytes. These methods are referred to as the High Level and Low Level methods. Both are listed below but depending on the samples only one of the methods may be required. If concentration levels are unknown, it is recommended that samples be collected using both procedures.

The Lab will analyze the high level sample first, followed by the low level sample if the results from the high level analysis show that the sample is clean or contains analytes at low levels. The typical reporting levels of the two methods are listed in Table 1.

6.1.2.1 High Level Soil Samples

Collect sample in a standard 40mL glass screw-cap vial with Teflon lined silicon septa (VOA vial). The vial is provided containing 15mL of Purge and Trap Grade methanol and is labeled and weighed prior to addition of sample. Record the weight of the vial with methanol on the vial label. Prepared trip blanks are provided to the client to accompany field samples for QC purposes.

Approximately 15g of soil is added to the vial in the field, making sure that the sample is completely covered by the methanol.

Maintain the hermetic seal on the VOA vial until the time of analysis.

An additional sample of the soil must also be obtained (without methanol) to be used for the determination of soil moisture content to allow for the calculation of the dry weight results, and to calculate the methanol dilution effect. (See Sections 11.2.2.2 and 11.2.2.2.1)

6.1.2.2 Low Level Soil Samples

Collect sample in a standard 40mL glass screw-cap vials with Teflon lined silicon septa (VOA vial). Two samples should be taken per sample location. Vials are provided containing a magnetic stirring bar and 5 mL of either 200g/L sodium bisulfate solution or water, prepared by a certified vendor. These vials are labeled and weighed prior to addition of sample. Record the weight of the vial with the stirring bar and preservative on the vial label.

Approximately 5g of soil is added to the vial in the field, making sure that the sample is completely covered by the sodium bisulfate solution or water.

Maintain the hermetic seal on the VOA until the time of analysis.

6.2 Sample Handling and Storage

Document client specific sample handling, preservation and collection criteria in the project file. The laboratory Log-in staff documents sample temperature at the time of receipt.

Record deviations from this SOP or client specific criteria on the chain of custody form.

Record holding time exceedance, improper preservation and observed sample headspace on the nonconformance report form.

6.2.1 Aqueous Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 1 and 4 °C. Sample receiving personnel must note on the sample delivery group form when samples received at the laboratory are not within the temperature criteria. If more than one vial is received for a sample, the vials are stored in separate refrigerators. Storing the vials apart provides a useful check if laboratory contamination of a sample is suspected. Samples must be analyzed within 14 days of collection. Unpreserved samples must be analyzed within 7 days of collection.

6.2.2 High Level Soil Samples

Ice or refrigerate all samples from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Sample receiving personnel note on the

nonconformance report form when samples received at the laboratory are not within the temperature criteria. Samples must be analyzed within 14 days of collection.

6.2.3 Low Level Soil Samples

Ice or refrigerate samples preserved with water or sodium bisulfate from the time of collection until analysis, maintaining the sample temperature between 2 and 6 °C. Samples preserved with water are to be immediately frozen after sampling. Sample receiving personnel note on the nonconformance report form when samples received at the laboratory are not within the temperature criteria. Samples must be analyzed within 14 days of collection.

6.3 Sample Shipping

Samples requiring shipment to the laboratory are shipped in ice-packed coolers via an overnight delivery service in accordance with applicable Department of Transportation regulations.

7. Equipment and Supplies

7.1 Purge and Trap System (For Aqueous samples): The purge-and-trap system consists of two separate pieces of equipment: a purging device (autosampler) (TekmarAtomxXYZ, EST Centurion or equivalents) coupled to the desorber (concentrator) (EST Encon or Encon EV, or equivalents).

7.1.1 Purge gas = Helium analytical grade (99.999%) or Nitrogen analytical grade (99.999%).

7.1.2 The purging device is configured with 25 mL sample purge tubes, and the purge gas is introduced at the bottom of the water column as finely divided bubbles.

7.1.3 The trap used in the desorber is typically a Supelco “K” trap. Different traps may be used if equivalent performance is demonstrated.

7.1.4 The desorber is capable of rapidly heating the trap to 260^{o}C. The trap is not heated above manufacturer’s specifications.

7.2. Purge and Trap System (For Low Level and High Level Soil Samples): The purge and trap system consists of two separate pieces of equipment: a purging device (autosampler) (Tekmar AtomxXYZ, EST Centurion or equivalents) coupled to the desorber (concentrator) (Tekmar Velocity or Atomx, or EST Encon or Encon EV, or equivalents).

7.2.1 Purge gas = Helium analytical grade (99.999%) Nitrogen analytical grade (99.999%).

7.2.2 The autosampler purging device is a closed system, designed to accept the 40mL VOA vials. The VOA vial, containing the soil sample, water (or sodium bisulfate), and stirring bar is placed into the autosampler tray. The instrument automatically adds reagent water, internal standards, and surrogates to the unopened VOA vial. The vial is heated to 40 °C, and the purge gas is introduced into the aqueous portion to purge the volatile components onto the trap.

7.2.3 The trap used in the desorber is typically a Supelco “K” trap. Different traps may be used if equivalent performance is demonstrated.The desorber is capable of rapidly heating the trap to 260 °C. The trap is not heated above manufacturer specifications.

7.3 Gas Chromatography/Mass Spectrometer/Data System:

7.3.1 Gas Chromatograph, Agilent 6890/7890 or equivalent: An analytical system complete with a temperature-programmable gas chromatograph with appropriate

interface for sample introduction device. The system includes all required accessories, including syringes, analytical columns, and gases. The capillary column is directly coupled to the source of the GC/MS system.

7.3.2 Typical Gas Chromatographic Columns:

7.3.2.1 Column 1: Restek 502.2, 40 meter, 0.18mm ID, 1.0um, or equivalent.

7.3.2.2 Column 2: Restek RTX-VMS, 30 meter, 0.25mm ID, 1.4um, or equivalent.

7.3.2.3 Column 3: Restek RTX-VMS, 20 meter, 0.18mm ID, 1.5um, or equivalent.

7.3.3 Mass Spectrometer, Agilent 5973/5975/5978 or equivalent: Capable of acquiring mass spectra from mass/charge (m/z) 35 to 270 at a rate fast enough to acquire at least five (but preferably 10 or more) mass spectra across each chromatographic peak of interest, using 70 volts (nominal) electron energy in the electron impact ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 4-Bromofluorobenzene (BFB) which meets all of the criteria in Table 3, when 50ng of the GC/MS tuning standard (BFB) are injected through the GC. For all SIM analysis, the mass spectrometer must also be able to acquire data in a dual acquisition mode (SIM and full scan).

7.3.4 Data System: Hewlett-Packard EnviroQuant software is used for data acquisition and allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program.

EnviroQuant E.02.02 (or equivalent) is used for data processing and allows searching of any GC/MS data file for ions of a specified m/z, and plotting such ion abundances versus time or scan-number.

The most recent version of the EPA/NIST Mass Spectral Library is loaded onto the Target / EnviroQuant data system.

7.4 Wiretrol or Micro syringes: 10µL - 1,000µL.

7.5 Syringes: 5mL, 10mL, or 25mL, glass with Luerlock tip.

7.6 Balances: Top-loading, capable of weighing 0.01g.

7.7 Vials: 2mL, 4mL.

7.8 Disposable Pipets.

7.9 Volumetric Flasks: Class A, appropriate sizes, with ground-glass stoppers.

7.10 Eppendorf pipets.

7.11 Ottawa Sand: SiO_2

8. Reagents and Standards

Reagent grade organic chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all organic reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

Great care must be taken to maintain the integrity of all standard solutions. Standards in methanol are stored at –10ºC or less, in amber vials with PTFE-lined screw-caps.

8.1 Organic-free Reagent Water:

All references to water in this method refer to organic-free reagent water, which is tap water passed through activated carbon and air bubbled through.

8.2 Methanol:

Purge and Trap Grade or equivalent. Store in flammables cabinet.

8.3 Stock Solutions:

All stock standards are purchased from commercial vendors as certified ampule solutions. When an ampule of stock solution is opened, it is transferred to a labeled amber screw-cap vial with minimal headspace. The expiration date of the stock solution is either the vendor specified expiration date or 6 months from the date the ampule was opened, whichever is sooner. Typical stock standard concentrations are listed in Table 4.

8.4 Intermediate Standards: Intermediate standards are prepared volumetrically by diluting the appropriate stock standard(s) with methanol. Initial Calibration solutions expire 2 months from the date of preparation, or sooner if daily continuing calibration checks do not achieve the method acceptance criteria. If the Intermediate Standards are used as a second source to verify a valid Initial Calibration solution, there is no expiration date.

8.4.1 Internal Standard Solutions:

The internal standards are Fluorobenzene, Chlorobenzene-d_5, and 1,4-Dichlorobenzene-d_4. The intermediate IS solution is prepared by diluting the stock solution(s) with methanol to a concentration of 100 µg/mL. The appropriate amount of IS solution is added to the water or soil sample or QC sample to achieve a final concentration of 100 ng/sample or standard. Internal standard is added at the same concentration to all standards, samples, and QC samples.

8.4.2 Surrogate Standard Solutions:

The surrogate standards are Dibromofluoromethane, 1,2-Dichloroethane-d_4, Toluene-d_8, and 4-Bromofluorobenzene. The intermediate surrogate solution is prepared by diluting the stock solution(s) with methanol to a concentration of 100 µg/mL. The appropriate amount of surrogate solution is added to the water or soil sample or QC sample to achieve a final concentration of 100 ng/sample.

8.4.3 Target Compound Solutions:

The target analytes routinely reported by this method are listed in Table 1 and 4. The intermediate target compound solutions are prepared by diluting the stock solution(s) with methanol. This set of solutions, at concentrations of 200 µg/mL, is used for preparation of the calibration standards.

8.4.4 4-Bromofluorobenzene (BFB) Tune solution:

8.4.4.1 A solution containing BFB at a concentration of 50 µg/mL is prepared by volumetrically diluting the BFB stock solution. 1 µL of this solution is direct-injected or purged into the GC/MS system to verify system performance prior to any standard or sample analysis.

8.4.4.2 BFB may be analyzed in full scan mode while standards, samples, and QC are analyzed in SIM

8.5 Calibration Standards:

There are two types of calibration standards used for this method – initial calibration standards and calibration verification standards.

8.5.1 Initial Calibration Standards:

Initial calibration standards can be prepared at the levels listed in Table 4 (other/different levels are allowed). The Initial Calibration needs to have a minimum of 5 standards, 6 if a quadratic curve fit is used. Prepare these solutions in organic-free reagent water. The standards correspond to the range of concentrations found in typical samples and do not exceed the working range of the GC/MS system. Initial calibration should be mixed from fresh stock standards and dilution standards when generating an initial calibration curve.

8.5.2 Initial Calibration Verification Standard (ICV):

The initial calibration verification standard is at the same concentration as the level 3 for liquids and level 4 for soils initial calibration standard. This standard is prepared from source materials from a second manufacturer or from a manufacturer's batch prepared independently from the batch used for calibration. A second lot# from the same manufacturer may be adequate to meet this requirement. The standard must contain all calibrated target analytes that will be reported for the project, if readily available.

8.5.3 Continuing Calibration Verification Standard:

The continuing calibration verification standard, or calibration check standard, should be analyzed near the action level of the project. Since most projects are focused on achieving low reporting limits, the continuing calibration verification standard is at the same concentrations as the level 3 for liquids and level 4 for soils initial calibration standard. This standard is run at the beginning of each analytical sequence, following the BFB tune standard, to verify system performance.

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

9.1.1 Before processing any samples, the analyst must demonstrate through the analysis of a method blank (MB) or instrument blank that equipment and reagents are free from contaminants and interferences. The blank must contain the internal standards and surrogates and must be from the same source of water used for preparing the standards, QC samples and sample dilutions.

9.1.2 Analyze a matrix-specific blank, i.e. methanol samples need to have methanol in the blank; sodium bisulfate samples need to have a sodium bisulfate blank analyzed;

TCLP samples need a TCLP blank, after each ICAL and CCV, and prior to any sample analysis to demonstrate that interferences from the analytical system are under control.

9.1.3 Low level soil blanks must include 5g of Ottawa sand and be vortexed.

9.1.4 Blanks are considered to be acceptable if target analyte concentrations are less than one half the LOQ or are less than project-specific requirements. Blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected (i.e., target analytes are not present in samples or sample concentrations/responses are >10X the blank).

9.1.5 If an analyte of interest is found in a sample in the batch near a concentration detected in the blank, the presence and/or concentration of that analyte should be considered suspect and may require qualification. Reanalysis is not necessary if the analyte concentration falls well below the action or regulatory limit or if the analyte is deemed not important for the project.

9.1.6 The laboratory must not subtract the results of the MB (or any blank) from those of any associated samples.

9.2 Laboratory Control Sample (LCS)/ Laboratory Control Sample Duplicate (LCSD)

An LCS/LCSD pair is analyzed at the beginning of each analytical sequence. Since the LCS contains the same compounds at the same concentrations as the continuing calibration check standard, the same analysis is used to satisfy both QC elements. The LCS/LCSD acceptance criteria are based on in-house control limits, unless specified by project/regulation. Less than 10% of total compounds may be outside of control limits if recoveries are >10%. (Note: this does not apply to difficult analytes listed in Table 7 which may be accepted at recoveries <10.) If >10% of analytes are recovered above control limits, this is deemed acceptable as long as the analytes in question are not detected in associated samples. If these criteria are not met, the entire batch is re-extracted. If re-extraction is not possible, due to insufficient sample or holding time exceedance, the analyst must write up the failure on a narrative sheet for inclusion in the client report.

9.3 Initial Calibration Verification (ICV)

Refer to 12

9.4 Continuing Calibration Verification (CCV)

Refer to Section 11.2.

9.5 Matrix Spike/ Matrix Spike Duplicate

Upon Client Request or state mandate, a matrix spike/matrix spike duplicate pair may be analyzed with each batch of 20 or less samples. The MS/MSD are sample aliquots spiked with the target compounds at the same concentration as the continuing calibration standard for water analysis and level 6 for soils. The MS/MSD acceptance criteria are based on in-house control limits. If the MS/MSD does not meet the criteria, but the LCSD does, the failure may be attributed to sample matrix. Report the MS/MSD, including a narrative sheet for inclusion with the client report.

9.6 Laboratory Duplicate

Not applicable.

9.7 Method-specific Quality Control Samples

9.7.1 Internal Standards

Area counts of the internal standard peaks in all samples and QC samples must be between 50-200% of the areas of the internal standards in the QC check standard.

If any individual percent recovery falls outside the range, that parameter has failed the acceptance criteria. For calibration standards, CCVs, LCS/LCSD or blanks the internal standard must be within the range for data to be reported to the clients. For samples, matrix spikes and duplicates: if the data is not within the range, the sample is rerun to confirm that the failure is due to sample matrix. A nonconformance report form is completed to ensure client notification and reporting if matrix effect is confirmed.

9.7.2 Surrogates

Surrogates are added to each field sample and QC sample. The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The surrogate acceptance criteria are listed in Table 2. Since the SIM analysis is acquired in dual mode, the surrogates from the full scan are used to evaluate the entire sample (SIM and full scan).

9.8 Method Sequence

In a 12-hour period, the typical analytical sequence is as follows:

- BFB
- QC Check Standard/Laboratory Control Sample/LCSD
- Method Blank
- Samples
- MS/MSD (upon Client request, may be run any time after the Method Blank)

10. Procedure

10.1 Equipment Set-up

Typical instrument operating conditions are listed below. Alternate conditions are allowed if method performance criteria can be met.

10.1.1 GC Conditions:

Temperature 1:	35°C	Carrier gas:	Helium 99.999% or Nitrogen 99.999%
Hold Time 1:	4 minutes	Carrier mode:	Constant flow
Ramp 1:	6°C/minute	Carrier flow:	1 mL/minute
Temperature 2:	150°C		
Hold Time 2:	0 minutes		
Ramp 2:	8°C/minute		
Temperature 3:	220°C		
Final Time:	1 minute		

10.1.2 MS Conditions:

Mass range:	m/z of 35 – 270 amu
Scan time:	0.5 minutes/scan
Source temperature:	230°C or according to manufacturer's specifications.

10.1.3 Velocity Concentrator Purge and Trap Conditions:

Purge time:	11 minutes
Dry purge:	2 minutes
Desorb preheat:	250°C
Desorb temp:	255°C
Desorb time:	2 minutes
Bake temp:	290°C
Bake time:	10 minutes

10.1.4 Encon Concentrator Purge and Trap Conditions:

Purge time:	11 minutes
Dry purge:	1 minute
Desorb preheat:	245°C
Desorb temp:	255°C
Desorb time:	1 minute
Bake temp:	270°C
Bake time:	10 minutes

10.2 Initial Calibration

10.2.1 The initial calibration is performed at a minimum of five (5) concentration levels listed in Table 4, the low level either at or below the reporting limit. The calibration is performed using instrument conditions listed in 10.1.

BFB must be analyzed prior to analysis of the initial calibration standards and must pass the criteria listed in Table 3. The mass spectrum of BFB should be acquired in the following manner:

(1) Three scans (the peak apex scan, the scan immediately preceding the apex and the scan immediately following the apex) are acquired and averaged.
(2) Background subtraction is performed using a single scan of no more than 20 scans prior to the elution of BFB.

This is done automatically with the EnviroQuant software.

10.2.1.1 Low Level/High Level Soil Curve on Centurion: To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 50mL volumetric flask using a micro syringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, then gently fill a 5mL syringe with standard and transfer to a 40mL VOA vial containing a magnetic stir bar. Load the vial onto autosampler.

10.2.1.2 Aqueous/High Level Soil Curve on Solatek or Centurion: To prepare a calibration standard, add the appropriate volume of standard solution(s) to a 100mL volumetric flask using a micro syringe. Remove the needle quickly and mix by inverting the flask 3 times. Pour several mLs of the aqueous standard into the waste vessel, and then gently fill a 40mL VOA vial to the top. Load the vial onto the autosampler.

10.2.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in Section 10.1. The same operating conditions are used for calibration and sample analyses. Create the analytical sequence using the HP EnviroQuant data acquisition software.

Relative Response Factors: The internal standard calibration technique is used. In each calibration standard, calculate the relative response factor for each analyte and the relative standard deviation (RSD) of the response factors using the EnviroQuant data processing software. The response factors are calculated using the areas of the characteristic (quantitation) ion for each target analyte and internal standard. The calculations are performed automatically using the EnviroQuant software, using the formulae listed in Alpha's Quality Manual.

10.2.3 Initial Calibration Criteria: The following sections outline the method acceptance criteria for an initial calibration curve. All criteria must be met for the calibration to be deemed acceptable, and for sample analysis to proceed. For MCP and CT projects see the related project specific addenda for modifications.

10.2.3.1 Relative Standard Deviation Criteria: If the RSD for each target analyte is less than or equal to 20%, then the response for this compound is considered linear over the calibration range and the mean calibration factor can be used to quantitate sample results. If the 20% RSD criterion is not met for an analyte linear regression may be used if $\mathbf{r^2} \geq 0.990$, weighted linear with a weighting factor of 1/SD2 and r > 0.995, or quadratic fit if $\mathbf{r^2} \geq 0.990$. A minimum of five points are required for linear fit and six points is required for quadratic fit. The calibration must be repeated for any compounds that fail. If more than 10% of the compounds exceed the 20% RSD limit and do not achieve the minimum correlation coefficient for alternative curve fits, sample analysis cannot proceed.

10.2.3.1.1 Percent Relative Error (%RE): For linear and quadratic fit compounds the %RE relative error must be calculated for the standard at or near the midpoint of the initial calibration and at the lowest level. The %RE between the calculated and expected amounts of an analyte should be ≤ 30% for all standards, otherwise recalibration is necessary.

10.2.4 Evaluation of Retention Times: The relative retention times used for identification of target analytes are +/- 0.06 RRT (Relative Retention Time) units, based on the most recent standard run. It has been determined that these limits work well, being wide enough to eliminate false-negative results while being tight enough to eliminate false positive results. Due to the selectivity of the mass spectrometer, compound identification is more definitive than when using a less selective detector.

10.2.5 Initial Calibration Verification: After each calibration and before the analysis of samples, an ICV must be analyzed at or near the midpoint of the curve. The ICV must be prepared using a different source than the Initial Calibration and must contain all target analytes. The percent recoveries must be between 70% and 130% for target analytes

except for “difficult” analytes (Table 7), which must exhibit percent recoveries between 40% and 160%. Corrective action is required if greater than 10% of all analytes are outside the prescribed criteria.

10.2.5.1 The ICV used for the initial calibration can be used as the continuing calibration verification. See Section 10.4 for criteria.

10.2.6 Additional considerations for SIM analysis

10.2.6.1 SIM may be useful for applications requiring quantitation limits below the normal range of electron impact quadrupole mass spectrometry, and both are allowable options for this method. Using the primary m/z for quantitation and at least one secondary m/z (or product ion) for confirmation, set up the descriptor windows based on their retention times. The selected m/z values should include any mass defect noted in the target analyte mass spectra acquired on the instrument, usually less than 0.2 amu. The dwell time for each ion may be automatically calculated by the instrument software or may be calculated based on the peak widths of the analytes of interest, the number of spectra needed to be acquired across each peak, and the number of concurrent ions that need to be acquired in each segment. When fewer m/z values are monitored in each segment, the acquisition time for each m/z can be increased, thereby increasing the sensitivity of the system. The total cycle time for the MS should be short enough that at least five, but preferably ten or more, spectra are acquired per chromatographic peak.

10.2.6.2 When compounds are analyzed in SIM mode, the following best practices are recommended:

10.2.6.2.1 Monitor at least two ions for each target analyte and use the mid-point of the calibration curve to establish proper ion ratios for each compound. The ratios of primary and secondary ions are the only qualitative tools available in SIM runs (other than RT), which increases their importance in proper identification. When interferences are expected or observed in a given matrix, acquiring multiple secondary ions may aid in qualitative identification.

10.2.6.2.2 Verify that all monitored ions are correctly integrated in order to achieve proper ion ratios. Update the primary/secondary ion ratios and reference mass spectra after each ICAL using a mid-range ICAL standard.

10.3 Equipment Operation and Sample Processing

The same GC, MS, and Purge and Trap conditions used for the initial calibration must be employed for sample analysis. After verification of system performance by analysis of BFB, the continuing calibration standard and method blank, samples are analyzed and processed as described below.

10.3.1 All samples are initially screened using Tekmar 7000 Headspace Autosamplers.

10.3.2 Retrieve sample VOA vials from the sample refrigerator just prior to loading onto the purge and trap system.

10.3.3 Aqueous samples:
Load the VOA vial directly on the sampling rack. Dilutions may be prepared volumetrically and poured into VOA vials ensuring there is no headspace left in the vial. The autosampler will then sample 10mL from the VOA vial.

10.3.4 Low level soil samples:

Low level soil sample should be shaken briefly to ensure that the stir bar is loose, and will spin on the autosampler unit. Take the low level VOA vial and place directly into the rack of the autosampler unit. Surrogate and internal standards are added automatically by the autosampler prior to sample purging.

10.3.5 High level soil samples:

Shake or vortex the sample for 15 seconds, ensuring the methanol has completely penetrated the soil in the vial. Let sample settle prior to taking methanol aliquot.

10.3.5.1 Through liquid path

Load a maximum of 860µL or appropriate dilution of the methanol into a half-full VOA vial. Fill the VOA vial up to the top with water and cap with no headspace. Allow the auto-sampler to sample 10mL out of the VOA vial which would be equivalent to injecting 100µL of the methanol extract. Prepare dilutions accordingly.

10.3.5.2 Through soil path

Into a VOA vial with a stir bar added, load 4.9mL of water plus a maximum of 100 µL of methanol or appropriate dilution of methanol extract from a 5mL luerlock syringe. Cap the vial and load onto the auto-sampler.

10.3.6 Qualitative Analysis:

10.3.6.1 The qualitative identification of each compound is based on retention time and on comparison of the sample mass spectrum with the reference mass spectrum. The reference mass spectrum must be generated by the laboratory on the same GC/MS system. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity, or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:

10.3.6.1.1 The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. The EnviroQuant data system is configured to make this check.

10.3.6.1.2 The relative retention time (RRT) of the sample component is within ±0.06 RRT units of the RRT of the standard component.

10.3.6.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%.)

10.3.6.1.4 Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is ≤ 50% of the average of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs (i.e., m and p-xylene).

10.3.6.1.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra is important.

10.3.6.1.6 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra, and in qualitative identification of compounds. When analytes coelute (i.e., only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the coeluting compound.

10.3.6.2 For samples containing non-target analytes, a library search will be performed at client request. Compound identification will be classified as “tentative”, and the concentration will be reported as an estimate as no quantitative standards are run for these compounds.

10.3.6.2.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) are present in the sample spectrum.

10.3.6.2.2 The relative intensities of the major ions should agree within ±20%. (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%.)

10.3.6.2.3 Molecular ions present in the reference spectrum are present in the sample spectrum. If the molecular ion is not present, carefully review library matches in order to avoid misidentification.

10.3.6.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

10.3.6.2.5 Ions present in the reference spectrum but not in the sample spectrum are reviewed for possible subtraction from the sample spectrum because of background contamination or coeluting peaks.

10.3.6.2.6 Mass spectral library search algorithms typically assign a match factor to the peak identity based on comparison of an unknown mass spectrum to library spectra. For spectra meeting the above conditions, match factors greater than 0.85 (85%) may be considered confirming evidence. Where a known limitation in data collection is identified (e.g., the presence of an incompletely resolved spectral interference), a lower match factor may be considered confirmatory. For multiple library spectra with similar match factors (e.g., for hydrocarbons with low abundance molecular ions, or structural isomers), the tentative identification assigned to the unknown may be better represented as a more generic structure (e.g., unknown hydrocarbon, C4 benzene structural isomer).

10.3.7 Quantitative Analysis:

10.3.7.1 Quantitation of a target compound detected in a sample is performed automatically by the EnviroQuant data processing software, using the formulae found in Alpha’s Quality Manual. Either the average response factor or calibration curve will be used for sample quantitation, depending on how the particular analyte was processed in the initial calibration curve. If non-target

compounds are to be reported, the quantitation is performed automatically by the EnviroQuant software using the total area of the compound and the nearest internal standard, and assuming a relative response factor of 1.0.

10.4 Continuing Calibration

Calibration verification consists of three steps that are performed at the beginning of each 12-hour analytical shift. For MCP and CT projects see the related project specific addenda for modifications.

10.4.1 Prior to the analysis of samples or calibration standards, inject or purge 1 µL (50 ng) of the 4-Bromofluorobenzene standard (Section 8.4.4) into the GC/MS system. The resultant mass spectra for the BFB must meet the criteria given in Table 3 before sample analysis begins.

10.4.2 The initial calibration curve for each compound of interest must be verified once every 12 hours prior to sample analysis. This is accomplished by analyzing the continuing calibration check standard (Section 8.5.3).

10.4.3 A method blank must be analyzed prior to any samples, typically immediately following the continuing calibration check standard, to ensure that the analytical system is free of contaminants. The method blank must not contain any target analytes at or above the required compound reporting limits.

10.4.4 The percent difference or drift for each target analyte must be less than or equal to 20% (30% for all SIM compounds). If greater than 20% of target analytes exceed the %D criteria corrective action must be taken prior to the analysis of samples. If less than or equal to 20% of compounds exceed the criteria, corrective action is not required.

10.4.5 Internal Standard Retention Time:

The absolute retention times of the internal standards in the calibration verification standard are evaluated after data acquisition. If the absolute retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must be made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required.

10.4.6 Internal Standard Response:

If the area for any of the internal standards in the calibration verification standard changes by a factor of two (50% to 200%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must be made, as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

10.5 Preventive Maintenance

Routine preventive maintenance should be performed on the analytical system. This includes replacement of GC septa and periodic rinsing or replacement of purge and trap tubes and sparge needles. The trap should be replaced every six months or sooner if performance criteria

cannot be met. Periodic cleaning (typically twice per year) of the mass spectrometer ion source is required. More frequent source cleaning may be needed, especially if dirty samples are analyzed.

If system performance deteriorates, additional maintenance may be required. This includes replacement of injector ports and seals, clipping several inches off of the front end of the GC column, or in extreme cases the replacement of the GC column. Flushing or replacement of purge and trap lines may be necessary if they become contaminated or develop active sites.

Perform routine preventative maintenance as described throughout this SOP. Record all maintenance in the instrument logbook.

11. Data Evaluation, Calculations and Reporting

11.1 LIMS Data Corrections

Please note that the Laboratory Information Management System (LIMS) automatically adjusts soil sample results to account for the % Total Solids of the sample (as determined per Alpha SOP/07-38) and the methanol preservation dilution effect.

11.2 Data Calculations

11.2.1 Results of Aqueous Sample Analysis:

$$Concentration\ (ug/L) = \frac{(Conc.)\ (Vp)\ (DF)}{(Vs)}$$

where:

Conc. = On-column concentration obtained from the quantitation report.
Vp = Volume purged, 10 mL is standard
Vs = Volume of sample purged
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".

11.2.2 Results of Sediment/Soil, Sludge, and Waste Analysis:

All solids including soils, sediments, and sludges must be reported on a dry-weight basis.

11.2.2.1 Low-Level Samples:

$$Concentration\ (ug/Kg) = \frac{(Conc.)\ (Vp)\ (DF)}{(W)\ (\%S)}$$

11.2.2.2 High-Level Samples:

$$\text{Concentration (ug/Kg)} = \frac{(\text{Conc.})\,(Vp)\,(5000)\,(DF)}{(W)\,(Ve)\,(\%S)}$$

where:

Conc. = On-column concentration obtained from the quantitation report.
DF = Dilution factor, for manually prepared dilutions, not instrumental "dilutions".
Ve = Extract volume, mL
Vp = Volume purged, 5 mL is standard
W = Aliquot of sample (wet), g
%S = Sample % solid
5000 = Constant representing the final volume of the methanol extraction.

11.2.2.2.1 High-Level Samples Corrected for Total Water/Solvent Mixture (V_t):

Samples that are extracted prior to analysis in a water miscible solvent such as methanol are diluted by the total volume of the water/solvent mixture. The total mixture volume can only be calculated based on the sample moisture present as determined by the % moisture calculation.

$$\%\ \text{moisture} = \frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100$$

$$V_t = \frac{[\text{mL of solvent} + (\%\text{moisture} \times \text{g of sample})]}{100} \times 1000\text{mL/mL}$$

The calculated V_t value is now added to the volume of methanol in the sample (typically 5000µL), and the corrected concentration is calculated using the equation below:

$$\text{Corrected concentration (mg/Kg)} = \frac{(\text{Conc.})\,(V_t + \text{methanol vol.})\,(Vp)\,(DF)}{(W)\,(Ve)\,(\%S)}$$

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

All batch and sample specific QC criteria outlined in section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis, sample cleanup, or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP/1732. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/1732 DL/LOD/LOQ Generation

SOP/1739 IDC/DOC Generation

SOP/1728 Waste Management and Disposal SOP

16. Attachments

Table 1: 8260 REPORTING LIMITS

Table 2: 8260 QC ACCEPTANCE CRITERIA

Table 3: BFB TUNING CRITERIA

Table 4: STANDARD SOLUTIONS and ICAL Levels

Table 5: 8260D Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Table 6: 8260D Quantitation Ions

Table 7: Difficult Analytes

Table 8: ICAL Low Point re-quantitation ranges

Table 1

Standard Reported Detection Limits

US EPA METHOD 8260D and 5035A/8260D

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG)(1)	RDL (µg/KG) (2)
Acetone (3,4,5)	0.01	5.0	25	1250
Acrolein (5)		5.0	25	1250
Acrylonitrile (3,4)		5.0	4	200
Allyl Chloride (7)		N/A	5	250
Benzene (3,4,5)	0.2	0.5	0.5	25
Bromobenzene (3,4)		2.5	2	100
Bromochloromethane (3,4,5)	0.1	2.5	2	100
Bromodichloromethane (3,4,5)	0.300	0.5	0.5	25
Bromoform (3,4,5)	0.100	2.0	4	200
Bromomethane (3,4,5)	0.01	1.0	2	100
2-Butanone (3,4,5)	0.01	5.0	10	500
Butyl acetate (7)		N/A	5	50
n-Butylbenzene (3,4)		0.5	1	50
sec-Butylbenzene (3,4)		0.5	1	50
tert-Butylbenzene (3,4)		2.5	2	100
Carbon disulfide (3,4,5)	0.100	5.0	10	500
Carbon tetrachloride (3,4,5)	0.100	0.5	1	50
Chlorobenzene (3,4,5)	0.400	0.5	0.5	25
Chlorodifluoromethane (7)		N/A	5	250
Chloroethane (3,4,5)	0.01	1.0	2	100
2-Chloroethylvinyl ether (3)		10.0	20	1000
Chloroform (3,4,5)	0.300	0.75	1.5	75
Chloromethane (3,4,5)	0.01	2.5	4	200
o-Chlorotoluene (3,4)		2.5	2	100
Cyclohexane (5)	0.01	10	10	500
Cyclohexanone		10	20	1000
p-Chlorotoluene (3,4)		2.5	2	100
cis-Decahydronaphthalene (7)		N/A	5	250
trans-Decahydronaphthalene (7)		N/A	5	250
n-Decane (7)		N/A	5	250
Dibromochloromethane (3,4,5)	0.200	0.5	1	50
1,2-Dibromo-3-chloropropane (3,4,5)	0.01	2.5	3	150
1,2-Dibromoethane (3,4,5)	0.200	2.0	1	50
Dibromomethane (3,4)		5.0	2	100
1,2-Dichlorobenzene (3,4,5)	0.600	2.5	2	100
1,3-Dichlorobenzene (3,4,5)	0.500	2.5	2	100
1,4-Dichlorobenzene (3,4,5)	0.600	2.5	2	100
1,4-Dichlorobutane (3,4)		5.0	10	500
trans-1,4-Dichloro-2-butene (3,4)		2.5	5	250

Table 1 (continued)

Standard Reported Detection Limits

US EPA METHOD 8260D and 5035A/8260D

Analyte	Recommended Minimum Response Factor	RDL (µg/L)	RDL(µg/KG)(1)	RDL (µg/KG) (2)
Dichlorodifluoromethane (3,4,5)	0.01	5.0	10	500
1,1-Dichloroethane (3,4,5)	0.300	0.75	1	50
1,2-Dichloroethane (3,4,5)	0.07	0.5	1	50
1,1-Dichloroethene (3,4,5)	0.06	0.5	1	50
cis-1,2-Dichloroethene (3,4,5)	0.200	0.5	1	50
trans-1,2-Dichloroethene (3,4,5)	0.100	0.75	1.5	75
1,2-Dichloropropane (3,4,5)	0.200	1.75	1	50
1,3-Dichloropropane (3,4)		2.5	2	100
2,2-Dichloropropane (3,4)		2.5	2	100
1,1-Dichloropropene (3,4)		2.5	0.5	25
cis-1,3-Dichloropropene (3,4,5)	0.300	0.5	0.5	25
p-Diethylbenzene (4)		2.0	2	100
Diisopropyl Ether (6)		2.0	2	100
1,4-Dioxane (5) (non-SIM)		250.0	80	4000
trans-1,3-Dichloropropene (3,4,5)	0.300	0.5	1	50
Ethanol (7)		250.0	1000	50000
Ethyl acetate		10.0	10	500
Ethylbenzene (3,4,5)	0.400	0.5	1	50
Ethyl ether (3,4)		2.5	2	100
4-Ethyltoluene (4)		2.0	2	100
Ethyl methacrylate (3,4)		5.0	10	500
Ethyl-Tert-Butyl-Ether (6)		2.0	2	100
Freon-113 (5)	0.05	10.0	4	200
n-Heptane (7)		N/A	5	250
Hexachlorobutadiene (3,4)		0.5	4	200
Hexachloroethane (7)		N/A	5	250
Hexane		1.0	5	250
2-Hexanone (3,4,5)	0.01	5.0	10	500
Iodomethane		5.0	10	500
Isopropyl Alcohol (IPA)		100.0	100	5000
Isopropylbenzene (3,4,5)	0.400	0.5	1	50
p-Isopropyltoluene (3,4)		0.5	1	50
Limonene (7)		N/A	5	250
Methyl Acetate (5)	0.01	10.0	4	200
Methylene chloride (3,4,5)	0.01	3.0	5	250
Methyl Cyclohexane (5)	0.05	10.0	4	200
Methyl isothiocyanate (7)		N/A	5	250
Methyl Methacrylate		2.5	5	250
4-Methyl-2-pentanone (3,4,5)	0.03	5.0	10	500

Methyl-tert-butyl-ether (3,4,5)	0.100	1.0	2	100
Naphthalene (3,4)		2.5	4	200
Nitrobenzene (7)		N/A	5	250
2-Nitropropane (7)		N/A	5	250
n-Nonane (7)		N/A	5	250
n-Octane (7)		N/A	5	250
n-Butanol (5)		100.0	300	15000
n-Propylbenzene (3,4)		0.5	1	50
n-Propyl bromide		5.0	N/A	N/A
Pentachloroethane		2.0	N/A	N/A
Styrene (3,4,5)	0.200	1.0	1	50
Tert-Butyl Alcohol (5)		10.0	20	1000
Tertiary-Amyl Methyl Ether (6)		2.0	2	100
1,1,1,2-Tetrachloroethane (3,4)		0.5	0.5	25
1,2,4,5-Tetramethylbenzene (4)		2.0	2	100
1,1,2,2-Tetrachloroethane (3,4,5)	0.200	0.5	0.5	25
Tetrachloroethene (3,4,5)	0.100	0.5	0.5	25
Tetrahydrofuran (3)		5.0	4	200
Toluene (3,4,5)	0.300	0.75	1	50
1,2,3-Trichlorobenzene (3,4,5)	0.400	2.5	2	100
1,2,4-Trichlorobenzene (3,4,5)	0.400	2.5	2	100
1,3,5-Trichlorobenzene (6)		2.0	2	100
1,1,1-Trichloroethane (3,4,5)	0.05	0.5	0.5	25
1,1,2-Trichloroethane (3,4,5)	0.200	0.75	1	50
Trichloroethene (3,4,5)	0.200	0.5	0.5	25
Trichlorofluoromethane (3,4,5)	0.01	2.5	4	200
1,2,3-Trichloropropane (3,4)		5.0	10	500
1,2,4-Trimethylbenzene (3,4)		2.5	2	100
1,3,5-Trimethylbenzene (3,4)		2.5	2	100
n-Undecane (7)		N/A	5	250
Vinyl acetate (3,4)		5.0	10	500
Vinyl chloride (3,4,5)	0.01	1.0	1	50
m/p-Xylenes (3,4,5)	0.200	1.0	2	100
o-Xylene (3,4,5)	0.200	1.0	1	50
Isobutyl Alcohol		10.0	N/A	N/A
2-Butanol		25.0	N/A	N/A
4-penten-2-ol		100.0	N/A	N/A
2-methyl-2-butanol		25.0	N/A	N/A
4-methyl-2-pentanol		25.0	N/A	N/A
Chloropicrin		20.0	100	5000
Halothane		2.5	10	500
1,4-Dioxane (5) SIM		3.0	N/A	N/A
1,1,2,2-Tetrachloroethane SIM		0.1	N/A	N/A

(1) Detection Limits are for Low-level Aqueous preserved samples.

(2) Detection Limits are for High-level Methanol preserved samples.
(3) Analyte reported by standard 8260 reporting list.
(4) Analyte reported by New York TCL reporting list.
(5) Analyte reported by New Jersey TCL reporting list.
(6) Analyte reported for New Hampshire in addition to standard 8260 reporting list.
(7) Analyte only reported for New York TCL report upon client request.
(8) Note: Reporting Limits are based on standard 8260 reporting list, RL's may vary for NY and NJ reporting lists.

Table 2

QUALITY CONTROL ACCEPTANCE CRITERIA

Surrogate Spike Percent Recovery	Aqueous Limits		Soil Limits	
	Lower Control Limit	Upper Control Limit	Lower Control Limit	Upper Control Limit
1,2-Dichloroethane-d_4	70%	130%	70%	130%
4-Bromofluorobenzene	70%	130%	70%	130%
Toluene-d_8	70%	130%	70%	130%
Dibromofluoromethane	70%	130%	70%	130%

Table 3

BFB (4-BROMOFLUOROBENZENE) MASS INTENSITY CRITERIA

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95 (not required)
75	30 to 60% of m/z 95 (not required)
95	50 to 200% of m/z 174
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	50 to 200% of m/z 95
175	5 to 9% of m/z 174
176	95% to 105% of m/z 174
177	5 to 10% of m/z 176

Table 4

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
Fluorobenzene	2500	20	20	20	20	20	20	20	20	20	20
Dichlorodifluoromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorodifluoromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chloromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Vinyl chloride	2000	0.5	1	2	4	20	40	60	100	200	300
Bromomethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Trichlorofluoromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
Ethanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
1,1-Dichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
Carbon disulfide	2000	0.5	1	2	4	20	40	60	100	200	300
Freon-113	2000	0.5	1	2	4	20	40	60	100	200	300
Iodomethane	2000	0.5	1	2	4	20	40	60	100	200	300
Acrolein	2000	0.5	1	2	4	20	40	60	100	200	300
Allyl chloride	2000	0.5	1	2	4	20	40	60	100	200	300
Methylene chloride	2000	0.5	1	2	4	20	40	60	100	200	300
Isopropyl alcohol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
Acetone	2000	0.5	1	2	4	20	40	60	100	200	300
trans-1,2-Dichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl acetate	2000	0.5	1	2	4	20	40	60	100	200	300
Hexane	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl tert-butyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
tert-Butyl alcohol	2000	2.5	5	10	20	100	200	300	500	1000	1500
Diisopropyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
1,1-Dichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Halothane	2000	0.5	1	2	4	20	40	60	100	200	300
Acrylonitrile	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl tert-butyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
Vinyl acetate	2000	0.5	1	2	4	20	40	60	100	200	300
cis-1,2-Dichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
2,2-Dichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
Bromochloromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Cyclohexane	2000	0.5	1	2	4	20	40	60	100	200	300

Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
Chloroform	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl acetate	2000	0.5	1	2	4	20	40	60	100	200	300
Carbon tetrachloride	2000	0.5	1	2	4	20	40	60	100	200	300
Tetrahydrofuran	2000	0.5	1	2	4	20	40	60	100	200	300
Dibromofluoromethane	2500	20	20	20	20	20	20	20	20	20	20
1,1,1-Trichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
2-Butanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
2-Butanone	2000	0.5	1	2	4	20	40	60	100	200	300
1,1-Dichloropropene	2000	0.5	1	2	4	20	40	60	100	200	300
Heptane	2000	0.5	1	2	4	20	40	60	100	200	300
Benzene	2000	0.5	1	2	4	20	40	60	100	200	300
tert-Amyl methyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dichloroethane-d4	2500	20	20	20	20	20	20	20	20	20	20
1,2-Dichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Isobutyl alcohol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
2-Methyl-2-butanol	2000	2.5	5	10	20	100	200	300	500	1000	1500
Methyl cyclohexane	2000	0.5	1	2	4	20	40	60	100	200	300
Trichloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
Dibromomethane	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
4-penten-2-ol	2000	2.5	5	10	20	100	200	300	500	1000	1500
2-Chloroethyl vinyl ether	2000	0.5	1	2	4	20	40	60	100	200	300
Bromodichloromethane	2000	0.5	1	2	4	20	40	60	100	200	300
Ethyl acrylate	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl methacrylate	2000	0.5	1	2	4	20	40	60	100	200	300
1,4-Dioxane	2000	N/A	40	80	200	1000	2000	3000	5000	10000	15000
cis-1,3-Dichloropropene	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorobenzene-d5	2500	20	20	20	20	20	20	20	20	20	20
Octane	2000	0.5	1	2	4	20	40	60	100	200	300
Toluene-d8	2500	20	20	20	20	20	20	20	20	20	20
Toluene	2000	0.5	1	2	4	20	40	60	100	200	300
4-Methyl-2-pentanone	2000	0.5	1	2	4	20	40	60	100	200	300
Tetrachloroethene	2000	0.5	1	2	4	20	40	60	100	200	300
2-Nitropropane	2000	0.5	1	2	4	20	40	60	100	200	300

Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
Chloropicrin	2000	N/A	50	N/A	100	200	300	400	500	600	700
trans-1,3-Dichloropropene	2000	0.5	1	2	4	20	40	60	100	200	300
Methyl isothiocyanate	2000	0.5	1	2	4	20	40	60	100	200	300
4-Methyl-2-pentanol	2000	N/A	20	N/A	80	200	400	600	1000	3000	4000
Ethyl methacrylate	2000	0.5	1	2	4	20	40	60	100	200	300
1,1,2-Trichloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorodibromomethane	2000	0.5	1	2	4	20	40	60	100	200	300
1,3-Dichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dibromoethane	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butyl Acetate	2000	0.5	1	2	4	20	40	60	100	200	300
2-Hexanone	2000	0.5	1	2	4	20	40	60	100	200	300
Nonane	2000	0.5	1	2	4	20	40	60	100	200	300
Chlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Ethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,1,1,2-Tetrachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
p/m Xylene	2000	1	2	4	8	40	80	120	200	400	600
o Xylene	2000	1	2	4	8	40	80	120	200	400	600
Styrene	2000	1	2	4	8	40	80	120	200	400	600
1,4-Dichlorobenzene-d4	2500	20	20	20	20	20	20	20	20	20	20
Bromoform	2000	0.5	1	2	4	20	40	60	100	200	300
Butyl acrylate	2000	0.5	1	2	4	20	40	60	100	200	300
Isopropylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
4-Bromofluorobenzene	2500	20	20	20	20	20	20	20	20	20	20
Bromobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Decane	2000	0.5	1	2	4	20	40	60	100	200	300
n-Propylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,4-Dichlorobutane	2000	0.5	1	2	4	20	40	60	100	200	300
1,1,2,2-Tetrachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
4-Ethyltoluene	2000	0.5	1	2	4	20	40	60	100	200	300
2-Chlorotoluene	2000	0.5	1	2	4	20	40	60	100	200	300
1,3,5-Trimethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,3-Trichloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
trans-1,4-Dichloro-2-butene	2000	0.5	1	2	4	20	40	60	100	200	300
4-Chlorotoluene	2000	0.5	1	2	4	20	40	60	100	200	300

Soil	Stock (µg/mL)	Level 0 (ug/kg)	Level 1 (ug/kg)	Level 1.5 (ug/kg)	Level 2 (ug/kg)	Level 3 (ug/kg)	Level 4 (ug/kg)	Level 5 (ug/kg)	Level 6 (ug/kg)	Level 7 (ug/kg)	Level 8 (ug/kg)
tert-Butylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Pentachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butyl methacrylate	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,4-Trimethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Limonene	2000	0.5	1	2	4	20	40	60	100	200	300
sec-Butylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
p-Isopropyltoluene	2000	0.5	1	2	4	20	40	60	100	200	300
1,3-Dichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,4-Dichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
trans-Decahydronaphthalene	2000	0.5	1	2	4	20	40	60	100	200	300
Undecane	2000	0.5	1	2	4	20	40	60	100	200	300
p-Diethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
n-Butylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Hexachloroethane	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
cis-Decahydronaphthalene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,4,5-Tetramethylbenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2-Dibromo-3-chloropropane	2000	0.5	1	2	4	20	40	60	100	200	300
1,3,5-Trichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Nitrobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Hexachlorobutadiene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,4-Trichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
Naphthalene	2000	0.5	1	2	4	20	40	60	100	200	300
1,2,3-Trichlorobenzene	2000	0.5	1	2	4	20	40	60	100	200	300
1,3-Dioxolane	2000	N/A	25	N/A	50	100	250	500	1000	1500	2000

Stock Standard Concentrations and Suggested Calibration Concentration Levels

Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
Fluorobenzene	2500	10	10	10	10	10	10	10	10	10	10	10
Dichlorodifluoromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Chloromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Vinyl chloride	2000	0.19	0.5	2	10	30	50	80	100	120	160	200
Bromomethane	2000		0.5	2	10	30	50	80	100	120	160	200
Chloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Trichlorofluoromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
Ethanol	2000		0.5	2	10	30	50	80	100	120	160	200
1,1-Dichloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
Carbon disulfide	2000		0.5	2	10	30	50	80	100	120	160	200
Freon-113	2000		0.5	2	10	30	50	80	100	120	160	200
Iodomethane	2000		0.5	2	10	30	50	80	100	120	160	200
Acrolein	2000		0.5	2	10	30	50	80	100	120	160	200
Methylene chloride	2000		0.5	2	10	30	50	80	100	120	160	200
Isopropyl alcohol	2000		2.5	10	50	150	250	400	500	600	800	1000
Acetone	2000		0.5	2	10	30	50	80	100	120	160	200
trans-1,2-Dichloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
Methyl acetate	2000		0.5	2	10	30	50	80	100	120	160	200
Methyl tert-butyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
tert-Butyl alcohol	2000		2.5	10	50	150	250	400	500	600	800	1000
Diisopropyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
1,1-Dichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Halothane	2000		0.5	2	10	30	50	80	100	120	160	200
Acrylonitrile	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl tert-butyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
Vinyl acetate	2000		0.5	2	10	30	50	80	100	120	160	200
cis-1,2-Dichloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
2,2-Dichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
Bromochloromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Cyclohexane	2000		0.5	2	10	30	50	80	100	120	160	200
Chloroform	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl acetate	2000		0.5	2	10	30	50	80	100	120	160	200

Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
Carbon tetrachloride	2000	0.19	0.5	2	10	30	50	80	100	120	160	200
Tetrahydrofuran	2000		0.5	2	10	30	50	80	100	120	160	200
Dibromofluoromethane	2500	10	10	10	10	10	10	10	10	10	10	10
1,1,1-Trichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
2-Butanol	2000		2.5	10	50	150	250	400	500	600	800	1000
2-Butanone	2000		0.5	2	10	30	50	80	100	120	160	200
1,1-Dichloropropene	2000		0.5	2	10	30	50	80	100	120	160	200
Benzene	2000	0.19	0.5	2	10	30	50	80	100	120	160	200
tert-Amyl methyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dichloroethane-d4	2500	10	10	10	10	10	10	10	10	10	10	10
1,2-Dichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Isobutyl alcohol	2000		2.5	10	50	150	250	400	500	600	800	1000
2-Methyl-2-butanol	2000		2.5	10	50	150	250	400	500	600	800	1000
Methyl cyclohexane	2000		0.5	2	10	30	50	80	100	120	160	200
Trichloroethene	2000	0.19	0.5	2	10	30	50	80	100	120	160	200
n-Butanol	2000		2.5	10	50	150	250	400	500	600	800	1000
Dibromomethane	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
4-penten-2-ol	2000		2.5	10	50	150	250	400	500	600	800	1000
2-Chloroethyl vinyl ether	2000		0.5	2	10	30	50	80	100	120	160	200
Bromodichloromethane	2000		0.5	2	10	30	50	80	100	120	160	200
Ethyl acrylate	2000		0.25	1	5	15	25	40	50	60	80	100
Methyl methacrylate	2000		0.25	1	5	15	25	40	50	60	80	100
1,4-Dioxane	2000		100	400	500	600	1000	800	1000	1200	1600	2000
cis-1,3-Dichloropropene	2000		0.5	2	10	30	50	80	100	120	160	200
Chlorobenzene-d5	2500	10	10	10	10	10	10	10	10	10	10	10
Toluene-d8	2500	10	10	10	10	10	10	10	10	10	10	10
Toluene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Methyl-2-pentanone	2000		0.5	2	10	30	50	80	100	120	160	200
Tetrachloroethene	2000		0.5	2	10	30	50	80	100	120	160	200
Chloropicrin	2000		30	50	80	120	200	160	400	200	320	400
trans-1,3-Dichloropropene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Methyl-2-pentanol	2000		2.5	10	50	150	250	400	500	600	800	1000

Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
Ethyl methacrylate	2000		0.5	2	10	30	50	80	100	120	160	200
1,1,2-Trichloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
Chlorodibromomethane	2000		0.5	2	10	30	50	80	100	120	160	200
1,3-Dichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dibromoethane	2000		0.5	2	10	30	50	80	100	120	160	200
2-Hexanone	2000		0.5	2	10	30	50	80	100	120	160	200
Chlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Ethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,1,1,2-Tetrachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
p/m Xylene	2000		1	4	20	60	100	160	200	240	320	400
o Xylene	2000		1	4	20	60	100	160	200	240	320	400
Styrene	2000		1	4	20	60	100	160	200	240	320	400
1,4-Dichlorobenzene-d4	2500	10	10	10	10	10	10	10	10	10	10	10
Bromoform	2000		0.5	2	10	30	50	80	100	120	160	200
Butyl acrylate	2000		0.25	1	5	15	25	40	50	60	80	100
Isopropylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Bromofluorobenzene	2500	10	10	10	10	10	10	10	10	10	10	10
Bromobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
n-Propylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,4-Dichlorobutane	2000		0.5	2	10	30	50	80	100	120	160	200
1,1,2,2-Tetrachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
4-Ethyltoluene	2000		0.5	2	10	30	50	80	100	120	160	200
2-Chlorotoluene	2000		0.5	2	10	30	50	80	100	120	160	200
1,3,5-Trimethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,3-Trichloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
trans-1,4-Dichloro-2-butene	2000		0.5	2	10	30	50	80	100	120	160	200
4-Chlorotoluene	2000		0.5	2	10	30	50	80	100	120	160	200
tert-Butylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Pentachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
n-Butyl methacrylate	2000		0.25	1	5	15	25	40	50	60	80	100
1,2,4-Trimethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
sec-Butylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200

Water	Stock (µg/mL)	Level 11 (ug/L)	Level 1 (ug/L)	Level 2 (ug/L)	Level 3 (ug/L)	Level 4 (ug/L)	Level 5 (ug/L)	Level 6 (ug/L)	Level 7 (ug/L)	Level 8 (ug/L)	Level 9 (ug/L)	Level 10 (ug/L)
							Optional		Optional		Optional	
p-Isopropyltoluene	2000		0.5	2	10	30	50	80	100	120	160	200
1,3-Dichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,4-Dichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
p-Diethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
n-Butylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,4,5-Tetramethylbenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2-Dibromo-3-chloropropane	2000		0.5	2	10	30	50	80	100	120	160	200
1,3,5-Trichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Hexachlorobutadiene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,4-Trichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
Naphthalene	2000		0.5	2	10	30	50	80	100	120	160	200
1,2,3-Trichlorobenzene	2000		0.5	2	10	30	50	80	100	120	160	200
1,3-Dioxolane	2000		10	40	100	250	N/A	500	N/A	750	N/A	1000
Pentachloroethane	2000		0.5	2	10	30	50	80	100	120	160	200
1,4-Dioxane (SIM)	100		0.5	2	10	20	30	50	100	200	N/A	N/A
1,1,2,2-Tetrachloroethane (SIM)	100		0.05	0.1	0.2	0.5	1	2	5	10	N/A	N/A

- For Low Level Soil analysis, the calibration levels are the same in µg/Kg units.
- For High Level Soil analysis, the calibration levels are at 50x the levels listed due to sample preparation requirements.

Table 5

8260D Volatile Internal Standards with Corresponding Target Compounds and Surrogates Assigned for Quantitation

Fluorobenzene	Chlorobenzene-d5	1,4-Dichlorobenzene-d4
Dichlorodifluoromethane	Toluene-d8 (surr)	Isopropylbenzene
Chloromethane	Toluene	Bromoform
Vinyl Chloride	Ethyl Methacrylate	1,4-dichloro-2-butane
Bromomethane	Trans-1,3-dichloropropene	1,1,2,2,-tetrachloroethane
Chloroethane	1,1,2-trichloroethane	4-bromofluorobenzene (surr)
Trichlorofluoromethane	2-hexanone	1,2,3-trichloropropane
Ethyl Ether	1,3-dichloropropane	trans-1,4-dichloro-2-butene
Freon 113	Tetrachloroethene	n-propylbenzene
Acrolein	Chlorodibromomethane	Bromobenzene
Acetone	1,2-dibromoethane	4-ethyltoluene
Ethanol	Chlorobenzene	1,3,5-trimethybenzene
1,1,-dichloroethene	1,1,1,2-tetrachloroethane	2-chlorotoluene
Tert-Butyl Alcohol	Ethylbenzene	4-chorotoluene
Methyl Acetate	p/m xylene	tert-butylbenzene
Carbon Disulfide	o xylene	1,2,4-trimethylbenzene
Methylene Chloride	Styrene	sec-butylbenzene
Acrylonitrile	Octane	p-isopropyltoluene
Methyl Tert Butyl Ether	2-Nitropropane	1,3-dichlorobenzene
Halothane	Methyl isothiocyanate	1,4-dichlorobenzene
Trans-1,2-dichloroethene	n-Butyl acetate	n-butylbenzene
Diisopropyl Ether	Nonane	p-diethylbenzene
Vinyl Acetate		1,2-dichlorobenzene
1,1-dichloroethane		1,2,4,5-tetramethylbenzene
Ethyl-Tert-Butyl-Ether		1,2-dibromo-3-chloropropane
2-butanone		1,3,5-trichlorobenzene
2,2-dichloropropane		1,2,4-trichlorobenzene
Cis-1,2-dichloroethene		Hexachlorobutadiene
Chloroform		Naphthalene
Bromochloromethane		1,2,3-trichlorobenzene
Tetrahydrofuran		Cyclohexanone
Dibromofluoromethane (surr)		Nitrobenzene

1,1,1-trichloroethane		Pentachloroethane
Cyclohexane		Decane
1,1-dichloropropene		Limonene
carbon Tetrachloride		trans-Decahydronaphthalene
Tertiary-Amyl Methyl Ether		Undecane
1,2-dichloroethane-d4 (surr)		Hexachloroethane
1,2-dichloroethane		cis-Decahydronaphthalene
Benzene		
Trichloroethene		
Methyl cyclohexane		
1,2-Dichloropropane		
Bromodichloromethane		
1,4-Dioxane		
Dibromomethane		
2-Chloroethylvinyl Ether		
4-methyl-2-pentanone		
cis-1,3-Dichloropropene		
Iodomethane		
Methyl methacrylate		
n-Butanol		
Ethyl acetate		
Isopropyl alcohol (IPA)		
Hexane		
n-Propyl bromide		
Chlorodifluoromethane		
Allyl chloride		
Heptane		

Table 6

8260D Quantitation Ions

Analyte	Quantitation Ion	Analyte	Quantitation Ion
Dichlorodifluoromethane	85	Ethyl Methacrylate	69
Chloromethane	50	Trans-1,3-dichloropropene	75
Vinyl Chloride	62	1,1,2-trichloroethane	83
Bromomethane	94	2-hexanone	43
Chloroethane	64	1,3-dichloropropane	76
Trichlorofluoromethane	101	Tetrachloroethene	166
Ethyl Ether	74	Chlorodibromomethane	129
Freon 113	101	1,2-dibromoethane	107
Acrolein	56	Chlorobenzene	112
Acetone	43	1,1,1,2-tetrachloroethane	131
1,1,-dichloroethene	96	Ethylbenzene	91
Tert-Butyl Alcohol	59	p/m xylene	106
Methyl Acetate	43	o xylene	106
Carbon Disulfide	84	Styrene	104
Methylene Chloride	76	Isopropylbenzene	105
Acrylonitrile	53	Bromoform	173
Methyl Tert Butyl Ether	73	1,4-dichloro-2-butane	55
Halothane	117	1,1,2,2,-tetrachloroethane	83
Trans-1,2-dichloroethene	96	1,2,3-trichloropropane	75
Diisopropyl Ether	45	Trans-1,4-dichloro-2-butene	53
Vinyl Acetate	43	n-propylbenzene	91
1,1-dichloroethane	63	Bromobenzene	156
Ethyl-Tert-Butyl-Ether	59	4-ethyltoluene	105
2-butanone	43	1,3,5-trimethybenzene	105
2,2-dichloropropane	77	2-chlorotoluene	91
Cis-1,2-dichloroethene	96	4-chlorotoluene	91
Chloroform	83	tert-butylbenzene	119
Bromochloromethane	128	1,2,4-trimethylbenzene	105
Tetrahydrofuran	42	sec-butylbenzene	105
1,1,1-trichloroethane	97	p-isopropyltoluene	119
Cyclohexane	56	1,3-dichlorobenzene	146
1,1-dichloropropene	75	1,4-dichlorobenzene	146
Carbon Tetrachloride	117	n-butylbenzene	91
Tertiary-Amyl Methyl Ether	73	p-diethylbenzene	119
1,2-dichloroethane	62	1,2-dichlorobenzene	146
Benzene	78	1,2,4,5-Tetramethylbenzene	119
Trichloroethene	95	1,2-Dibromo-3-chloropropane	75
Methyl Cyclohexane	83	1,3,5-Trichlorobenzene	180
1,2-Dichloropropane	63	1,2,4-Trichlorobenzene	180
Bromodichloromethane	83	Hexachlorobutadiene	225
1,4-dioxane	88	Naphthalene	128
Dibromomethane	93	1,2,3-trichlorobenzene	180
2-Chloroethylvinyl Ether	63	Ethanol	45
4-methyl-2-pentanone	58	Cyclohexanone	55
Cis-1,3-dichloropropene	75	Ethyl acetate	43

Table 6

8260D Quantitation Ions (continued)

Analyte	Quantitation Ion	Analyte	Quantitation Ion
Toluene	92	Iodomethane	142
Methyl methacrylate	69	n-Butanol	56
Pentachloroethane	167	Isopropyl Alcohol (IPA)	45
Hexane	57	n-Propyl bromide	43
Chlorodifluoromethane	51	Iodomethane	142
Allyl chloride	76	Heptane	71
Octane	85	2-Nitropropane	41
Methyl isothiocyanate	73	n-Butyl Acetate	43
Nonane	57	Decane	57
Limonene	68	Undecane	57
trans-Decahydronaphthalene	138	cis-Decahydronaphthalene	138
Hexachloroethane	117	Nitrobenzene	77

Table 7

List of 8260 Difficult Analytes:

1,1,2,2-Tetrachloroethane
1,2-Dibromo-3-chloropropane (DBCP)
1,4-Dioxane
2-Butanone
2-chloroethylvinyl ether
2-Hexanone
2,2-dichloropropane
4-Methyl-2-pentanone
Acetone
Bromoform
Bromomethane
Carbon disulfide
Chloroethane
Chloromethane
cis-1,3-Dichloropropene
Dichlorodifluoromethane (Freon 12)
Ethanol
Iodomethane
Isobutyl Alcohol
Naphthalene
Nitrobenzene
n-butanol
Styrene
Tert-Butyl Alcohol
Trichlorofluoromethane (Freon 11)
Isopropyl Alcohol (IPA)

Table 8

ICAL Low Point re-quantitation ranges

Water	Level 11 (ug/L)	L11 (70% - 130%)	Level 1 (ug/L)	L1 (70% - 130%)	Level 2 (ug/L)	L2 (70% - 130%)	Level 3 (ug/L)	L3 (70% - 130%)
Dichlorodifluoromethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Chloromethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Vinyl chloride	0.19	0.133 - 0.247	0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Bromomethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Chloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Trichlorofluoromethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Ethyl ether			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Ethanol			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1-Dichloroethene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Carbon disulfide			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Freon-113			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Iodomethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Acrolein			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Methylene chloride			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Isopropyl alcohol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65

Water	Level 11 (ug/L)	L11 (70% - 130%)	Level 1 (ug/L)	L1 (70% - 130%)	Level 2 (ug/L)	L2 (70% - 130%)	Level 3 (ug/L)	L3 (70% - 130%)
Acetone			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
trans-1,2-Dichloroethene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Methyl acetate			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Methyl tert-butyl ether			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
tert-Butyl alcohol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
Diisopropyl ether			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1-Dichloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Halothane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Acrylonitrile			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Ethyl tert-butyl ether			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Vinyl acetate			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
cis-1,2-Dichloroethene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
2,2-Dichloropropane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Bromochloromethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Cyclohexane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Chloroform			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Ethyl acetate			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Carbon tetrachloride	0.19	0.133 - 0.247	0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Tetrahydrofuran			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1,1-Trichloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
2-Butanol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
2-Butanone			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1-Dichloropropene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Benzene	0.19	0.133 - 0.247	0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
tert-Amyl methyl ether			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2-Dichloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Isobutyl alcohol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
2-Methyl-2-butanol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
Methyl cyclohexane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Trichloroethene	0.19	0.133 - 0.247	0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
n-Butanol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
Dibromomethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2-Dichloropropane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
4-penten-2-ol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
2-Chloroethyl vinyl ether			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Bromodichloromethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Ethyl acrylate			0.25	0.175 - 0.325	1	0.7 - 1.3	5	3.5 - 6.5
Methyl methacrylate			0.25	0.175 - 0.325	1	0.7 - 1.3	5	3.5 - 6.5
1,4-Dioxane			100	70 - 130	400	280 - 520	500	350 - 650
cis-1,3-Dichloropropene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Toluene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
4-Methyl-2-pentanone			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Tetrachloroethene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0

Water	Level 11 (ug/L)	L11 (70% - 130%)	Level 1 (ug/L)	L1 (70% - 130%)	Level 2 (ug/L)	L2 (70% - 130%)	Level 3 (ug/L)	L3 (70% - 130%)
Chloropicrin			30	21 - 39	50	35 - 65	80	56 - 104
trans-1,3-Dichloropropene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
4-Methyl-2-pentanol			2.5	1.75 - 3.25	10	7 - 13.0	50	35 - 65
Ethyl methacrylate			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1,2-Trichloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Chlorodibromomethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,3-Dichloropropane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2-Dibromoethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
2-Hexanone			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Chlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Ethylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1,1,2-Tetrachloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
p/m Xylene			1	0.7 - 1.3	4	2.8 - 5.2	20	14 - 26
o Xylene			1	0.7 - 1.3	4	2.8 - 5.2	20	14 - 26
Styrene			1	0.7 - 1.3	4	2.8 - 5.2	20	14 - 26
Bromoform			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Butyl acrylate			0.25	0.175 - 0.325	1	0.7 - 1.3	5	3.5 - 6.5
Isopropylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Bromobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
n-Propylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,4-Dichlorobutane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1,2,2-Tetrachloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
4-Ethyltoluene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
2-Chlorotoluene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,3,5-Trimethylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2,3-Trichloropropane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
trans-1,4-Dichloro-2-butene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
4-Chlorotoluene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
tert-Butylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Pentachloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
n-Butyl methacrylate			0.25	0.175 - 0.325	1	0.7 - 1.3	5	3.5 - 6.5
1,2,4-Trimethylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
sec-Butylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
p-Isopropyltoluene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,3-Dichlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,4-Dichlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
p-Diethylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
n-Butylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2-Dichlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2,4,5-Tetramethylbenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2-Dibromo-3-chloropropane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,3,5-Trichlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
Hexachlorobutadiene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2,4-Trichlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0

Naphthalene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,2,3-Trichlorobenzene			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,3-Dioxolane			10	7 - 13.0	40	28 - 52	100	70 - 130
Pentachloroethane			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,4-Dioxane (SIM)			0.5	0.35 - 0.65	2	1.4 - 2.6	10	7 - 13.0
1,1,2,2-Tetrachloroethane (SIM)			0.05	0.035 - 0.065	0.1	0.07 - 0.13	0.2	0.14 - 0.26

ICAL Low Point re-quantitation ranges

Soil	Level 0 (ug/kg)	L0 (70% - 130%)	Level 1 (ug/kg)	L1 (70% - 130%)	Level 1.5 (ug/kg)	L1.5 (70% - 130%)	Level 2 (ug/kg)	L2 (70% - 130%)	Level 3 (ug/kg)	L3 (70% - 130%)
Dichlorodifluoromethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chlorodifluoromethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chloromethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Vinyl chloride	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Bromomethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Trichlorofluoromethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Ethyl ether	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Ethanol	N/A	N/A	20	14 - 26	N/A	N/A	80	56 -104	200	140 - 260
1,1-Dichloroethene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Carbon disulfide	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Freon-113	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Iodomethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Acrolein	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Allyl chloride	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Methylene chloride	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Isopropyl alcohol	N/A	N/A	20	14 - 26	N/A	N/A	80	56 -104	200	140 - 260
Acetone	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
trans-1,2-Dichloroethene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Methyl acetate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Hexane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26

Soil	Level 0 (ug/kg)	L0 (70% - 130%)	Level 1 (ug/kg)	L1 (70% - 130%)	Level 1.5 (ug/kg)	L1.5 (70% - 130%)	Level 2 (ug/kg)	L2 (70% - 130%)	Level 3 (ug/kg)	L3 (70% - 130%)
Methyl tert-butyl ether	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
tert-Butyl alcohol	2.5	1.75 - 3.25	5	3.5 - 6.5	10	7.0 - 13.	20	14 - 26	100	70 - 130
Diisopropyl ether	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,1-Dichloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Halothane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Acrylonitrile	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Ethyl tert-butyl ether	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Vinyl acetate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
cis-1,2-Dichloroethene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
2,2-Dichloropropane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Bromochloromethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Cyclohexane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chloroform	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Ethyl acetate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Carbon tetrachloride	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Tetrahydrofuran	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,1,1-Trichloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
2-Butanol	N/A	N/A	20	14 - 26	N/A	N/A	80	56 - 104	200	140 - 260
2-Butanone	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,1-Dichloropropene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Heptane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Benzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
tert-Amyl methyl ether	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2-Dichloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Isobutyl alcohol	N/A	N/A	20	14 - 26	N/A	N/A	80	56 - 104	200	140 - 260
2-Methyl-2-butanol	2.5	1.75 - 3.25	5	3.5 - 6.5	10	7	20	14 - 26	100	70 - 130
Methyl cyclohexane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26

Soil	Level 0 (ug/kg)	L0 (70% - 130%)	Level 1 (ug/kg)	L1 (70% - 130%)	Level 1.5 (ug/kg)	L1.5 (70% - 130%)	Level 2 (ug/kg)	L2 (70% - 130%)	Level 3 (ug/kg)	L3 (70% - 130%)
Trichloroethene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
n-Butanol	N/A	N/A	20	14 - 26	N/A	N/A	80	56 - 104	200	140 - 260
Dibromomethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2-Dichloropropane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
4-penten-2-ol	2.5	1.75 - 3.25	5	3.5 - 6.5	10	7	20	14 - 26	100	70 - 130
2-Chloroethyl vinyl ether	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Bromodichloromethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Ethyl acrylate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Methyl methacrylate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,4-Dioxane	N/A	N/A	40	28 - 52	80	56 – 104	200	140 - 260	1000	700 - 1300
cis-1,3-Dichloropropene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Octane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Toluene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
4-Methyl-2-pentanone	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Tetrachloroethene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
2-Nitropropane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chloropicrin	N/A	N/A	50	35 - 65	N/A	N/A	100	70 - 130	200	140 - 260
trans-1,3-Dichloropropene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Methyl isothiocyanate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
4-Methyl-2-pentanol	N/A	N/A	20	14 - 26	N/A	N/A	80	56 - 104	200	140 - 260
Ethyl methacrylate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,1,2-Trichloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chlorodibromomethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,3-Dichloropropane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2-Dibromoethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
n-Butyl Acetate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26

Soil	Level 0 (ug/kg)	L0 (70% - 130%)	Level 1 (ug/kg)	L1 (70% - 130%)	Level 1.5 (ug/kg)	L1.5 (70% - 130%)	Level 2 (ug/kg)	L2 (70% - 130%)	Level 3 (ug/kg)	L3 (70% - 130%)
2-Hexanone	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Nonane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Chlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Ethylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,1,1,2-Tetrachloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
p/m Xylene	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	8	5.6 - 10.4	40	28 - 52
o Xylene	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	8	5.6 - 10.4	40	28 - 52
Styrene	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	8	5.6 - 10.4	40	28 - 52
Bromoform	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Butyl acrylate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Isopropylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Bromobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Decane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
n-Propylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,4-Dichlorobutane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,1,2,2-Tetrachloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
4-Ethyltoluene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
2-Chlorotoluene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,3,5-Trimethylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2,3-Trichloropropane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
trans-1,4-Dichloro-2-butene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
4-Chlorotoluene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
tert-Butylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Pentachloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
n-Butyl methacrylate	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2,4-Trimethylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26

Soil	Level 0 (ug/kg)	L0 (70% - 130%)	Level 1 (ug/kg)	L1 (70% - 130%)	Level 1.5 (ug/kg)	L1.5 (70% - 130%)	Level 2 (ug/kg)	L2 (70% - 130%)	Level 3 (ug/kg)	L3 (70% - 130%)
Limonene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
sec-Butylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
p-Isopropyltoluene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,3-Dichlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,4-Dichlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
trans-Decahydronaphthalene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Undecane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
p-Diethylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
n-Butylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Hexachloroethane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2-Dichlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
cis-Decahydronaphthalene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2,4,5-Tetramethylbenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2-Dibromo-3-chloropropane	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,3,5-Trichlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Nitrobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Hexachlorobutadiene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2,4-Trichlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
Naphthalene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,2,3-Trichlorobenzene	0.5	0.35 - 0.65	1	0.7 - 1.3	2	1.4 - 2.6	4	2.8 - 5.2	20	14 - 26
1,3-Dioxolane	N/A	N/A	25	17.5 - 32.5	N/A	N/A	50	35 - 65	100	70 - 130

Volatile Petroleum Hydrocarbons (VPH)

Reference Method No.: Method for the Determination of Volatile Petroleum Hydrocarbons (VPH), Massachusetts DEP, ORS, BWSC. Revision 2.1, February 2018.

Quality Control Requirements and Performance Standards for the ***Analysis of Volatile Petroleum Hydrocarbons (VPH)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP), Section IV C, Rev 0, January, 2017

1. Scope and Application

Matrices: This method is suitable for the analysis of waters, soils, sludges, sediments, wastes and non-aqueous-phase liquids (NAPL). Water samples may be analyzed directly for volatile petroleum hydrocarbons by purge-and-trap concentration and gas chromatography. Soil/sediment samples are dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanol solution is then analyzed by purge-and-trap GC.

This method is designed to measure the collective concentrations of volatile aliphatic and aromatic petroleum hydrocarbons in water and soil. Volatile aliphatic hydrocarbons are collectively quantitated within two ranges: C_5 through C_8, and C_9 through C_{12}. Volatile aromatic hydrocarbons are collectively quantitated within the C_9 to C_{10} range. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling range between approximately 36ºC and 220ºC.

This method is based on a purge-and-trap, gas chromatography (GC) procedure using in-series Photoionization and Flame Ionization Detectors (PID/FID). This method should be used by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatographs. The analysts should be skilled in the interpretation of chromatograms and their use as a quantitative tool. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

This method is also able to measure the individual concentrations of the VPH Target Analytes: benzene, toluene, ethylbenzene, xylenes, naphthalene, and methyl-tert-butyl ether (MTBE). Results for these individual analytes are reported at client request.

Petroleum products suitable for evaluation by this method include gasoline, mineral spirits, and certain petroleum naphthas. This method, in and of itself, is not suitable for the evaluation of kerosene, jet fuel, heating oils, lubricating oils, and/or other petroleum products which contain a significant percentage of hydrocarbons heavier than C_{12}.

The Reporting Limits (RLs) of this method for each of the collective aliphatic and aromatic ranges are typically 5 - 10 mg/kg in soil and 100 - 150 µg/L in aqueous samples. The RLs of this method for the Target VPH Analytes are typically 0.05 to 0.25 mg/kg in soil for all compounds. In aqueous samples, the typical reporting limits are 1 to 5 µg/L for all compounds.

This method includes a data adjustment step to subtract the concentration of VPH Target Analytes from the concentration of the aliphatic range results when the individual compounds are reported (Alpha product VPH Deluxe). This step is taken by the laboratory, and both adjusted and unadjusted values are reported to the client.

Data generated using this method is reported using the format specified in the MADEP VPH method.

Like all GC procedures, this method is subject to a “false positive” bias in the reporting of Target VPH analytes, in that non-targeted hydrocarbon compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantitated as a Target VPH analyte.

Confirmatory analysis by gas chromatography/mass spectrometry (GC/MS) analysis may be performed in cases where target analyte identification is suspect.

Regulatory Parameter List:

Parameter	CAS
C5-C8 Aliphatic Hydrocarbons	-
C9-C12 Aliphatic Hydrocarbons	-
C9-C10 Aromatic Hydrocarbons	-
Benzene	71-43-2
Toluene	108-88-3
Ethylbenzene	100-41-4
m/p-Xylenes	108-38-3/106-42-3
o-Xylene	95-47-6
Naphthalene	91-20-3
Methyl-tert-butyl ether	1634-04-4

Definitions: See Alpha Analytical Quality Manual.

♦ **Analytical Batch** is defined as a group of field samples with similar matrices, which are processed as a unit. For Quality Control purposes, if the number of samples in such a group is greater than 20, then each group of 20 samples or less is defined as a separate analytical batch.

♦ **Calibration Check Standard** is defined as a calibration standard which is analyzed daily to verify the calibration state of an instrument. The calibration check standard is prepared from the same stock solution as the initial calibration standards, and is spiked at the concentration of the Level 4 initial calibration standard.

♦ **Calibration Standards** are defined as a series of standard solutions prepared from a stock standard solution, containing known concentrations of each analyte and surrogate compound of interest.

♦ **Unadjusted C_5 through C_8 Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds, which elute on the FID chromatogram from n-pentane (C_5) to just before n-nonane (C_9).

♦ **Unadjusted C_9 through C_{12} Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds, which elute on the FID chromatogram from n-nonane (C_9) to just before naphthalene.

♦ **C_9 through C_{10} Aromatic Hydrocarbons** are defined as all aromatic hydrocarbon compounds, which elute on the PID chromatogram from just after o-xylene to just before naphthalene. Although it is an aromatic compound with 10 carbon atoms, naphthalene is excluded from this range because it is evaluated as a separate (Target) analyte.

♦ **Field Duplicates** are defined as two separate samples collected at the same time and place, under identical circumstances and managed the same throughout field and laboratory procedures. Analyses of field duplicates give a measure of the precision associated with sample collection, preservation, and storage, as well as laboratory procedures.

- **Target VPH Analytes** are defined as benzene, toluene, ethylbenzene, m-xylene, p-xylene, o-xylene, naphthalene, and methyl tert-butyl ether.

- **Adjusted C_5 through C_8 Aliphatic Hydrocarbons** are defined as all hydrocarbon compounds which elute on the FID chromatogram from n-pentane (C_5) to just before n-nonane (C_9), corrected for MTBE, Benzene, and Toluene.

- **Adjusted C_9 through C_{12} Aliphatic Hydrocarbons** are defined as all hydrocarbon compounds which elute on the FID chromatogram from n-nonane (C_9) to just before naphthalene, corrected for Ethylbenzene, m/p-Xylene, and o-Xylene.

- **Volatile Petroleum Hydrocarbons (VPH)** are defined as collective fractions of hydrocarbon compounds eluting from n-pentane to naphthalene, excluding Target VPH Analytes. VPH is comprised of C5 through C8 Aliphatic Hydrocarbons, C9 through C12 Aliphatic Hydrocarbons, and C9 through C10 Aromatic Hydrocarbons.

- **Volatile Petroleum Hydrocarbon (VPH) Component Standard** is defined as a 15-component mixture of the aliphatic and aromatic compounds listed in Table 2. The compounds comprising the VPH Component Standard are used to (a) define the individual retention times and chromatographic response factors for each of the Target VPH Analytes, (b) define and establish the windows for the collective aliphatic and aromatic hydrocarbon ranges of interest, and (c) determine average chromatographic response factors that can in turn be used to calculate the collective concentration of hydrocarbons within these ranges.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one of the following laboratory personnel before performing the modification: Area Supervisor, Laboratory Services Manager, Laboratory Director, or Quality Assurance Officer.

A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the QA Officer and/or Laboratory Director on a case-by-case basis.

2. Summary of Method

Samples are analyzed using purge-and-trap sample concentration. The gas chromatograph is temperature programmed to facilitate the separation of organic compounds. Detection is achieved by a photoionization detector (PID) and flame ionization detector (FID) connected in series. Quantitation is based on comparing the PID and FID detector response of a sample to a standard comprised of aromatic and aliphatic hydrocarbons. The PID chromatogram is used to determine the individual concentrations of Target Analytes and collective concentration of aromatic hydrocarbons within the C_9 through C_{10} range. The FID chromatogram is used to determine the collective concentration of aliphatic hydrocarbons within the C_5 - C_8 and C_9 - C_{12} ranges.

2.1 Method Modifications from Reference

2.1.1 Trap

We currently use a Supelco K trap. A trap desorption efficiency study was performed using a gasoline standard. Results were acceptable within method required criteria. Results of the comparison study are kept on file by the QA department.

3. Reporting Limits

Table 1 lists our routine reporting limits.

4. Interferences

4.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be free from contamination under the conditions of the analysis. Running laboratory reagent blanks as described in Section 9.1 demonstrates the system is free of contamination. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system must be avoided.

4.2 Sample contamination occurs by diffusion of volatile organics through the septum seal into the sample during shipment and storage. Trip blanks are prepared from reagent water or methanol as appropriate, and are carried through sampling and subsequent storage and handling steps to serve as a check on such contamination.

4.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the sample syringe must be rinsed with reagent water between sample analyses. Each autosampler position is also monitored for positive hits and subsequent sample analyses are checked for potential carry-over. If carry-over is suspected, the sample is reanalyzed for confirmation. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high purgeable levels, it may be necessary to wash the purging device with a detergent solution, rinse it with reagent water, and then dry it in a 105°C oven between analyses. The trap and other parts of the system are subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4.4 The response selectivity of a photoionization detector (PID) is used in this method to differentiate aromatic hydrocarbons from aliphatic hydrocarbons. All compounds eluting on the PID chromatogram after o-xylene are identified by the method as aromatic hydrocarbons. This will lead to an overestimation of aromatic hydrocarbons within samples, as the PID will also detect certain aliphatic compounds, particularly unsaturated compounds such as alkenes. This is a limitation of the method, and the significance and implication of the overestimation will vary from sample to sample.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, standards, or solvents.

5.2 All standard preparation must be performed in the volatiles hood.

5.3 All expired standards must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be labeled properly with hazard warning labels indicating the container contents. Unopened expired standards get placed in the proper VOA lab pack area designated by the Hazardous Waste Manager.

5.4 Bottles containing flammable solvents must be stored in the flammables cabinet.

6. Sample Collection, Preservation, Shipping and Handling

A summary of sample collection, preservation and holding times is provided in Table 3.

6.1 Sample Collection and Preservation

6.1.1 Aqueous Samples

Aqueous samples are collected in triplicate, without agitation and without headspace, in contaminant-free, 40mL glass VOA vials with Teflon-lined septa screw caps. The Teflon liner must contact the sample. Samples are acidified to a pH of 2.0 or less at the time of collection. This is typically accomplished by adding 3 to 4 drops (0.1 to 0.2mL) of 1:1 HCl (1 part reagent water and 1 part concentrated HCl) to a 40mL sample vial. Samples must be cooled to 4ºC immediately after collection.

The laboratory must determine the pH of all water samples. The pH measurement may be performed on leftover sample. If the pH of any sample is above 2, it must be noted on a narrative report for inclusion in the client report.

6.1.2 Soil Samples

Soil samples are collected in duplicate contaminant free 40mL VOC vials with Teflon-lined septa screwcaps. Soil samples are collected in a manner that minimizes sample handling and agitation. The use of specially designed airtight collection samplers is recommended. All sediment must be removed from the glass threads of the vial to ensure an adequate seal. Samples must be cooled to 4ºC immediately after collection.

Methanol preservation of soil samples is mandatory. Methanol (purge-and-trap grade) must be added to the sample vial before or immediately after sample collection. In lieu of the in-field preservation of samples with methanol, soil samples may be collected in specially designed, airtight sampling devices, provided that the samples are extruded and preserved in methanol with 48 hours of collection.

6.1.2.1 The desired ratio of methanol-to-soil is 1mL methanol / 1 gram soil, ± 25%. The exact weight of the soil sample and volume of methanol must be known or ascertained by the laboratory when calculating and reporting soil concentration data.

In all cases, the soil sample in the vial must be completely covered by the methanol.

6.1.2.2 An additional sample of the soil must also be obtained (without methanol) to be used for the determination of soil moisture content to allow for the calculation of the dry weight results, and to calculate the methanol dilution effect. (See Section 11.3)

6.2 Sample Shipping

A reagent water trip blank accompanies each batch of water samples.

A methanol trip blank accompanies each batch of soil samples.

6.3 Sample Handling

A chain of custody form must accompany all sampling vials and must document the date and time of sample collection and, where appropriate, the volume of methanol added. Observations of vial leakage must be so noted on the laboratory/data report sheet. Any sample received by the laboratory that is not packed in ice or cooled to 4ºC must be so noted on the sample delivery group form.

Aqueous samples must be analyzed within 14 days of collection, soil samples within 28 days of collection.

7. Equipment and Supplies

7.1 Glassware

7.1.1 VOC Vials: 40mL VOC vials with Teflon/silicone septa for soils or waters

7.1.2 Class "A" Volumetric Flasks: 10mL, 50mL, 100mL, and 1,000mL with a ground-glass stopper.

7.1.3 Pasteur Pipets: Disposable.

7.2 Analytical Balance: A top-loading balance capable of weighing to the nearest 0.01g is used for weighing soil samples and taring prepared soil VOA vials containing the methanol.

7.3 Gas Chromatograph:

7.3.1 Gas Chromatograph, Hewlett Packard 6890 (or equivalent): An analytical system complete with temperature programmable gas chromatograph and purge-and-trap concentrator. The data station must be capable of storing and reintegrating chromatographic data and must be capable of determining peak areas using a forced baseline projection.

7.3.2 Chromatographic Column: Restek RTx-502.2, 105M x 0.53mm I.D., 3.0-micron film thickness. Alternative columns may be used as long as the method performance criteria can be met.

7.3.3 Detectors: A Photoionization Detector (PID) is used in series with a Flame Ionization Detector (FID); the PID is first in the series.

7.3.4 **Purge and Trap System:** The purge-and-trap system consists of two separate pieces of equipment: a purging device (autosampler) (EST Centurion or equivalent) coupled to the desorber (concentrator) (EST Encon or equivalent).

7.3.5 Purge/carrier gas = Helium, analytical grade, (99.999%) or Nitrogen analytical grade (99.999%).

7.3.6 The purging device is configured with 5 mL sample purge tubes, and the helium purge gas is introduced at the bottom of the water column as finely divided bubbles

7.3.7 The trap used in the desorber is typically a Supelco "K" trap. Different traps may be used if equivalent performance is demonstrated.

7.3.8 The desorber is capable of rapidly heating the trap to 260°C. The trap is not heated above manufacturer's specifications

7.4 Data System: A computer system is interfaced to the GC for collection of data. Chemstation software is used for data acquisition, and Enviroquant software is used for data reduction and processing.

7.5 Syringes: 5mL, 10mL, and 25mL gas-tight syringe.

7.6 Microsyringes: 1µL, 5µL, 10µL, 25µL, 100µL, 250µL, 500µL, and 1,000µL.

7.7 Spatula: Stainless steel.

7.8 Ottawa sand: SiO2.

8. Reagents and Standards

8.1 Organic-free reagent water: Tap water passed through activated carbon. All references to "reagent water" in this SOP refer to water prepared in this manner.

8.2 Methanol: Purge and Trap grade or equivalent, demonstrated to be free of volatile analytes. Store in flammables cabinet, away from other solvents.

8.3 Stock Standards: All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. When an ampulated stock solution is opened, it is transferred to a labeled amber screw cap vial. The expiration date of the stock solution is either the vendor specified expiration date or 1 month from the date the ampule was opened, whichever is sooner. Store at -10°C to -20°C, protected from light.

8.3.1 **Primary VPH Stock Standard:** (Absolute Standards, Cat # 93624) or equivalent

Component	Concentrations, µg/mL
Pentane	50
2-Methyl pentane	50
Methyl-t-butyl-ether	50
2,2,4-Trimethylpentane	50
Benzene	50
Toluene	50

Nonane	50
Decane	50
Ethylbenzene	50
m-Xylene	50
p-Xylene	50
o-Xylene	50
1,2,4-Trimethylbenzene	50
n-Butylcyclohexane	50
Naphthalene	50
2,5-Dibromotoluene (surrogate)	50

8.3.2 Surrogate Stock Standard: 2,5-Dibromotoluene, 10,000µg/mL; Restek Cat # 30453 or equivalent.

8.3.3 Water LCS/LFB Standard: Contains the same compounds as Section 8.3.1 at a concentration of 50µg/mL, Ultra Scientific Cat # SMA-111-1 or equivalent, obtained from a second primary source.

8.3.4 Soil LCS/MS Stock Standard: VPH Matrix Spike Surrogate (revised), 500ug/mL each compound, Absolute Standard Cat# 93625 or equivalent, obtained from a second primary source.

8.3.5 Liquid MS Stock Standard: VPH Matrix Spike Surrogate (revised), 50ug/mL each compound, Restek Cat# 30605 or equivalent, obtained from a second primary source.

8.4 Intermediate Standards: Intermediate standards are prepared volumetrically by diluting the appropriate stock standard(s) with methanol. Intermediate standards expire one month from the date of preparation, or on the earliest expiration date of any of the stock solutions used to prepare the intermediate standard. Store with limited headspace at -10°C to -20°C.

8.4.1 Surrogate standard solutions: Into a 20mL ground glass stoppered volumetric flask, add 1mL of the stock listed in Section 8.3.2. Bring to volume with methanol. The concentration of the intermediate solution is 500µg/mL. The standard is then transferred into labeled 40mL amber VOA screw cap vials and stored in the volatiles freezer at -10° C to -20°C. Record all preparation information, including preparation date, expiration date, preparer's initials, and methanol lot number in the volatiles standard prep logbook.

8.4.2 VPH Water Surrogate: Take 100µl of the intermediate standard prepared in section 8.4.1, add to 900 ul of methanol. The concentration of this solution is 50µl/ml. The standard is stored in a mininert and stored in the volatiles freezer at -10° C to -20°C. Record all preparation information, including preparation date, expiration date, preparer's initials, and methanol lot number in the volatiles standard prep logbook.

8.5 Initial Aqueous Calibration Standards: Initial calibration standards are prepared at the six concentrations listed in Table 2 by adding the amounts of the stock standard (from Section 8.3.1) for levels one and two listed below to a 100mL volumetric flask filled with organic-free water. Mix aqueous standards by inverting the flask three times. Discard the solution contained in the neck of the flask, and fill a 40ml amber sample vial from the standard solution contained in the expanded area of the flask. For levels three to six repeat process by adding the amounts of the stock standard listed into a 50ml volumetric flask. Aqueous standards are not stable and should be discarded after one hour.

Calibration Level	Amount of Primary VPH Stock (µL)
1	2
2	10
3	20
4	50
5	100
6	200

8.6 Initial Soil/Solid Calibration Standards: Initial calibration standards are prepared at the six concentrations listed in Table 2 by adding the amounts of the stock standard (from Section 8.3.1) for levels one and two listed below to a 100mL volumetric flask filled with organic-free water. Mix aqueous standards by inverting the flask three times. Discard the solution contained in the neck of the flask, fill a 5ml syringe with the calibration standard and add to a non-preserved 40ml amber vial. For levels three to six spike listed amounts from the table below into a 5ml syringe filled with organic free water. Add contents of syringe to a non-preserved 40ml amber vial.

Calibration Level	Amount of Primary VPH Stock (µL)
1	2
2	10
3	2
4	5
5	10
6	20

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Analyze a method blank once per analytical batch prior to sample analysis to demonstrate that the system is free of contaminants. The method blank for water samples is 5ml of water with 5µl of VPH Water surrogate (8.4.2) added. The method blank for soil is created by weighing 15g of Ottawa sand into 15ml of methanol, and spiking with 75µl surrogate (8.4.1). A 100µl aliquot of this is taken and added to 4.9ml reagent water to create a method blank for soil samples.

The method blank must not contain any target analytes at or above the compound reporting limits. Samples may not be run until a passing blank is analyzed.

9.2 Laboratory Control Sample (LCS) or Laboratory Fortified Blank (LFB)

An LCS is analyzed with each batch of 20 or less field samples.

The LCS for soil samples consists of weighing 15g of Ottawa sand, adding it to 15ml methanol and spiking with 75µl Soil LCS/MS Stock Standard (8.3.4). A 100µl aliquot of this is taken and added to 4.9ml reagent water.

The LCS for aqueous samples consists of a 5.0mL aliquot of reagent water spiked with 5µL of the LCS stock standard (Section 8.3.3).

The recovery of individual target compounds and hydrocarbon ranges must be between 70% and 130%. If any recovery criteria are not met, the cause of the failure (i.e. degraded standard or instrument problems) must be determined and corrected prior to sample analysis.

- If the recoveries are low and outside of the acceptance limits, reanalyze the LCS and associated samples.
    - If still outside of the acceptance limits recalibrate.
- If the recoveries are high and outside of the acceptance limits and the affected compound was detected in the associated samples, reanalyze the LCS and the associated samples.
    - If recoveries are still outside of the acceptance limits recalibrate.
- If the recoveries are high and sample results were nondetect, data can be reported without qualification; however, the high recoveries should be noted in the laboratory narrative.

9.2.1 Laboratory Fortified Blank Duplicate or Laboratory Control Sample Duplicate (LFBD or LCSD)

A LCSD is analyzed with each batch of 20 or less field samples.

The LCSD for soil samples consists weighing 15g of Ottawa sand, adding it to 15ml methanol and spiking with 75µl Soil LCS/MS Stock Standard (8.3.4). A 100µl aliquot of this is taken and added to 4.9ml reagent water.

The LCSD for aqueous samples consists of a 5.0mL aliquot of reagent water spiked with 5µL of the LCS stock standard (Section 8.3.3).

The recovery of individual target compounds and hydrocarbon ranges must be between 70% and 130%. The RPD for the LCS/LCSD pair must be less than or equal to 25%

9.3 Initial Calibration Verification (ICV)

Refer to Section 10.3.5.

9.4 Continuing Calibration Verification (CCV)

Refer to Section 10.4.

9.5 Matrix Spike (MS) / Matrix Spike Duplicate (MSD)

A Matrix Spike and/or Matrix Spike Duplicate pair is performed at the client's request.

For a liquid sample being purged as a soil/solid, the MS/MSD consists of a 5mL aliquot of a field sample, spiked with 5µL the MS stock standard (Section 8.3.5) or LCS standard (Section 8.3.3).

For a solid sample, the MS/MSD sample vial (a separate VOA sample vial preserved with Methanol) is spiked with 75µL of the MS stock standard (Section 8.3.4) and 100µL aliquot of the Methanol is added to 4.9mL of water and purged.

For a liquid sample the MS/MSD consists of a 43ml aliquot of a field sample, spiked with 43µL the MS stock standard (Section 8.3.5).

The recovery of individual target compounds and hydrocarbon ranges in the MS/MSD, corrected for the concentrations found in the neat sample, must be between 70% and 130% and the %RPD must be <50%. If the recovery criteria are not met but are met in the LCS, the failure may be attributed to sample matrix effects and must be noted on a narrative sheet for inclusion in the client report.

9.6 Laboratory Duplicate

A duplicate sample is analyzed with each batch of 20 or less field samples at the client's request. A duplicate is a split sample, taken from the same sample container and analyzed separately under the same conditions. The %RSDs of the sample and duplicate results are calculated, and must not exceed 50%.

- If RPD exceeds 50% and the results are > 5X the RL, sample analysis must be repeated.
- If an analyte is detected in one of the runs, but not in the other, then analysis must be repeated.
- If an analyte is detected in one analysis at < 5x the RL and not detected in the duplicate analysis, the RPD is not calculable and the analysis does not have to be repeated.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogates

All samples and associated QC samples are spiked with the surrogate spiking solution (Section 8.4.1). The surrogate recoveries must be between 70% and 130%. If any surrogates fail the recovery criteria, the sample should be re-analyzed to determine if the failure is due to sample matrix effects. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report.

Exceptions are as follows:

1. When a large amount of target or non-target material is present that co-elutes with the surrogate compound, resulting in a false high recovery. When this is evident by examination of the chromatogram, re-analysis is not required. Note the cause of the high recovery on a narrative sheet for inclusion in the client report.
2. Percent moisture of a soil/sediment sample is >25% and surrogate recovery is 10% or more.
3. The surrogate recovery is high and the sample is non-detect for all target analytes and hydrocarbon ranges.

9.7.2 Additional Instrumental Quality Control

The n-pentane (C_5) and MTBE peaks must be adequately resolved from any solvent front that may be present on the FID and PID chromatograms, respectively. This is achievable using the recommended trap and purge and trap procedures. Coelution of the m- and p- xylene isomers is permissible.

9.8 Method Sequence

The sequence of analysis is as follows:

- Calibration Standards (initial) or mid-range Calibration Check Standard (daily check of initial calibration) (REQUIRED)
- Laboratory Fortified Blank or Laboratory Control Sample (REQUIRED)
- Laboratory Fortified Blank Duplicate or Laboratory Control Sample Duplicate (REQUIRED)
- Laboratory Method Blank (REQUIRED)
- Samples (up to 20 samples)
- Duplicate sample (As requested by Client)
- Laboratory Fortified Matrix/duplicate (As requested by Client)
- Closing mid-range Calibration Check Standard after 20 samples or at the end of the batch. This standard may also be used as the opening standard for a batch. (REQUIRED)

10. Procedure

10.1 Equipment Set-up

10.1.1 Instrument Set-up Conditions

Typical instrument operating conditions are listed below. Alternate conditions are allowed, as long as method performance criteria can be met.

10.1.1.1 GC Conditions

Injection port temperature:	250ºC
Column head pressure:	20psi
Carrier gas:	Helium
Carrier gas flow:	12.5mL/min.
Temperature 1:	45 °C
Hold time 1:	2 minutes
Ramp 1:	3 ºC / minute
Temperature 2:	100 °C
Hold Time 2:	0 minute
Ramp 2:	8 °C / minute
Temperature 3:	160 °C
Hold Time 3:	0 minutes
Ramp 3	20 ºC/ minute
Temperature 4:	230 ºC
Hold Time 4:	11.6 minutes

10.1.1.2 Detector Conditions

The carrier gas flow is augmented with an additional 24mL of helium flow before entering the photoionization detector. This make-up gas is necessary to ensure optimal response from both detectors.

FID detector

Detector temperature: 250 °C
Air flow: 350 mL/minute
Hydrogen flow: 30 mL/minute

PID detector

Detector temp: 250 °C
PID lamp: 10.2 eV
Makeup gas flow: 17.5 mL/minute

10.1.1.3 Purge and Trap Conditions

Purge Time: 11 minutes
Dry purge: 4 minutes
Desorb Preheat: 225 °C
Desorb temp: 230 °C
Desorb Time: 6 minutes

Bake temp: 260 °C
Bake time: 15 minutes

10.2 Equipment Operation and Sample Processing

The same GC, Purge and Trap, and detector conditions used for the initial calibration must be employed for sample analysis. After verification of system performance by analysis of the continuing calibration standard (Section 10.4), a method blank (Section 9.1) and samples are analyzed and processed as described below.

10.2.1 Retrieve sample VOA vials from the sample bank refrigerator just prior to loading onto the Purge and Trap. Soil samples preserved with methanol and QC samples must have Surrogate Standard Solution (Section 8.4.1) added to the sample and methanol and should be vortexed to extract the volatile components into the methanol and mix the surrogate throughout the sample. Let sample settle prior to taking methanol aliquot. Add the spike volume below;

- 75uL for the 15mL Methanol vial.
- 50uL for the 10mL Methanol vial.
- 25uL for the 5mL Methanol vial.

10.2.2 For soil samples, fill the syringe with 4.9mL of reagent water. Add the appropriate amount of the methanol (typically 100µL) to the syringe containing the reagent water.

10.2.3 Samples may be analyzed continuously for up to 24 hours from the analysis start time of the continuing calibration standard.

10.2.4 Identification of an individual analyte occurs when a peak from a sample falls within the daily retention time window (Section 10.3.6). The concentration of an identified target compound is calculated using the Enviroquant data processing software. The Target

methods have been configured to utilize the quantitation formulae found in Alpha's Quality Manual.

Proper quantitation requires the appropriate selection of a baseline from which the peak area can be determined. For the integration of individual Target Analytes and surrogate compounds, a valley-to-valley approach should typically be used, though this approach may be modified on a case-by-case basis by an experienced analyst. See the Manual Integration SOP for integration guidelines.

If the response for any individual analyte exceeds the calibration range of the system, the sample must be rerun on dilution. Whenever possible, dilutions should be made to keep the concentration of the compound in the middle of the calibration curve.

10.2.5 Aliphatic and aromatic ranges of interest are determined by the collective integration of all peaks that elute between the specified range "marker" compounds (Table 4). When quantifying on a peak area basis, collective peak area integration for the fractional ranges must be from baseline (i.e. must include the unresolved complex mixture "hump" areas). This will often require the analyst to perform a manual integration. See the Manual Integration SOP for integration guidelines.

For non-target analytes eluting in the aliphatic or aromatic fractions, the upper linear range of the system is defined by peak height measurement, based upon the maximum peak height documented for an aliphatic or aromatic standard within the fraction that is shown to be within the linear range of the detector.

10.3 Initial Calibration

10.3.1 Prepare a minimum of 5 calibration standards as per Section 8.5 or 8.6.

10.3.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in Section 10.1.1.1. The same operating conditions are used for calibration and sample analyses. Create the analytical sequence using the Chemstation data acquisition software.

10.3.3 Relative Response Factors

The external standard calibration technique is used.

10.3.3.1 Individual Analytes

In each calibration standard, calculate the calibration factors and the relative standard deviation (RSD) of the calibration factors for all aromatic compounds using the Enviroquant data processing software. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

10.3.3.2 Hydrocarbon Ranges

A collective calibration factor must also be established for each hydrocarbon range. Calculate the collective Calibration Factors (CF) for C_5-C_8 Aliphatic Hydrocarbons and C_9-C_{12} Aliphatic Hydrocarbons using the FID chromatogram. Calculate the collective CF for the C_9-C_{10} Aromatic Hydrocarbons using the PID chromatogram. The VPH marker compounds that determine the various ranges are listed in Table 4. The compounds that are included in each of the 3 ranges are listed below:

C_5-C_8 Aliphatic Hydrocarbons:

n-Pentane, 2-Methylpentane, and 2,2,4-Trimethylpentane

C_9-C_{12} Aliphatic Hydrocarbons:

n-Nonane, n-Decane and n-butylcyclohexane

C_9-C_{10} Aromatic Hydrocarbons:

1,2,4-Trimethylbenzene.

10.3.4 Initial Calibration Criteria

If the RSD for an individual analyte is $\leq$ 20% and $\leq$ 25% for hydrocarbon ranges, then the response for this compound is considered linear over the range and the mean calibration factor can be used to quantitate sample results. If the RSD criterion is not met for an analyte, linearity through the origin cannot be assumed. The calibration may be repeated for any compounds that fail, or a separate calibration curve may be generated for this compound and used for sample quantitation.

In order for the linear regression model to be used for quantitative purposes, r (correlation coefficient) must be ≥0.99. In addition, the resulting calibration curve from the linear regression must be verified by using Percent Relative Error (%RE). For linear fit compounds, the %RE must be calculated for the standard at or near the midpoint and at the lowest level of the initial calibration. The %RE between the calculated and expected concentration of an analyte must be ≤ 30%, otherwise recalibration maybe necessary. If recalculated concentrations from the lowest calibration standard are outside the 70-130% recovery range, raise the RL to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the final calibration curve.

NOTE: Use of non-linear calibration is not allowed and is considered a significant modification.

10.3.5 ICV

After the calibration an ICV needs to be analyzed with a $\pm$30% recovery. The ICV must be prepared from a separate source standard than used for initial and continuing calibrations. The calibration standard must be analyzed immediately following the initial calibration. The ICV should be prepared at a mid-range calibration curve concentration.

Percent recoveries must be between 70-130%. Recalibrate if >10% of all analytes are outside of criteria.

10.3.6 Retention Time Windows

The retention time windows used for identification of the individual analytes are ± 0.05 RRT (Relative Retention Time) units, based on the most recent standard run. It has been determined that these limits work well, being wide enough to eliminate false-negatives while being tight enough to eliminate false-positive results. Analyst experience also weighs heavily in the determination of positive hits. If uncertainty still exists, confirmation by GC/MS is required.

VPH retention time (Rt) windows of the aliphatic ranges are defined as beginning 0.1 minutes before the Rt of the beginning marker compound and ending 0.1 before the Rt of the ending marker compound, except for C_9, which is both a beginning and ending marker compound for two different ranges.

The C_5 – C_8 Aliphatic Hydrocarbon range ends immediately (0.01 min) before the elution of the n-nonane peak.

The C_9 – C_{12} Aliphatic Hydrocarbon range begins 0.01 min. before the elution of n-nonane.

Therefore, there is no overlap of the two ranges and the n-nonane peak is only included in the C_9 – C_{12} Aliphatic Hydrocarbon range.

The VPH retention time (Rt) window for the C_9-C_{10} Aromatic Hydrocarbons is defined as beginning 0.1 minutes after the Rt of the beginning marker compound and ending 0.1 before the Rt of the ending marker compound.

VPH marker compounds and windows are summarized in Table 4.

10.4 Continuing Calibration

Verify calibration daily, after every 20 samples or less, and at the end of the analytical sequence by analyzing a continuing calibration standard. The concentration of the continuing calibration standard is the same as the Level 4 standard from the initial calibration (Section 8.5 or 8.6). The %D of each response factor must be ≤20% for target VPH analytes and the surrogate, and ≤25% for hydrocarbon ranges except for n-nonane, which must be ≤30%.

10.4.1 If these criteria are not met, inspect the analytical system to determine the cause of the drift and perform necessary maintenance before verifying calibration and proceeding with sample analyses.

10.4.2 If routine maintenance does not return the instrument performance to meet the %D criteria when compared to the last initial calibration curve, then a new initial calibration must be performed.

10.4.3 Closing Mid-range Calibration Verification

Following a batch of twenty or less samples, another CCV (Section 10.4) is analyzed. If this CCV achieves the acceptance criteria specified in Section 10.4, the sample results are considered valid. If this standard fails acceptance criteria, the samples and QC samples analyzed since the last acceptable CCV must be reanalyzed.

This QC sample may also be used as the opening CCV Standard for the next analytical batch if batches are processed continuously.

10.5 Preventive Maintenance

If the response of the PID detector decreases significantly, perform a cleaning of the PID lamp per the manufacturer's instructions. If the response is still low, the PID lamp should be replaced with a new one (PID lamp life is typically 90 - 180 days).

Additional maintenance should be performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and clipping several inches off of the injector end of the capillary column. The trap used in the Purge and Trap equipment will also need periodic replacement, when system performance degrades.

11. Data Evaluation, Calculations and Reporting

11.1 Data Manipulations

By definition, the collective concentrations of aliphatic and aromatic fractions of interest **exclude** the individual concentrations of VPH Target Analytes. Accordingly, a series of

data manipulation steps are necessary to adjust the collective range concentrations to eliminate "double counting" of analytes.

This necessary data manipulation step is taken by Alpha for inclusion on the client report. Subtract the individual concentrations of the Target analytes from the collective concentration of Unadjusted Aliphatic Hydrocarbon ranges. This calculation is performed automatically by Alpha's LIMS system according to the steps listed below.

11.1.1 Subtract the collective concentration of $C_9 – C_{10}$ Aromatic Hydrocarbons from the collective concentration of $C_9 – C_{12}$ Aliphatic Hydrocarbons. If this value is less than the Reporting Limit, report "Not Detected".

11.1.2 Subtract the individual concentrations of the VPH Target Analytes from the appropriate aliphatic range (i.e. $C_5 – C_8$, or $C_9 – C_{12}$ Aliphatic Hydrocarbons) in which they elute. If the individual concentrations of Target Analytes have not been quantitated, report the values as Unadjusted $C_5 – C_8$ Aliphatic Hydrocarbons and Unadjusted $C_9 – C_{12}$ Aliphatic Hydrocarbons, and indicate "Not Determined" for Adjusted $C_5 – C_8$ Aliphatic Hydrocarbons and Adjusted C9 – C_{12} Aliphatic Hydrocarbons.

The extent of data manipulations taken by the laboratory must be noted on the data reporting form.

11.2 Data Reporting Format

The purpose Alpha's reporting format is to provide data users with a succinct and complete summary of pertinent information and data, a clear indication of whether significant modifications were made to the VPH Method, and a clear affirmation on whether the QA/QC procedures and standards specified in this method were followed and achieved. While it is permissible to alter the form and presentation of the data, all of the information must be provided in a clear, concise, and succinct manner.

11.3 LIMS Data Corrections

Please note that the Laboratory Information Management System (LIMS) automatically adjusts soil sample results to account for the % Total Solids of the sample (as determined per Alpha SOP/07-38) and the methanol preservation dilution effect.

12. Contingencies for Handling Out-of-Control or Unacceptable Data

Holding time exceedance, improper preservation and observed sample headspace are noted on a project narrative report by the analyst for inclusion in the data report.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument maintenance logbook.

All batch and sample specific QC criteria outlined in Section 10 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

The laboratory follows the procedure found in the MADEP "Method for the Determination of Volatile Petroleum Hydrocarbons (VPH), Revision 2.1, February 2018" to determine the MDL on an annual basis. The method detection limits determined by the laboratory are on file for review.

Method performance data is on file in the laboratory QC department. Method performance data for the laboratory is compared to the MADEP VPH method criteria.

13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD)

The laboratory follows the procedure to determine the MDL, and LOD as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDOC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

13.3 Initial Demonstration of Accuracy (IDA)

Prepare and analyze seven (7) replicate Laboratory Control Samples (LCSs) fortified at a concentration of 50% of the highest calibration curve standard (100µg/L for waters and 5mg/Kg for soils/sediments). An LCS must be prepared and treated exactly as a typical field sample submitted for analysis, including exposure to all glassware, equipment, solvents and reagents. An LCS for water analyses is prepared by adding 5µL of LCS spiking solution (Section 8.3.3) in purge-and-trap grade Methanol to 5mL of organic-free water (ASTM Type I reagent grade). An LCS for soil/sediment analyses is prepared by adding 5µL of LCS spiking solution (Section 8.3.3) in purge-and-trap grade Methanol to 5 mL of organic-free water (ASTM Type I reagent grade) to obtain an equivalent concentration of 50% of the highest calibration curve standard (5mg/Kg). In addition a matrix specific method blank must be prepared and analyzed. Calculate the mean measured concentration (C_{mean}) of the replicate LCSs for VPH Target Analytes and ranges as follows:

Equation D: Calculation of C_{mean}

$$C_{mean} = \frac{(C_1 + C_2 + C_3 + ...C_n)}{n}$$

Where:

C_{mean} = Mean recovered concentration of the replicate analysis

$C_1 + C_2 + C_3 + ...C_n$ = Recovered concentrations of the replicate 1.2...n

$n = 7$

The value derived for C_{mean} must be within ± 30% of the true value or between 70µg/L and 130µg/L for waters and 3.5mg/Kg and 6.5mg/Kg for solids.

13.4 Initial Demonstration of Precision (IDP)

Using the results calculated from Section 13.3, calculate the percent relative standard deviation (%RSD) of the seven (7) replicate analyses, as indicated in the Equation E below. The %RSD must be less than or equal to 25% for both waters and solids. Higher %RSDs are allowed for n-Nonane, if included in the collective calibration standard for the C_9 through C_{12} aliphatic range. Such allowable non-conformances must be documented.

Equation E: Calculation of %RSD

$$\%RSD = \frac{S_{n-1}}{C_{mean}} \times 100$$

Where:

S_{n-1} = Sample standard deviation (n-1) of the replicate analyses

C_{mean} = mean recovered concentration of the replicate analyses

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

Chemical Hygiene Plan

SOP/08-05 MDL/LOD/LOQ Generation

SOP/08-12 IDC/DOC Generation

SOP/14-01 Waste Management and Disposal SOP

16. Attachments

TABLE 1: Reporting Limits

TABLE 2: List of Compounds and Calibration Levels

TABLE 3: Holding Times and Preservation Requirements

TABLE 4: VPH Marker Compounds

TABLE 1
REPORTING LIMITS

Parameter	Aqueous	Soil
C5-C8 Aliphatic Hydrocarbons	100.00 µg/L	5.0 mg/kg
C9-C12 Aliphatic Hydrocarbons	100.00 µg/L	5.0 mg/kg
C9-C10 Aromatic Hydrocarbons	100.00 µg/L	5.0 mg/kg
Benzene	2.00 µg/L	0.10 mg/Kg
Toluene	2.00 µg/L	0.10 mg/Kg
Ethylbenzene	2.00 µg/L	0.10 mg/Kg
m/p-Xylenes	2.00 µg/L	0.10 mg/Kg
o-Xylene	2.00 µg/L	0.10 mg/Kg
Naphthalene	4.00 µg/L	0.20 mg/Kg
Methyl-tert-butyl ether	3.00 µg/L	0.05 mg/Kg

TABLE 2
LIST OF COMPOUNDS AND CALIBRATION LEVELS

Component	Concentration (µg/L)					
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Pentane	1	5	20	50	100	200
2-Methylpentane	1	5	20	50	100	200
Methyl-t-butyl ether	1	5	20	50	100	200
2,2,4-Trimethylpentane	1	5	20	50	100	200
Benzene	1	5	20	50	100	200
Toluene	1	5	20	50	100	200
Nonane	1	5	20	50	100	200
Decane	1	5	20	50	100	200
Ethylbenzene	1	5	20	50	100	200
p/m-Xylene	2	10	40	100	200	400
o-Xylene	1	5	20	50	100	200
1,2,4-Trimethylbenzene	1	5	20	50	100	200
n-Butylcyclohexane	1	5	20	50	100	200
Naphthalene	1	5	20	50	100	200
2,5-Dibromotoluene (surrogate)	1	5	20	50	100	200

TABLE 3
HOLDING TIMES AND PRESERVATION REQUIREMENTS

Matrix	Container	Preservation	Holding Time
Aqueous Samples	40mL VOC vials w/Teflon-lined septa screw caps, in triplicate	Add 3 to 4 drops of 1:1 HCl; cool to 4ºC	14 days
Soil/Sediment Samples	VOC vials w/Teflon- lined septa screw caps, in duplicate 40mL vials: add 15 g soil	1mL methanol for every gram of soil; add before or at time of sampling; cool to 4ºC	28 days

TABLE 4
VPH MARKER COMPOUNDS

Hydrocarbon Range	Beginning Marker	Ending Marker
C_5-C_8 Aliphatic Hydrocarbons (FID)	0.1 min. before n-Pentane	0.01 min. before n-Nonane
C_9-C_{12} Aliphatic Hydrocarbons (FID)	0.01 min. before n-Nonane	0.1 min. before Naphthalene *
C_9-C_{10} Aromatic Hydrocarbons (PID)	0.1 min. after o-Xylene	0.1 min. before Naphthalene

* The retention time for Dodecane (C_{12}) is approximately 2 minutes less than the retention time for naphthalene, using the column and chromatographic conditions recommended for this method. For simplicity, naphthalene is used as the ending marker for the C_9-C_{12} Aliphatic Hydrocarbon range.

ENV-SOP-WEST-0040 Extractable Petroleum Hydrocarbons (MA-EPH) 2.1

Reference: Method for the Determination of Extractable Petroleum Hydrocarbons (EPH), Massachusetts DEP, BWSC. MADEP-EPH-19-1, Revision 2.1 December 2019.

Quality Control Requirements and Performance Standards for the ***Analysis of Extractable Petroleum Hydrocarbons (EPH)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP), Section IV B, Rev 1, July 1, 2010

1. Scope and Application

Matrices: This method is suitable for the analysis of waters, soils, sediments, wastes, sludges and non-aqueous phase liquids (NAPL).

Definitions: Refer to Alpha Analytical Quality Manual.

This method is used to measure the collective concentrations of extractable aliphatic and aromatic petroleum hydrocarbons in water and soil. Extractable aliphatic hydrocarbons are collectively quantitated within two ranges: C_9 through C_{18}, and C_{19} through C_{36}. Extractable aromatic hydrocarbons are collectively quantitated within the C_{11} – C_{22} range. These extractable aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 150 °C and 265 °C.

This method is based on a solvent extraction, silica gel solid-phase extraction/fractionation process (SPE), and gas chromatography (GC) analysis using a flame ionization detector (FID). This procedure is used by, or under the supervision of, analysts experienced in extractable organics analysis. Analysts must be skilled in the interpretation of gas chromatograms and their use as a quantitative tool. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

This method is also able to measure the individual concentrations of Target Polynuclear Aromatic Hydrocarbons (PAH) Analytes, including Diesel PAH Analytes, in water and soil. Results for these individual analytes are reported at client request. The Method Detection Limits for some of these PAH compounds in water are greater than the notification and/or cleanup standards specified in the Massachusetts Contingency Plan for sites located in drinking water resource areas. In cases where it is necessary to demonstrate compliance with these standards, the use of a GC/MS method in the SIM mode may be necessary.

The fractionation step described in this method may be eliminated to allow for the determination of a Total Petroleum Hydrocarbon (TPH) value, and/or obtain qualitative “fingerprinting” information. While TPH provides little information on the chemistry, toxicity, or environmental fate of petroleum mixtures, it may be a cost-effective screening tool in cases where relatively low concentrations of contamination are suspected.

Petroleum products suitable for evaluation by this method include kerosene, fuel oil #2, fuel oil #4, fuel oil #6, diesel fuel, jet fuel, and certain lubricating oils. This method, in and of itself, is not suitable for the evaluation of gasoline, mineral spirits, petroleum naphthas, or other petroleum products that contain a significant percentage of hydrocarbons lighter than C_9. This method, in and of itself, is also not suitable for the evaluation of petroleum products that contain a significant percentage of hydrocarbons heavier than C_{36}.

This method includes a data adjustment step to subtract the concentration of Target PAH Analytes from the concentration of C_{11} through C_{22} Aromatic Hydrocarbons. This step is taken by the laboratory, and both adjusted and unadjusted values are reported to the client.

Data generated using this method is reported using the required format.

Like all GC procedures, this method is subject to a "false positive" bias in the reporting of Target PAH Analytes, in that non-targeted hydrocarbon compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantified as a Target or Diesel PAH Analyte. In addition, this method is subject to a "false negative" bias in the reporting of Target PAH Analytes, in that the ability to identify Target PAH Analytes at low concentrations may be inhibited if a large unresolved complex mixture is present. While cleanup procedures specified in this method to segregate aliphatic and aromatic fractions will serve to mitigate this concern, confirmatory analysis by gas chromatography/mass spectrometry (GC/MS) analysis may be performed in cases where a target PAH analyte identification is suspect.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/FID and in the interpretation of GC/FID data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability.

Parameter	CAS
C9-C18 Aliphatic Hydrocarbons	-
C19-C36 Aliphatic Hydrocarbons	-
C11-C22 Aromatic Hydrocarbons	-
Naphthalene	91-20-3
2-Methylnaphthalene	91-57-6
Acenaphthylene	208-96-8
Acenaphthene	83-32-9
Fluorene	86-73-7
Phenanthrene	85-01-8
Anthracene	120-12-7
Fluoranthene	206-44-0
Pyrene	129-00-0
Benzo(a)anthracene	56-55-3
Chrysene	218-01-9
Benzo(b)fluoranthene	205-99-2
Benzo(k)fluoranthene	207-08-9
Benzo(a)pyrene	50-32-8
Indeno(1,2,3-cd)pyrene	193-39-5
Dibenzo(a,h)Anthracene	53-70-3
Benzo(g,h,i)perylene	191-24-2

2. Summary of Method

Samples submitted for EPH analysis are extracted with methylene chloride, dried over sodium sulfate, solvent exchanged into hexane, and concentrated in a Kuderna-Danish apparatus. Sample cleanup and separation into aliphatic and aromatic fractions is conducted using commercially available silica gel cartridges. The two extracts produced are then re-concentrated to final volumes of 1mL each (i.e., an aliphatic extract and an aromatic extract). The extracts are then separately analyzed by a capillary column gas chromatograph equipped with a flame ionization detector.

Average calibration factors are determined using an aliphatic hydrocarbon standard mixture and are used to calculate the collective concentrations of C_9 through C_{18} and C_{19} through C_{36} aliphatic hydrocarbons. An average calibration factor is determined using a PAH standard mixture and is used to calculate the collective C_{11} through C_{22} aromatic hydrocarbon concentration. Calibration factors determined for individual components of the PAH standard mixture are also used to calculate individual concentrations of Target PAH Analytes

2.1 Method Modifications from Reference

None.

3. Reporting Limits

The Reporting Limits (RLs) of this method for each of the collective aliphatic and aromatic fractional ranges are typically 6.67 mg/kg in soil and 100µg/L in water. The RLs of this method for the Target PAH Analytes is typically 0.33 mg/kg in soil and 10µg/L in water. Table 1 lists our routine reporting limits.

4. Interferences

4.1 High purity reagents are used to minimize interference problems.

4.2 Only high purity gases are used in the GC system to eliminate this source of possible contamination. The air and hydrogen (carrier gas – 99.999%, and detector make-up gas) are certified by the gas supplier.

4.3 Preventive instrument maintenance is performed routinely, and whenever highly contaminated extracts are analyzed that could result in chromatographic interferences or result in degradation of system performance. Section 10.5 details the maintenance steps.

4.4 Glassware must be scrupulously cleaned. This procedure is detailed in the extraction SOPs. Store dry glassware in a clean environment.

4.5 The leaching of plasticizers and other compounds have been observed from commercially available silica gel cartridges used to fractionate EPH sample extracts. This possible source of contamination is continuously monitored and documented by analysis of Laboratory Method Blanks.

4.6 Contamination by carry-over can occur whenever high-concentration and low-concentration samples are sequentially analyzed. Whenever an unusually concentrated sample is encountered, it must be followed by the analysis of a solvent blank to check for possible carry-over. If this is not done and the sample analyzed immediately following the high-concentration sample is free from contamination, then it may be assumed that carry-over is not an issue. However, if this sample did detect analytes which were present in the unusually concentrated sample, reanalysis is required for all samples analyzed after this highly concentrated sample which detected similar analytes.

4.7 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interference will vary considerably from one source to another depending upon the nature and diversity of the site being sampled. The silica gel SPE cleanup procedure is used to overcome many of these interferences, but some samples may require additional cleanup approaches that are beyond the scope of this method.

4.8 Certain organic contaminants mixed with petroleum product releases, including chlorinated hydrocarbons, phenols, and phthalate esters, will be quantitated as Extractable and Total Petroleum Hydrocarbons. If necessary and/or desirable, additional sample cleanup and/or analytical procedures may be employed to minimize or document the presence of such compounds.

4.9 Because of their weak polar nature, naphthalene and substituted naphthalene's are readily fractionated into the aliphatic extract if excessive amounts of hexane are used to elute the silica gel cartridge/column. Because these compounds constitute a significant percentage of the water-soluble fraction of fuel oils, this occurrence is especially problematic in the analysis of water samples. For this reason, the method requires the evaluation of the aliphatic fraction for the presence of naphthalene and 2-methylnaphthalene in the LCS/LCSD pair on a per batch basis. The fractionation surrogate, 2-Bromonaphthalene, is used to monitor sample-specific fractionation efficiency.

5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel

involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

5.1 Lab coats, safety glasses, and gloves must be worn when handling samples, extracts, standards, or solvents.

5.2 All solvent and extract transfers must be handled in the vented bench area in the GC laboratory.

5.3 All stock standards, working standards, and vialed sample extracts must be placed into the waste bucket in the lab, for future disposal by the Hazardous Waste Manager. The container must be labeled properly with hazard warning labels indicating the container contents.

5.4 Bottles containing flammable solvents must be stored in the flammable's cabinet.

6. Sample Collection, Preservation, Shipping and Handling

6.1 Sample Collection

Aqueous samples are collected in two 1L amber glass jars with teflon-lined lids. Solid samples are collected in 250mL wide-mouth amber glass jars with teflon-lined lids. All containers are purchased pre-cleaned and certified from commercial vendors.

6.2 Sample Preservation

Both aqueous and solid samples are then preserved by packing in coolers with ice or ice packs, to maintain a temperature of 4 ±2 ° C. Additionally, aqueous samples are preserved at the time of sampling by the addition of approximately 5mLs of 1:1 HCl. The sample pH must be less than 2.0 after addition of the acid. Upon receipt at the laboratory, the samples are transferred into sample storage refrigerators to maintain at a temperature of 4 ±2 ° C.

6.3 Sample Shipping

No specific requirements.

6.4 Sample Handling

Aqueous and soil/sediment samples must be extracted within 14 days of sample collection and be analyzed within 40 days of the extraction date. Additionally, if the sample must be frozen it needs to be within 24hours after collection and then it can be extracted up to 14 days after thawing.

7. Equipment and Supplies

7.1 **Gas Chromatograph, Agilent 6890A and 7890A:** An analytical system complete with gas chromatograph configured for split-splitless injection and all required accessories including autosampler syringes, analytical columns, gases, and flame ionization detectors (FID). The systems used for this analysis are configured with dual injectors/dual columns for

7.2 **GC Column:** is listed below. Alternative columns may be used as long as the method performance criteria can be met.

RTx-5MS: Cat. #12624 from Restek or equivalent; 30m, 0.32mm, 0.25µm

7.3 **Microsyringes:** 10µL, 100µL, 250µL, 500µL and 1000µL

7.4 **Volumetric Flasks:** 10mL and 25mL, used for standard preparation.

7.5 Precision liner: Cat. #AGO-4680 from Phenomenex or equivalent

7.6 Siltek FID jet: Cat. #21683, #20673 from Restek or equivalent

8. Reagents and Standards

Reagent grade or pesticide grade chemicals are used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficient high purity to permit its use without lessening the accuracy of the determination.

NOTE: Store the standard solutions (stock, composite, calibration, internal, and surrogate) at 4 ±2 ºC in Teflon(R)-sealed containers in the dark.

8.1 Reagents

8.1.1 Reagent Water: All reagent water is from Alpha's DI treatment system and is demonstrated to be free of interferences or contaminants.

8.1.2 Solvents: Hexane, Methylene Chloride (DCM), and Acetone; pesticide grade or better. Store away from other solvents.

8.2 Stock Standard Solutions: All stock standard solutions are purchased from commercial vendors as ampulated certified solutions. They can be replaced with different standards as long as they contain all target analytes. Stock solutions are stored per manufacturer's recommendations.

8.3 Intermediate/Working Standard Solutions

Intermediate and working standards expire 6 months from the date of preparation or on the earliest expiration date of any of the stock solutions used to prepare it. Standards are stored in the refrigerator. Standard preparation information including the analyst's initials, preparation date, expiration date and standard identifier are recorded.

8.3.1 Calibration Standards

The Aliphatic calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane. The Aromatic calibration standards are prepared volumetrically by diluting the appropriate stock standard(s) with methylene chloride. Calibrations are performed at the 6 concentration levels listed in Table 2. The list of typical ampulated calibration standards:

- **ALIPHATIC WORKING STANDARD at 200µg/mL:**
  - 1-Chlorooctadecane Solution, Ultra, #IST-470-1 at 2000µg/mL
  - Fractionation Surrogate Spike Mix, Accustandard, #DRH-MA-FSS-50X at 2000µg/mL
  - Naphthalene, Accustandard, #M-502-40-10X at 2000µg/mL
  - 2-Methylnaphthalene, Accustandard, #APP-9-133-20X at 2000µg/mL
  - MA-EPH n-Hydrocarbon Standard, Absolute, #93459 at 2000µg/mL
- **AROMATIC WORKING STANDARD at 200µg/mL:**
  - Fractionation Surrogate Spike Mix, Accustandard, #DRH-MA-FSS-50X at 2000µg/mL

- O-Terphenyl Standard, Accustandard, #DRH-006-SS at 1000µg/mL
- Aromatic Hydrocarbon Standard, Ultra, #SMA-300-1 at 1000µg/mL

8.3.2 Second Source (ICV):

The Aliphatic and Aromatic ICV standards are prepared volumetrically by diluting the appropriate stock standard(s) with hexane and ethylene chloride accordingly. The list of typical ampulated calibration standards:

- **ALIPHATIC ICV STANDARDS at 10ug/mL**:
  - Proposed DEP(MA)-Aliphatic Hydrocarbons, Accustandard, #DRH-007S at 1000µg/mL
  - Naphthalene, Absolute, #90298 at 2000µg/mL
  - 2-Methylnaphthalene, Absolute, #90798 at 2000µg/mL
  - 1-Chlorooctadecane, Restek, #31098 at 10000µg/mL
  - MA-Fractionation Surrogate Spike Mix, Restek, #31480 at 4000µg/ml
- **AROMATIC ICV STANDARDS at 10ug/mL:**
  - Proposed DEP(MA)-PAH Mix, Accustandard, #DRH-006S at 1000ug/mL
  - MA Fractionation Surrogate Spike Mix, Restek, #31480 at 4000µg/mL
  - O-Terphenyl, Restek, #31097 at 10000µg/mL

8.3.3 Surrogate Spiking Solution

The extraction surrogates are 1-chloro-octadecane (COD) and Ortho-Terphenyl (OTP).The surrogate spiking solution is prepared by adding 10mL of MA-EPH Surrogate Spike (Absolute, Cat. #51075 at 2000µg/mL) to a 500mL volumetric flask and bringing to volume with acetone. The final concentration of the spiking solution is **40µg/mL**.

8.3.4 LCS/MS Spiking Solution

This solution is used for preparing Laboratory Control Samples (LCS) and Matrix Spikes (MS), if requested. The solution is prepared by adding 10mL of the MA-EPH Aromatic Hydrocarbons (Absolute, Cat. #50003, at 2000µg/mL, consists of the 17 PAH compounds listed in Table 1) and 10mL of the MA-EPH n-Hydrocarbon Standard (Absolute, Cat. #93459 at 2000µg/mL, consists of the 14 normal alkanes listed in Table 2) to a 200mL volumetric flask and bringing to volume with pentane. The final concentration of the spiking solution is **100µg/mL**. This solution must always be brought to room temperature prior to use.

8.3.5 Fractionation Surrogate Spiking Solution

The fractionation surrogates are 2-Fluorobiphenyl and 2-Bromonaphthalene. This solution is added to all extracted samples and QC samples prior to sample cleanup and fractionation using the silica gel cartridges. This serves as an important check on the ability of the fractionation to accurately separate the aliphatic from the aromatic compounds. The solution is prepared by adding 10mL of the MA Fractionation Surrogate Spike Mix (Restek, Cat. #31480 at 4000µg/mL) and volumetric dilution to 1000mL with hexane. The final concentration of the surrogate solution is **40µg/mL**.

8.3.6 Fractionation Check Solution

This solution is used to monitor the fractionation efficiency of the silica gel cartridge, and to establish the optimum volume of hexane needed to sufficiently elute aliphatic

hydrocarbons from the cartridge without eluting the aromatic hydrocarbons prior to the methylene chloride elution. Fractionation checks are performed whenever a new lot of silica gel cartridges is received, prior to using them for actual samples.

The solution is prepared by adding 1mL of the Aromatic Hydrocarbon Standard (Ultra, Cat. #SMA-300-1 at 1000µg/mL), 1mL of the Aliphatic Hydrocarbon Standard (Ultra, Cat. #SMA-310-1 at 1000µg/mL) and 1mL of Surrogate Spiking Solution (Ultra, Cat. #ISM-580-1 at 2000µg/mL) to a 50mL volumetric flask, bringing to volume with hexane. The final concentration of the solution is **40µg/mL** for Surrogates and **20µg/mL** for aliphatic and aromatic compounds.

8.4 Hydrogen gas: From Alpha generator.

8.5 Zero Air: From Alpha generator

9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

9.1 Blank(s)

Extraction blanks are performed with each extraction batch of 20 or less samples, according to the extraction SOPs.

The extraction blank must not contain any of the individual target analytes or hydrocarbon ranges above the reporting limits. If any reportable analytes are detected in the blank, the entire extraction batch is suspect.

- If concentration of contaminant in sample is <10x concentration in blank all samples associated with this blank must be re-extracted and re-analyzed. If re-extraction cannot be performed due to insufficient sample volume CS must be notified immediately.
- No corrective action is required if concentration of contaminant in sample is >10x concentration in blank or if contaminant is not detected in sample.

The surrogate recoveries must also be within the acceptance criteria listed in Section 9.7.1. If surrogate acceptance criteria are exceeded, the extraction batch must be evaluated to determine if re-extraction or re-analysis is necessary.

- If any of the surrogates in the blank are high, but blank is undetected for all target analytes, no corrective action is required.
- If any of the surrogates in the blank are low, but associated samples are undetected for all target analytes with passing surrogates, no corrective action is required.

If any of the surrogates in the blank are low and associated samples have positive hits, re-fractionation and/or re-extraction of the appropriate extracts is required.

9.2 Laboratory Control Sample (LCS)

An LCS/LCSD pair is extracted with each analytical batch of 20 samples or less. The LCS/LCSD consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS/LCSD standards must be from a different source than that used for the initial calibration. The LCS/LCSD pair is spiked with aliphatic and aromatic compounds.

The recovery of **alkanes, PAHs and all 3 ranges** must be between **40%-140%** except for **nonane** which must be between **30-140%**.

- If >10% of compounds are above the acceptance criteria (>140%), re-extraction is not required if affected compounds were not detected in associated samples.
- If any recovery criteria are not met, the extract may be re-fractionated to see if the recoveries improve. If the criteria are still not met, the entire batch must be re-extracted. If this is not possible, due to insufficient sample volume or holding time exceedances, the analyst must submit the failure on a narrative sheet for inclusion in the client report.
- The Analytical Batch Precision is determined from the Relative Percent Difference (RPD) of the concentrations (not recoveries) of LCS/LCSD pair. The **RPD** for alkanes and individual Target PAH analytes, aliphatic and aromatic hydrocarbon range concentrations must be **≤ 25 for waters and soils**.

Breakthrough of naphthalene and 2-methylnaphthalene in the aliphatic fraction is calculated. If the breakthrough is **>5%**, all associated samples and QC samples must be re-fractionated.

9.3 Initial Calibration Verification (ICV)

See Section 10.2

9.4 Continuing Calibration Verification (CCV)

See Section 10.4

9.5 Matrix Spike

A Matrix Spike and/or a Matrix Spike/Matrix Spike Duplicate pair are performed at client's request. The MS/MSD is an aliquot of actual sample spiked with the aliphatic and aromatic compounds. The recoveries in the **MS/MSD**, corrected for the concentrations found in the parent sample, must be between **40% and 140%.** If the recovery criteria are not met, but are met in the LCS, the failure may be attributed to sample matrix effects and must be noted on a narrative sheet for inclusion in the client report. If an MS/MSD is performed, the **%RPD** must be **less than 50%** for alkanes and PAHs for results >5x the reporting limit.

9.6 Laboratory Duplicate

Upon Client request, a laboratory duplicate is extracted with each analytical batch of 20 samples or less. The duplicate is a sample aliquot split from the same container as the sample and is extracted and analyzed separately following the same procedures. The RPDs for any hits in the samples, for hydrocarbon ranges or individual analytes (if reported) must be less than 50% when the results are greater than 5x the Reporting Limit. If the RPD criteria are not met, and all other QC criteria are met for the sample and it's duplicate, inspect the sample for possible non-homogeneity. If this is the suspected cause of the high RPD result(s) note this on a narrative sheet for inclusion in the client report. Otherwise, re-extraction of the sample and duplicate, and possibly the entire extraction batch, may be required. If RPD is greater than 50 and both results are >5x the RL you must repeat the analysis. If a Target PAH or Range is detected in one analysis at >5x the RL and not in the duplicate analysis, you must repeat the analysis.

9.7 Method-specific Quality Control Samples

9.7.1 Surrogate

All extracted samples and associated QC are spiked with two extraction surrogates **COD** and **OTP**. Sample extracts are also spiked with the fractionation surrogates **2-bromonaphthalene and 2-fluorobiphenyl** prior to cleanup and fractionation

using the silica gel cartridges. All surrogate recoveries must be between **40% and 140%**. If any surrogates fail the recovery criteria, samples may be re-fractionated and re-analyzed. If the surrogate limits are still not met, the affected samples must be re-extracted to confirm that the failure was due to sample matrix. If matrix effect is confirmed, this must be noted on a narrative sheet for inclusion in the client report; both sets of data are reported.

The only exception to this is when large amounts of extracted material co-elute with any of the surrogate compounds, resulting in false high recoveries. When this is evident by examination of the sample chromatogram, re-extraction or re-fractionation is not required. Note the cause for the high recovery on a narrative sheet for inclusion in the client report.

9.8 Method Sequence

The typical daily analytical sequence is:

- Calibration Check Standard
- Laboratory Method Blank
- Laboratory Control Sample
- Laboratory Control Sample Duplicate
- 20 or less samples
- Duplicate sample (as requested)
- Laboratory Fortified Matrix/duplicate (as requested)
- Calibration Check Standard

10. Procedure

10.1 Equipment Set-up

10.1.1 Sample Extraction

Aqueous samples are extracted at acid pH (less than pH 2) with methylene chloride using a separatory funnel (Method 3510C). See extraction SOP for details.

Solid samples are extracted with methylene chloride using Soxhlet extraction (Method 3540C) or microwave extraction (Method 3546) procedures. See appropriate extraction SOP for details.

10.1.2 Extract Fractionation

Silica gel fractionation is used for sample cleanup and separation into aliphatic and aromatic extracts. Silica gel cartridges are purchased from commercial vendors and tested prior to use. See appropriate extraction SOP for details.

10.1.3 GC Conditions:

A dual-injector/dual-column/dual-detector approach involves the use of the columns listed in section 7.2. This allows for the simultaneous analysis of both fractions. Typical GC conditions are listed below but may be altered as long as method performance criteria are met.

Temperature1: 35 ºC
Time1: 1.14 minutes
Ramp1: 14.05ºC/minute
Final Temperature: 320ºC
Time2: 4 minutes
Total Time: 22.99 minutes
Temperature3: 230ºC

Injector Temperature: 285ºC
FID Temperature: 350ºC
Carrier gas: Hydrogen
Carrier pressure program:
9.89 psi initial
79cm/sec 20.6psi final
Detector Air: 300mL/minute
Detector Hydrogen: 30mL/minute
Injection Volume: 1 µL

10.2 Retention Time Windows

10.2.1 Prior to running an Initial Calibration on the instruments and when a new column has been installed you must compare the retention time windows.

10.2.2 Make 3 separate injections over a 72-hour period of Aliphatic and Aromatic standards to give the windows a broader window.

10.2.3 Record all retention times for each compound and range data points for Aliphatic and Aromatic sides on a spreadsheet to get the standard deviation and three times the standard deviation.

10.2.4 Save the retention time spreadsheet with the instrument for ten years.

10.3 Initial Calibration

10.3.1 Prepare calibration standards using the procedures in Section 8.3.1 at the concentrations listed in Table 2. At the minimum 5 standards must be used or 6 if non-linear regression will be used.

10.3.2 Establish the GC operating conditions by loading the appropriate GC method. Typical instrument conditions are listed in section 10.1.3. The same operating conditions are used for calibrations and sample analyses. Create the analytical sequence using the Agilent Chemstation data acquisition software. Record the calibration standard, unique lot number (EPH#) and analyst's initials in the analytical sequence list.

10.3.3 A 1µL injection volume of each calibration standard is typically used. Other injection volumes may be employed, provided that the analyst can demonstrate adequate sensitivity for the compounds of interest. The same injection volume must be used for calibration standards, samples and QC samples.

10.3.4 Calibration Factors

The external standard calibration technique is employed in this method. Calibration factors are determined for the individual PAH target compounds, the aromatic hydrocarbon range, and the two aliphatic hydrocarbon ranges as described below.

10.3.4.1 Individual PAH Target Compounds: In each standard, calculate the calibration factor for each analyte, the mean calibration factor, and the relative standard deviation (RSD) of the calibration factors. The calculations are performed automatically, using the formula listed in Alpha's Quality Manual and the CF equation below:

Calibration factor: Target PAH Analytes:

$$\textit{Calibration Factor (CF)} = \frac{\text{area of peak}}{\text{concentration injected (ng/µL)}}$$

10.3.4.2 Hydrocarbon Ranges: In each standard, calculate the calibration factor for each range, the mean calibration factor, and the relative standard deviation (RSD) of the calibration factors. The calculations are performed automatically, using the formulae listed in Alpha's Quality Manual.

Range Calibration Factor: Hydrocarbon Ranges:

$$\textit{Range CF} = \frac{\text{Area of Summation of Range Components}}{\text{Total concentration injected (ng/µL)}}$$

10.3.5 Initial Calibration Acceptance Criteria

If the **RSD for an analyte or range is < 25%,** then the response of the instrument for this compound is considered linear over the range and the mean calibration factor can be used to quantitate sample results.

If the **RSD for any analyte or range is > 25%**, then linearity through the origin cannot be assumed. An alternative calculation may be done by the use of linearity as long as the correlation coefficient (r) is **>0.99**. If both of these quantitation methods fail criteria for any compound in the initial calibration, then the system must be reevaluated, and a new calibration curve must be analyzed.

Percent Relative Error (%RE): For linear and quadratic fit compounds, the %RE must be calculated for the standard at or near the midpoint and at the lowest level of the initial calibration. The %RE between the calculated and expected concentration of an analyte must be ≤ 30%, otherwise recalibration is necessary.

10.3.6 Initial Calibration Verification

The initial calibration is verified using a standard source different than that used for the initial calibration. This standard is analyzed immediately after each initial calibration. Percent recoveries must be between **80-120%** for each target analyte.

10.3.6.1 EPH retention time (Rt) windows are defined as beginning 0.1 minutes before the elution of the beginning marker compound and ending 0.1 minutes after the elution of the ending marker compound, except for n-C_{19}, which is both a beginning and ending marker compound for two different ranges.

The C_9 – C_{18} Aliphatic Hydrocarbon range ends immediately (0.1minutes) before the elution of the n-C_{19} peak. The C_{19} – C_{36} Aliphatic Hydrocarbon range begins 0.1 before the elution of the n-C_{19} peak; therefore, there is no overlap of the two ranges and the n-C_{19} peak is only included in the C_{19} – C_{36} Aliphatic Hydrocarbon range.

EPH marker compounds and windows are summarized in Table 4.

10.4 Equipment Operation and Sample Processing

10.4.4 Additional Instrument QC

10.4.1.1 All four surrogate compounds must be adequately resolved from any individual analytes in the aliphatic and aromatic standards.

10.4.1.2 The n-nonane peak must be adequately resolved from the solvent peak.

10.4.1.3 All individual compound peaks in the aliphatic standard must be adequately resolved to baseline. In the aromatic standard, baseline separation must be seen for Phenanthrene and Anthracene. Benzo(a)anthracene, Chrysene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Dibenzo(a,h)anthracene, and Indeno(1,2,3-cd)pyrene will not demonstrate baseline separation, and may be reported as an un-resolved mixture, unless adequate resolution is obtained. (Adequate resolution is assumed to be achieved if the height of the valley between the two peaks is less than 25% of the average height of the two peaks.)

10.4.1.4The response ratio of C_{28} to C_{20} must be at least 0.85, to demonstrate that the system is not exhibiting mass discrimination. The response ratio for all CCVs is recorded in the instrument sequence log.

10.4.1.5Surrogates 1-Chloro-Octadecane and o-Terphenyl must be adequately resolved from any individual components in the aliphatic and aromatic hydrocarbon standards.

10.4.1.6Peaks for Naphthalene and n-dodecane in the aliphatic hydrocarbon fraction must be adequately resolved to allow for an accurate determination of the naphthalene concentration in the LCS/LCSD pair.

10.4.2 Sample Analysis

10.4.2.1 The same GC operating conditions used for the initial calibration must be employed for sample analyses, including sample injection volume (Section 10.1.3).

10.4.2.2 Samples are analyzed in a set referred to as an analytical sequence. The sequence begins with a mid-level calibration standard, followed by QC samples and sample extractsand ends with a mid-level calibration standard. An instrument solvent blank(s) can be loaded in the sequence after samples that appear to have a high concentration of petroleum hydrocarbons to prevent carryover into following samples. If any samples appear to have carryover in them from the previous sample, they are rerun. Instrument solvent blanks cannot be loaded before any instrument QC such as calibration standards. Inject 1µL sample extract using the solvent flush technique. The sample and calibration standard injection volume must be equal.

10.4.2.3 Establish daily retention time windows for each analyte of interest. Use the absolute retention time for each analyte as the midpoint of the window for that day. The daily retention time window equals the midpoint ± 3 times the standard deviation determined in Section 10.2.6.1. Alternatively, the default value of 0.1 minutes may be used for daily retention time window.

10.4.2.4 Tentative identification of an analyte occurs when a peak from a sample extract falls within the daily retention time window. Confirmation on a second GC column or by GC/MS analysis may be necessary.

10.4.2.5 Validation of GC system qualitative performance must be accomplished by the analysis of midlevel standards within the analysis sequence. If any of the standards fall outside their daily retention time window, the system is out of control. In such cases, the cause of the problem must be determined and corrected. All samples analyzed after the last passing CCV must be reanalyzed.

10.4.2.6 Aliphatic and aromatic ranges of interest are determined by the collective integration of all peaks eluting between specified range “marker”

compounds. The markers are the start time of the eluting peak and the stop time of the eluting peak.

10.4.2.7 When quantifying the aliphatic and aromatic ranges the integration must be from baseline (i.e., must include the unresolved complex mixture, or “hump”, areas). For the integration of individual Target Analytes and surrogate compounds, a valley-to-valley approach should typically be used, though this approach may be modified on a case-by-case basis by an experienced analyst.

10.4.2.8 Baseline correction using a system solvent blank is only permissible for the calculation of aliphatic and aromatic hydrocarbon range concentrations in accordance with the method. However, this correction is not routinely performed by Alpha.

10.4.2.9 If the Target PAH Analytes are to be quantitated using this method and the response for an individual analyte exceeds the linear range of the system, dilute the extract with methylene chloride and reanalyze. Extracts must be diluted so that all peaks are on scale. Overlapping peaks are not always evident when peaks are off scale. Computer reproduction of chromatograms, manipulated to ensure that all peaks are on scale over a 100-fold range, is acceptable if linearity is demonstrated prior to the analysis of samples.

10.4.3.0 For non-target analytes eluting in the aliphatic, aromatic or TPH fractions, the upper linear range of the system must be defined by peak height measurement, based upon the maximum peak height documented for an aliphatic or aromatic standard within the fraction that is shown to be within the linear range of the detector.

Under circumstances that a sample dilution is required because either the concentration of one or more of the target analytes exceeds the concentration of the respective calibration standard, or any non-target peak eluting with any aliphatic or aromatic range exceeds the peak height documented for the highest range-specific calibration standard, the RL for each target analyte and/or range must be adjusted in direct proportion to the dilution factor.

10.4.3.1 When performing dilutions for target analytes over the curve, the target analyte with the highest concentration is diluted to be with 60-80% of the highest calibration standard, so that the RLs will not be unnecessarily raised for analytes not needing dilutions. If there is a diluted and an undiluted run of the same sample the lowest dilution must be sent for TARGETS and the hydrocarbon range.

10.4.4 Data Transmission

When reporting multiple sets of data for a particular QC element or Sample, the “Arolist” file must be sent to the LIMS last in order to ensure that the C11-C22 Aromatic Adjusted Range is calculated correctly.

10.5 Continuing Calibration

10.5.1 The initial calibration curve is verified on each working day prior to the analysis of samples by the injection of the midpoint Calibration Standard. Continuing calibrations are also run throughout the analytical sequence, typically after every 20 samples or every 24 hours (whichever is more frequent) and at the end of analytical sequence. The percent difference (**%D**) for all aliphatic and aromatic analytes must be **less than or equal to**

25% except for **n-nonane**, which must be **<30.** In closing CCAL: up to four (4) compounds may exhibit a %D or % drift >25 but <40.

10.5.2 The Retention Times of each analyte are updated every time a CC is injected.

10.5.3 If the %D criteria cannot be met, inspect the gas chromatographic system to determine the cause and perform whatever maintenance is necessary before verifying calibration and proceeding with sample analysis.

10.5.4 If after maintenance the system still fails the calibration verification criteria, a new initial calibration curve must be analyzed.

10.6 Preventive Maintenance

10.6.1 Preventive Maintenance

Routine preventive maintenance must be performed to maintain GC system performance. This includes periodic replacement of injector septa, replacement of injector liner(s), and clipping several inches off the injector end of the capillary column.

10.6.2 Additional Maintenance

Additional maintenance may be required if system performance degrades. Autosampler syringes and syringe needles must be periodically inspected, especially if erratic response or peak discrimination is seen. If the maintenance steps listed above do not improve system performance, it may be necessary to replace the GC column(s).

11. Data Evaluation, Calculations and Reporting

The concentration reported for a particular hydrocarbon range or individual target analyte in an extract is calculated using the Enviroquant data processing software. The Enviroquant methods have been configured to utilize the quantitation formulas found in Alpha's Quality Manual. Proper quantitation requires the appropriate selection of a baseline from which the peak area or height can be determined. See the Manual Integration SOP for integration guidelines.

11.1 Hydrocarbon Ranges

11.1.1 Aliphatic Fraction

11.1.1.1 Determine the total area count for all peaks start time eluting 0.1 minutes before n-C_9 and 0.1 minutes before the start time elution of n-C_{19}. It is not necessary to identify or quantitate individual aliphatic compounds within this range. The area due to the aliphatic extraction surrogate (COD) must not be included in this area.

11.1.1.2 Determine the total area count for all peaks start time eluting at 0.1 minutes before n-C_{19} and 0.1 minutes stop time after the elution of n-C_{36}. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

11.1.1.3 Using the average calibration factor from the initial calibration, or linear regression analysis (if used), calculate the collective concentrations of C_9 through C_{18} Aliphatic Hydrocarbons, C_{19} through C_{36} Aliphatic Hydrocarbons, and individual concentrations of the sample surrogate, COD.

11.1.2 Aromatic Fraction

11.1.2.1 Determine the total area count for all peaks start time eluting 0.1 minutes before naphthalene and 0.1 minutes stop time after

benzo(g,h,i)perylene. The area due to the aromatic extraction surrogate (OTP) and the fractionation surrogates (2-Fluorobiphenyl and 2-Bromonaphthalene) must not be included in this area.

11.1.2.2 Using the average calibration factor from the initial calibration, or linear regression analysis (if used), calculate the concentrations of Unadjusted C_{11} through C_{22} Aromatic Hydrocarbons, the sample surrogate standard (OTP), and the fractionation surrogates.

11.2 Individual Aromatic Target Analytes

Determine the area for each of the individual PAH analytes found in the aromatic fraction. Using the average calibration factor from the initial calibration, or linear regression analysis (if used), calculate the concentrations of the individual analytes.

11.3 Data Manipulations

11.3.1 The collective concentration of the aromatic fraction (and/or TPH) **excludes** the individual concentrations of the Target PAH Analytes. Accordingly, a data manipulation step is necessary to adjust the collective range concentration calculated in Sections 11.1.1 and 11.1.2, to eliminate 'double counting' of analytes.

This necessary data manipulation step is taken by Alpha for inclusion on the client report. Subtract the individual concentrations of the Target or Diesel PAH Analytes from the collective concentration of Unadjusted C_{11} through C_{22} Aromatic Hydrocarbons and/or Unadjusted TPH. This calculation is performed automatically by Alpha's LIMS system.

Soil samples are reported on a dry-weight basis, utilizing formulae found in Alpha's Quality Manual. This calculation is performed automatically by Alpha's LIMS system.

The extent of data manipulations taken by the laboratory must be noted on the data reporting form. Refer to the MCP EPH WSC-CAM Section 3 and Table IV B-4 for reporting requirements for MCP.

11.3.2 Contamination of SPE Cartridges

11.3.2.1 Range integration areas may be affected by peaks identified during the injection of a Laboratory Method Blank and determined to be attributable to the leaching of plasticizers or other contaminants from silica gel SPE cartridges. In general, this contamination affects the C_{11} – C_{22} Aromatics. **Blank correction is not permissible.**

11.3.2.2 The laboratory must report the presence of this contamination in the associated range. Optionally, the laboratory may perform GC/MS analysis of the laboratory method blank extract to demonstrate that the contaminant in question is not a C_{11} – C_{22} aromatic compound. Analysis of only the method blank is acceptable as long as the associated samples exhibit the same contaminant peak at the same retention time. If demonstrated not to be a C_{11} – C_{22} aromatic compound, the contaminant does not need to be included in the calculation of the hydrocarbon range concentration. The laboratory must provide a discussion

in the case narrative if this approach is used (MADEP-EPH-19-1, section 11.2.6).

12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

Holding time exceedances, improper preservation and observed sample headspace are noted on the nonconformance report form.

Perform instrument maintenance as described throughout this SOP as needed when instrument calibration criteria are not met. Record all maintenance in the instrument maintenance logbook.

All batch and sample specific QC criteria outlined in Section 9 are evaluated by the analyst prior to approval of the data. When any QC criteria fail, the cause for the failure must be identified and corrected. This may include instrument recalibration followed by sample reanalysis or sample re-extraction. If it is determined that the failure is due to sample matrix effects, a project narrative report is written by the analyst for inclusion in the data report. If there is insufficient sample volume to perform the re-analysis for confirmation, this is also noted in the narrative and included in the client report.

13. Method Performance

13.1 Detection Limit Study (DL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the DL, LOD, and/or LOQ as outlined in Alpha SOP ID 1732. These studies performed by the laboratory are maintained on file for review.

13.1.1 Additional MAEPH Criteria

Analyze over three days a minimum of seven replicates of reagent water and/or clean sand blanks that have been fortified with all analytes or interest. Spiking concentrations are typically 10µg/L for water, and 0.17mg/kg for soil.

13.1.1.1 Water MDLs are determined by extracting a minimum of 7 replicates of 1L reagent water blanks spiked with OTP, COD and each analyte of interest.

13.1.1.2 Soil/sediment MDLs are determined by extracting a minimum of 7 replicates of 15g sand blanks spiked with OTP, COD, and each analyte of interest.

13.1.1.3 The mean accuracy of Nonane (C9), expressed as a percentage of the true value, must be between 30% and 140%. For other analytes, excluding C36, the mean accuracy must be between 40% and 140%. For each analyte, the %RSD must be less than or equal to 25%.

13.2 Demonstration of Capability Studies

Refer to Alpha SOP ID 1739 for further information regarding IDC/DOC Generation.

13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

13.2.3 Additional MAEPH Criteria

13.2.3.1 Accuracy and Precision

13.2.3.1.1 Extract each replicate according to the extraction SOP and analyze each replicate according to the procedures described in Section 10.3.2.

13.2.3.1.2 Calculate the measured concentrations of each analyte in all replicates, the mean accuracy (as percentage of true value) for each analyte, and the precision (as %RSD) of the measurements for each analyte.

13.2.3.1.3 For each analyte, excluding n-C_{36}, the mean accuracy, expressed as a percentage of the true value, must be within ± 40% of the true value for both aqueous and soil matrices. Poorer recoveries may be experienced for the n-C_{36} standard. For each analyte, the %RSD must be ± 25% for both aqueous and soil matrices. A higher %RSD is allowed for nonane if it is included in the collective calibration standard for the C_9 – C_{18} aliphatic range. Such allowable non-conformances must be documented.

13.2.3.2 Fractionation Efficiency

13.2.3.2.1 To demonstrate the initial capability of properly fractionating aliphatic and aromatic hydrocarbons in a sample, the analyst must first prepare and analyze the Fractionation Check Solution specified in Section 8.3.5.

13.2.3.2.2 To demonstrate the initial capability of properly fractionating aliphatic and aromatic hydrocarbons at least four (4) replicate FCCs must be extracted, fractionated per the appropriate extraction SOP, and analyzed. The mean % Recovery of the subject fractionation surrogates is determined.

13.2.3.2.3 For each analyte within the Fractionation Check Solution, excluding n-nonane, the % mean accuracy for four (4) replicate samples, expressed as a percentage of the mean true value, must be between 40% - 140%. Lower recoveries are permissible for n-nonane. If recovery of n-nonane is < 30%, the source of the problem must be found, and the fractionation check repeated.

13.2.3.3 Subsequent to the initial demonstration of laboratory capability, a Fractionation Check Solution is analyzed for each new lot of silica gel cartridges.

14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Hazardous Waste Management and Disposal SOP for further pollution prevention and waste management information.

15. Referenced Documents

2121 Chemical Hygiene Plan

1732 Detection Limit (DL), Limit of Detection (LOD) & Limit of Quantitation (LOQ) SOP

1739 Demonstration of Capability (DOC) Generation SOP

1728 Hazardous Waste Management and Disposal SOP

16. Attachments

Table 1 – REPORTING LIMITS
Table 2 – ALIPHATIC HYDROCARBON STANDARD
Table 3 – LIST OF COMPOUNDS AND CALIBRATION LEVELS
Table 4 – EPH MARKER COMPOUNDS

Table 1
Reporting Limits

Parameter	Aqueous	Soil
C9-C18 Aliphatic Hydrocarbons	100 µg/L	6.67 mg/Kg
C19-C36 Aliphatic Hydrocarbons	100 µg/L	6.67 mg/Kg
C11-C22 Aromatic Hydrocarbons	100 µg/L	6.67 mg/Kg
Naphthalene	10 µg/L	0.33 mg/Kg
2-Methylnaphthalene	10 µg/L	0.33 mg/Kg
Acenaphthylene	10 µg/L	0.33 mg/Kg
Acenaphthene	10 µg/L	0.33 mg/Kg
Fluorene	10 µg/L	0.33 mg/Kg
Phenanthrene	10 µg/L	0.33 mg/Kg
Anthracene	10 µg/L	0.33 mg/Kg
Fluoranthene	10 µg/L	0.33 mg/Kg
Pyrene	10 µg/L	0.33 mg/Kg
Benzo(a)anthracene	10 µg/L	0.33 mg/Kg
Chrysene	10 µg/L	0.33 mg/Kg
Benzo(b)fluoranthene	10 µg/L	0.33 mg/Kg
Benzo(k)fluoranthene	10 µg/L	0.33 mg/Kg
Benzo(a)pyrene	10 µg/L	0.33 mg/Kg

Indeno(1,2,3-cd)pyrene	10 µg/L	0.33 mg/Kg
Dibenzo(a,h)Anthracene	10 µg/L	0.33 mg/Kg
Benzo(g,h,i)perylene	10 µg/L	0.33 mg/Kg

Table 2
Aliphatic Hydrocarbon Standard

Compound	Carbon Number	Compound	Carbon Number
n-Nonane	C_9	n-Eicosane	C_{20}
n-Decane	C_{10}	n-Docosane	C_{22}
n-Dodecane	C_{12}	n-Tetracosane	C_{24}
n-Tetradecane	C_{14}	n-Hexacosane	C_{26}
n-Hexadecane	C_{16}	n-Octacosane	C_{28}
n-Octadecane	C_{18}	n-Tricontane	C_{30}
n-Nonadecane	C_{19}	n- Hexatriacontane	C_{36}

Table 3
List of Compounds and Calibration Levels

Component	Concentration of standard analytes (µg/mL)					
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Individual Aliphatic, Aromatic and Surrogate Compounds	1	5	10	20	50	100
Total Mass C_9 – C_{18} Aliphatic Hydrocarbons, ng (6 components)	6	30	60	120	300	600
Total Mass C_{19} – C_{36} Aliphatic Hydrocarbons, ng (8 components)	8	40	80	160	400	800
Total Mass C_{11} – C_{22} Aromatic Hydrocarbons/PAHs, ng (17 components)	17	85	170	340	850	1700

Table 4
EPH Marker Compounds

Range/Hydrocarbon Standard	Beginning Marker Compound	Ending Marker Compound
C_9 – C_{18} Aliphatic Hydrocarbons	n-Nonane	Just before n-Nonadecane
C_{19} – C_{36} Aliphatic Hydrocarbons	n-Nonadecane	n-Hexatriacontane
C_{11} – C_{22} Aromatic Hydrocarbons	Naphthalene	Benzo (g,h,i) Perylene

Appendix C Lab QAM and Certifications

NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. JOHN TRIMBLE
ALPHA ANALYTICAL
320 FORBES BOULEVARD
MANSFIELD, MA 02048

NY Lab Id No: 11627

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES AIR AND EMISSIONS
All approved analytes are listed below:

Acrylates	
Acetonitrile	EPA TO-15
Acrylonitrile	EPA TO-15
Methyl methacrylate	EPA TO-15
Chlorinated Hydrocarbons	
1,2,4-Trichlorobenzene	EPA TO-15
Hexachlorobutadiene	EPA TO-15
Polychlorinated Biphenyls	
PCBs and Aroclors	EPA TO-10A
	EPA TO-4A
Polynuclear Aromatics	
Acenaphthene	EPA TO-13A
Acenaphthylene	EPA TO-13A
Anthracene	EPA TO-13A
Benzo(a)anthracene	EPA TO-13A
Benzo(a)pyrene	EPA TO-13A
Benzo(b)fluoranthene	EPA TO-13A
Benzo(g,h,i)perylene	EPA TO-13A
Benzo(k)fluoranthene	EPA TO-13A
Chrysene	EPA TO-13A
Dibenzo(a,h)anthracene	EPA TO-13A
Fluoranthene	EPA TO-13A
Fluorene	EPA TO-13A
Indeno(1,2,3-cd)pyrene	EPA TO-13A
Naphthalene	EPA TO-13A
	EPA TO-15
Phenanthrene	EPA TO-13A

Serial No.: 68854

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Polynuclear Aromatics

Pyrene	EPA TO-13A

Purgeable Aromatics

1,2,4-Trimethylbenzene	EPA TO-15
1,2-Dichlorobenzene	EPA TO-15
1,3,5-Trimethylbenzene	EPA TO-15
1,3-Dichlorobenzene	EPA TO-15
1,4-Dichlorobenzene	EPA TO-15
2-Chlorotoluene	EPA TO-15
Benzene	EPA TO-15
Chlorobenzene	EPA TO-15
Ethyl benzene	EPA TO-15
Isopropylbenzene	EPA TO-15
m/p-Xylenes	EPA TO-15
o-Xylene	EPA TO-15
Styrene	EPA TO-15
Toluene	EPA TO-15
Total Xylenes	EPA TO-15

Purgeable Halocarbons

1,1,1-Trichloroethane	EPA TO-15
1,1,2,2-Tetrachloroethane	EPA TO-15
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA TO-15
1,1,2-Trichloroethane	EPA TO-15
1,1-Dichloroethane	EPA TO-15
1,1-Dichloroethene	EPA TO-15
1,2-Dibromo-3-chloropropane	EPA TO-15
1,2-Dibromoethane	EPA TO-15

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All approved analytes are listed below:

Purgeable Halocarbons

1,2-Dichloroethane	EPA TO-15
1,2-Dichloropropane	EPA TO-15
3-Chloropropene (Allyl chloride)	EPA TO-15
Bromodichloromethane	EPA TO-15
Bromoform	EPA TO-15
Bromomethane	EPA TO-15
Carbon tetrachloride	EPA TO-15
Chloroethane	EPA TO-15
Chloroform	EPA TO-15
Chloromethane	EPA TO-15
cis-1,2-Dichloroethene	EPA TO-15
cis-1,3-Dichloropropene	EPA TO-15
Dibromochloromethane	EPA TO-15
Dichlorodifluoromethane	EPA TO-15
Methylene chloride	EPA TO-15
Tetrachloroethene	EPA TO-15
trans-1,2-Dichloroethene	EPA TO-15
trans-1,3-Dichloropropene	EPA TO-15
Trichloroethene	EPA TO-15
Trichlorofluoromethane	EPA TO-15
Vinyl bromide	EPA TO-15
Vinyl chloride	EPA TO-15

Volatile Chlorinated Organics

Benzyl chloride	EPA TO-15

Volatile Organics

1,2-Dichlorotetrafluoroethane	EPA TO-15

Serial No.: 68854

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. JOHN TRIMBLE
ALPHA ANALYTICAL
320 FORBES BOULEVARD
MANSFIELD, MA 02048

NY Lab Id No: 11627

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES AIR AND EMISSIONS
All approved analytes are listed below:

Volatile Organics

1,3-Butadiene	EPA TO-15
1,4-Dioxane	EPA TO-15
2,2,4-Trimethylpentane	EPA TO-15
2-Butanone (Methylethyl ketone)	EPA TO-15
4-Methyl-2-Pentanone	EPA TO-15
Acetaldehyde	EPA TO-15
Acetone	EPA TO-15
Acrolein (Propenal)	EPA TO-15
Carbon Disulfide	EPA TO-15
Cyclohexane	EPA TO-15
Hexane	EPA TO-15
Isopropanol	EPA TO-15
Methanol	EPA TO-15
Methyl tert-butyl ether	EPA TO-15
n-Heptane	EPA TO-15
tert-butyl alcohol	EPA TO-15
Vinyl acetate	EPA TO-15

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All approved analytes are listed below:

Metals I

Arsenic, Total	EPA 200.8 Rev. 5.4
Barium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Cadmium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Chromium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Copper, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Iron, Total	EPA 200.7 Rev. 4.4
Lead, Total	EPA 200.8 Rev. 5.4
Manganese, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Mercury, Total	EPA 245.1 Rev. 3.0
Selenium, Total	EPA 200.8 Rev. 5.4
Silver, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Zinc, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4

Metals II

Aluminum, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Antimony, Total	EPA 200.8 Rev. 5.4
Beryllium, Total	EPA 200.8 Rev. 5.4
Nickel, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4
Thallium, Total	EPA 200.8 Rev. 5.4

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All approved analytes are listed below:

Metals II

Vanadium, Total	EPA 200.7 Rev. 4.4
	EPA 200.8 Rev. 5.4

Metals III

Boron, Total	EPA 200.7 Rev. 4.4
Calcium, Total	EPA 200.7 Rev. 4.4
Magnesium, Total	EPA 200.7 Rev. 4.4
Potassium, Total	EPA 200.7 Rev. 4.4
Sodium, Total	EPA 200.7 Rev. 4.4

Miscellaneous

1,4-Dioxane	EPA 522

Non-Metals

Calcium Hardness	EPA 200.7 Rev. 4.4

Perfluorinated Alkyl Acids

11CL-PF3OUDS	EPA 533
	EPA 537.1
4:2FTS	EPA 533
6:2FTS	EPA 533
8:2FTS	EPA 533
9CL-PF3ONS	EPA 533
	EPA 537.1
ADONA	EPA 533
	EPA 537.1
Hexafluoropropylene Oxide Dimer Aci	EPA 533
	EPA 537.1
NETFOSAA	EPA 537.1
NMEFOSAA	EPA 537.1

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All approved analytes are listed below:

Perfluorinated Alkyl Acids

Nonafluoro-3,6-Dioxaheptanoic Acid	EPA 533
Perflourotridecanoic Acid (PFTRDA)	EPA 537.1
Perfluordecanoic Acid (PFDA)	EPA 533
	EPA 537.1
Perfluoro-3-Methoxypropanoic Acid	EPA 533
Perfluoro-4-Methoxybutanoic Acid	EPA 533
Perfluorobutanesulfonic Acid (PFBS)	EPA 533
	EPA 537.1
Perfluorobutanoic Acid (PFBA)	EPA 533
Perfluorododecanoic Acid (PFDOA)	EPA 533
	EPA 537.1
Perfluoroheptanesulfonic Acid (PFHPS	EPA 533
Perfluoroheptanoic Acid (PFHPA)	EPA 533
	EPA 537.1
Perfluorohexanesulfonic Acid (PFHXS	EPA 533
	EPA 537.1
Perfluorohexanoic Acid (PFHXA)	EPA 533
	EPA 537.1
Perfluorononanoic Acid (PFNA)	EPA 533
	EPA 537.1
Perfluorooctanesulfonic Acid (PFOS)	EPA 533
	EPA 537.1
Perfluorooctanoic Acid (PFOA)	EPA 533
	EPA 537.1
Perfluoropentanesulfonic Acid (PFPES	EPA 533
Perfluoropentanoic Acid (PFPEA)	EPA 533
Perfluorotetradecanoic Acid (PFTA)	EPA 537.1

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Perfluorinated Alkyl Acids

Perfluoroundecanoic Acid (PFUNA)	EPA 533
	EPA 537.1
PFEESA	EPA 533

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Amines

1,2-Diphenylhydrazine	EPA 8270E
2-Nitroaniline	EPA 8270E
3-Nitroaniline	EPA 8270E
4-Chloroaniline	EPA 8270E
4-Nitroaniline	EPA 8270E
Aniline	EPA 8270E
Carbazole	EPA 8270E
Pyridine	EPA 8270E

Benzidines

3,3'-Dichlorobenzidine	EPA 8270E
Benzidine	EPA 8270E

Chlorinated Hydrocarbon Pesticides

4,4'-DDD	EPA 8081B
4,4'-DDE	EPA 8081B
4,4'-DDT	EPA 8081B
Aldrin	EPA 8081B
alpha-BHC	EPA 8081B
alpha-Chlordane	EPA 8081B
beta-BHC	EPA 8081B
Chlordane Total	EPA 8081B
delta-BHC	EPA 8081B
Dieldrin	EPA 8081B
Endosulfan I	EPA 8081B
Endosulfan II	EPA 8081B
Endosulfan sulfate	EPA 8081B
Endrin	EPA 8081B

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Chlorinated Hydrocarbon Pesticides

Endrin aldehyde	EPA 8081B
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 8081B
Heptachlor epoxide	EPA 8081B
Isodrin	EPA 8081B
Lindane	EPA 8081B
Methoxychlor	EPA 8081B
Mirex	EPA 8081B
PCNB	EPA 8270E
Toxaphene	EPA 8081B

Chlorinated Hydrocarbons

1,2,4,5-Tetrachlorobenzene	EPA 8270E
1,2,4-Trichlorobenzene	EPA 8270E
2-Chloronaphthalene	EPA 8270E
Hexachlorobenzene	EPA 8081B
	EPA 8270E
Hexachlorobutadiene	EPA 8270E
Hexachlorocyclopentadiene	EPA 8270E
Hexachloroethane	EPA 8270E

Fuel Oxygenates

Ethanol	EPA 8015D
tert-amyl alcohol	EPA 8015D
tert-butyl alcohol	EPA 8015D

Haloethers

2,2'-Oxybis(1-chloropropane)	EPA 8270E

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Haloethers

4-Bromophenylphenyl ether	EPA 8270E
4-Chlorophenylphenyl ether	EPA 8270E
Bis(2-chloroethoxy)methane	EPA 8270E
Bis(2-chloroethyl)ether	EPA 8270E

Low Level Polynuclear Aromatics

Acenaphthene Low Level	EPA 8270E SIM
Acenaphthylene Low Level	EPA 8270E SIM
Anthracene Low Level	EPA 8270E SIM
Benzo(a)anthracene Low Level	EPA 8270E SIM
Benzo(a)pyrene Low Level	EPA 8270E SIM
Benzo(b)fluoranthene Low Level	EPA 8270E SIM
Benzo(g,h,i)perylene Low Level	EPA 8270E SIM
Benzo(k)fluoranthene Low Level	EPA 8270E SIM
Chrysene Low Level	EPA 8270E SIM
Dibenzo(a,h)anthracene Low Level	EPA 8270E SIM
Fluoranthene Low Level	EPA 8270E SIM
Fluorene Low Level	EPA 8270E SIM
Indeno(1,2,3-cd)pyrene Low Level	EPA 8270E SIM
Naphthalene Low Level	EPA 8270E SIM
Phenanthrene Low Level	EPA 8270E SIM
Pyrene Low Level	EPA 8270E SIM

Metals I

Barium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)

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Metals I

Cadmium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Calcium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
Chromium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Copper, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Iron, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Lead, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Magnesium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
Manganese, Total	EPA 200.7, Rev. 4.4 (1994)

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Metals I

Manganese, Total	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Nickel, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Potassium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Silver, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Sodium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Strontium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B

Metals II

Aluminum, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)

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Metals II

Antimony, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Arsenic, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Beryllium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Mercury, Low Level	EPA 1631E
Mercury, Total	EPA 245.1, Rev. 3.0 (1994)
	EPA 7470A
Selenium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Vanadium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Zinc, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)

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Metals III

Cobalt, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Molybdenum, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Thallium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
	EPA 200.8, Rev. 5.4 (1994)
Tin, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
Titanium, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D

Mineral

Hardness, Total	SM 2340B-2011

Miscellaneous

Boron, Total	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D
	EPA 6020B
Silica, Dissolved	EPA 200.7, Rev. 4.4 (1994)
	EPA 6010D

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Nitroaromatics and Isophorone

2,4-Dinitrotoluene	EPA 8270E
2,6-Dinitrotoluene	EPA 8270E
Isophorone	EPA 8270E
Nitrobenzene	EPA 8270E

Nitrosoamines

N-Nitrosodimethylamine	EPA 8270E
N-Nitrosodi-n-propylamine	EPA 8270E
N-Nitrosodiphenylamine	EPA 8270E

Organophosphate Pesticides

Atrazine	EPA 8270E

Perfluorinated Alkyl Acids

11CL-PF3OUDS	EPA 1633 (Draft)
4:2FTS	EPA 1633 (Draft)
6:2FTS	EPA 1633 (Draft)
8:2FTS	EPA 1633 (Draft)
9CL-PF3ONS	EPA 1633 (Draft)
ADONA	EPA 1633 (Draft)
Hexafluoropropylene Oxide Dimer Acic	EPA 1633 (Draft)
NETFOSAA	EPA 1633 (Draft)
NMEFOSAA	EPA 1633 (Draft)
Nonafluoro-3,6-Dioxaheptanoic Acid	EPA 1633 (Draft)
Perflourotridecanoic Acid (PFTRDA)	EPA 1633 (Draft)
Perfluordecanoic Acid (PFDA)	EPA 1633 (Draft)
Perfluoro-3-Methoxypropanoic Acid	EPA 1633 (Draft)
Perfluoro-4-Methoxybutanoic Acid	EPA 1633 (Draft)
Perfluorobutanesulfonic Acid (PFBS)	EPA 1633 (Draft)

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. JOHN TRIMBLE
ALPHA ANALYTICAL
320 FORBES BOULEVARD
MANSFIELD, MA 02048

NY Lab Id No: 11627

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Perfluorinated Alkyl Acids

Perfluorobutanoic Acid (PFBA)	EPA 1633 (Draft)
Perfluorododecanoic Acid (PFDOA)	EPA 1633 (Draft)
Perfluoroheptanesulfonic Acid (PFHPS	EPA 1633 (Draft)
Perfluoroheptanoic Acid (PFHPA)	EPA 1633 (Draft)
Perfluorohexanesulfonic Acid (PFHXS	EPA 1633 (Draft)
Perfluorohexanoic Acid (PFHXA)	EPA 1633 (Draft)
Perfluorononanoic Acid (PFNA)	EPA 1633 (Draft)
Perfluorooctanesulfonic Acid (PFOS)	EPA 1633 (Draft)
Perfluorooctanoic Acid (PFOA)	EPA 1633 (Draft)
Perfluoropentanesulfonic Acid (PFPES	EPA 1633 (Draft)
Perfluoropentanoic Acid (PFPEA)	EPA 1633 (Draft)
Perfluorotetradecanoic Acid (PFTA)	EPA 1633 (Draft)
Perfluoroundecanoic Acid (PFUNA)	EPA 1633 (Draft)
PFEESA	EPA 1633 (Draft)

Petroleum Hydrocarbons

Diesel Range Organics	EPA 8015D

Phthalate Esters

Benzyl butyl phthalate	EPA 8270E
Bis(2-ethylhexyl) phthalate	EPA 8270E
Diethyl phthalate	EPA 8270E
Dimethyl phthalate	EPA 8270E
Di-n-butyl phthalate	EPA 8270E
Di-n-octyl phthalate	EPA 8270E

Polychlorinated Biphenyls

Aroclor 1016 (PCB-1016)	EPA 8082A
Aroclor 1221 (PCB-1221)	EPA 8082A

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Polychlorinated Biphenyls

Aroclor 1232 (PCB-1232)	EPA 8082A
Aroclor 1242 (PCB-1242)	EPA 8082A
Aroclor 1248 (PCB-1248)	EPA 8082A
Aroclor 1254 (PCB-1254)	EPA 8082A
Aroclor 1260 (PCB-1260)	EPA 8082A
Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A

Polynuclear Aromatics

Acenaphthene	EPA 8270E
Acenaphthylene	EPA 8270E
Anthracene	EPA 8270E
Benzo(a)anthracene	EPA 8270E
Benzo(a)pyrene	EPA 8270E
Benzo(b)fluoranthene	EPA 8270E
Benzo(g,h,i)perylene	EPA 8270E
Benzo(k)fluoranthene	EPA 8270E
Chrysene	EPA 8270E
Dibenzo(a,h)anthracene	EPA 8270E
Fluoranthene	EPA 8270E
Fluorene	EPA 8270E
Indeno(1,2,3-cd)pyrene	EPA 8270E
Naphthalene	EPA 8270E
Phenanthrene	EPA 8270E
Pyrene	EPA 8270E

Priority Pollutant Phenols

2,3,4,6 Tetrachlorophenol	EPA 8270E

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Priority Pollutant Phenols

2,4,5-Trichlorophenol	EPA 8270E
2,4,6-Trichlorophenol	EPA 8270E
2,4-Dichlorophenol	EPA 8270E
2,4-Dimethylphenol	EPA 8270E
2,4-Dinitrophenol	EPA 8270E
2-Chlorophenol	EPA 8270E
2-Methyl-4,6-dinitrophenol	EPA 8270E
2-Methylphenol	EPA 8270E
2-Nitrophenol	EPA 8270E
3-Methylphenol	EPA 8270E
4-Chloro-3-methylphenol	EPA 8270E
4-Methylphenol	EPA 8270E
4-Nitrophenol	EPA 8270E
Pentachlorophenol	EPA 8270E
Phenol	EPA 8270E

Semi-Volatile Organics

1,1'-Biphenyl	EPA 8270E
1,2-Dichlorobenzene, Semi-volatile	EPA 8270E
1,3-Dichlorobenzene, Semi-volatile	EPA 8270E
1,4-Dichlorobenzene, Semi-volatile	EPA 8270E
2-Methylnaphthalene	EPA 8270E
Acetophenone	EPA 8270E
Benzaldehyde	EPA 8270E
Benzoic Acid	EPA 8270E
Benzyl alcohol	EPA 8270E
Caprolactam	EPA 8270E
Dibenzofuran	EPA 8270E

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All approved analytes are listed below:

Volatiles Organics

1,4-Dioxane	EPA 8270E SIM
Ethylene Glycol	EPA 8015D
Isobutyl alcohol	EPA 8015D
Methanol	EPA 8015D
Propylene Glycol	EPA 8015D

Sample Preparation Methods

	EPA 3015A
	EPA 3005A
	EPA 3510C

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All approved analytes are listed below:

Amines

1,2-Diphenylhydrazine	EPA 8270E
2-Nitroaniline	EPA 8270E
3-Nitroaniline	EPA 8270E
4-Chloroaniline	EPA 8270E
4-Nitroaniline	EPA 8270E
Aniline	EPA 8270E
Carbazole	EPA 8270E

Benzidines

3,3'-Dichlorobenzidine	EPA 8270E
Benzidine	EPA 8270E

Chlorinated Hydrocarbon Pesticides

4,4'-DDD	EPA 8081B
4,4'-DDE	EPA 8081B
4,4'-DDT	EPA 8081B
Aldrin	EPA 8081B
alpha-BHC	EPA 8081B
alpha-Chlordane	EPA 8081B
beta-BHC	EPA 8081B
Chlordane Total	EPA 8081B
delta-BHC	EPA 8081B
Dieldrin	EPA 8081B
Endosulfan I	EPA 8081B
Endosulfan II	EPA 8081B
Endosulfan sulfate	EPA 8081B
Endrin	EPA 8081B
Endrin aldehyde	EPA 8081B

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Chlorinated Hydrocarbon Pesticides

Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 8081B
Heptachlor epoxide	EPA 8081B
Lindane	EPA 8081B
Methoxychlor	EPA 8081B
Mirex	EPA 8081B
Pentachloronitrobenzene	EPA 8270E
Toxaphene	EPA 8081B

Chlorinated Hydrocarbons

1,2,4,5-Tetrachlorobenzene	EPA 8270E
1,2,4-Trichlorobenzene	EPA 8270E
2-Chloronaphthalene	EPA 8270E
Hexachlorobenzene	EPA 8270E
Hexachlorobutadiene	EPA 8270E
Hexachlorocyclopentadiene	EPA 8270E
Hexachloroethane	EPA 8270E

Haloethers

2,2'-Oxybis(1-chloropropane)	EPA 8270E
4-Bromophenylphenyl ether	EPA 8270E
4-Chlorophenylphenyl ether	EPA 8270E
Bis(2-chloroethoxy)methane	EPA 8270E
Bis(2-chloroethyl)ether	EPA 8270E

Low Level Polynuclear Aromatic Hydrocarbons

Acenaphthene Low Level	EPA 8270E SIM
Acenaphthylene Low Level	EPA 8270E SIM

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Low Level Polynuclear Aromatic Hydrocarbons

Anthracene Low Level	EPA 8270E SIM
Benzo(a)anthracene Low Level	EPA 8270E SIM
Benzo(a)pyrene Low Level	EPA 8270E SIM
Benzo(b)fluoranthene Low Level	EPA 8270E SIM
Benzo(g,h,i)perylene Low Level	EPA 8270E SIM
Benzo(k)fluoranthene Low Level	EPA 8270E SIM
Chrysene Low Level	EPA 8270E SIM
Dibenzo(a,h)anthracene Low Level	EPA 8270E SIM
Fluoranthene Low Level	EPA 8270E SIM
Fluorene Low Level	EPA 8270E SIM
Indeno(1,2,3-cd)pyrene Low Level	EPA 8270E SIM
Naphthalene Low Level	EPA 8270E SIM
Phenanthrene Low Level	EPA 8270E SIM
Pyrene Low Level	EPA 8270E SIM

Metals I

Barium, Total	EPA 6010D
	EPA 6020B
Cadmium, Total	EPA 6010D
	EPA 6020B
Calcium, Total	EPA 6010D
	EPA 6020B
Chromium, Total	EPA 6010D
	EPA 6020B
Copper, Total	EPA 6010D
	EPA 6020B
Iron, Total	EPA 6010D
	EPA 6020B

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Metals I

Lead, Total	EPA 6010D
	EPA 6020B
Magnesium, Total	EPA 6010D
	EPA 6020B
Manganese, Total	EPA 6010D
	EPA 6020B
Nickel, Total	EPA 6010D
	EPA 6020B
Potassium, Total	EPA 6010D
	EPA 6020B
Silver, Total	EPA 6010D
	EPA 6020B
Sodium, Total	EPA 6010D
	EPA 6020B
Strontium, Total	EPA 6010D
	EPA 6020B

Metals II

Aluminum, Total	EPA 6010D
	EPA 6020B
Antimony, Total	EPA 6010D
	EPA 6020B
Arsenic, Total	EPA 6010D
	EPA 6020B
Beryllium, Total	EPA 6010D
	EPA 6020B
Mercury, Total	EPA 7471B
	EPA 7474

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Metals II

Selenium, Total	EPA 6010D
	EPA 6020B
Vanadium, Total	EPA 6010D
	EPA 6020B
Zinc, Total	EPA 6010D
	EPA 6020B

Metals III

Cobalt, Total	EPA 6010D
	EPA 6020B
Molybdenum, Total	EPA 6010D
	EPA 6020B
Thallium, Total	EPA 6010D
	EPA 6020B
Tin, Total	EPA 6010D
	EPA 6020B
Titanium, Total	EPA 6010D

Miscellaneous

Boron, Total	EPA 6010D
Organic Carbon, Total	Lloyd Kahn Method
	EPA 9060A

Nitroaromatics and Isophorone

2,4-Dinitrotoluene	EPA 8270E
2,6-Dinitrotoluene	EPA 8270E
Isophorone	EPA 8270E
Nitrobenzene	EPA 8270E
Pyridine	EPA 8270E

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Nitrosoamines

N-Nitrosodimethylamine	EPA 8270E
N-Nitrosodi-n-propylamine	EPA 8270E
N-Nitrosodiphenylamine	EPA 8270E

Perfluorinated Alkyl Acids

8:2FTS	EPA 1633 (Draft)
NETFOSAA	EPA 1633 (Draft)
NMEFOSAA	EPA 1633 (Draft)
Perflourotridecanoic Acid (PFTRDA)	EPA 1633 (Draft)
Perfluordecanoic Acid (PFDA)	EPA 1633 (Draft)
Perfluorobutanoic Acid (PFBA)	EPA 1633 (Draft)
Perfluorododecanoic Acid (PFDOA)	EPA 1633 (Draft)
Perfluoroheptanoic Acid (PFHPA)	EPA 1633 (Draft)
Perfluorohexanoic Acid (PFHXA)	EPA 1633 (Draft)
Perfluorononanoic Acid (PFNA)	EPA 1633 (Draft)
Perfluorooctanesulfonic Acid (PFOS)	EPA 1633 (Draft)
Perfluorooctanoic Acid (PFOA)	EPA 1633 (Draft)
Perfluoropentanoic Acid (PFPEA)	EPA 1633 (Draft)
Perfluorotetradecanoic Acid (PFTA)	EPA 1633 (Draft)
Perfluoroundecanoic Acid (PFUNA)	EPA 1633 (Draft)

Petroleum Hydrocarbons

Diesel Range Organics	EPA 8015D

Phthalate Esters

Benzyl butyl phthalate	EPA 8270E
Bis(2-ethylhexyl) phthalate	EPA 8270E
Diethyl phthalate	EPA 8270E
Dimethyl phthalate	EPA 8270E

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Phthalate Esters

Di-n-butyl phthalate	EPA 8270E
Di-n-octyl phthalate	EPA 8270E

Polychlorinated Biphenyls

Aroclor 1016 (PCB-1016)	EPA 8082A
Aroclor 1221 (PCB-1221)	EPA 8082A
Aroclor 1232 (PCB-1232)	EPA 8082A
Aroclor 1242 (PCB-1242)	EPA 8082A
Aroclor 1248 (PCB-1248)	EPA 8082A
Aroclor 1254 (PCB-1254)	EPA 8082A
Aroclor 1260 (PCB-1260)	EPA 8082A
Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A

Polynuclear Aromatic Hydrocarbons

Acenaphthene	EPA 8270E
Acenaphthylene	EPA 8270E
Anthracene	EPA 8270E
Benzo(a)anthracene	EPA 8270E
Benzo(a)pyrene	EPA 8270E
Benzo(b)fluoranthene	EPA 8270E
Benzo(g,h,i)perylene	EPA 8270E
Benzo(k)fluoranthene	EPA 8270E
Chrysene	EPA 8270E
Dibenzo(a,h)anthracene	EPA 8270E
Fluoranthene	EPA 8270E
Fluorene	EPA 8270E
Indeno(1,2,3-cd)pyrene	EPA 8270E

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Polynuclear Aromatic Hydrocarbons

Naphthalene	EPA 8270E
Phenanthrene	EPA 8270E
Pyrene	EPA 8270E

Priority Pollutant Phenols

2,3,4,6 Tetrachlorophenol	EPA 8270E
2,4,5-Trichlorophenol	EPA 8270E
2,4,6-Trichlorophenol	EPA 8270E
2,4-Dichlorophenol	EPA 8270E
2,4-Dimethylphenol	EPA 8270E
2,4-Dinitrophenol	EPA 8270E
2-Chlorophenol	EPA 8270E
2-Methyl-4,6-dinitrophenol	EPA 8270E
2-Methylphenol	EPA 8270E
2-Nitrophenol	EPA 8270E
3-Methylphenol	EPA 8270E
4-Chloro-3-methylphenol	EPA 8270E
4-Methylphenol	EPA 8270E
4-Nitrophenol	EPA 8270E
Pentachlorophenol	EPA 8270E
Phenol	EPA 8270E

Semi-Volatile Organics

1,1'-Biphenyl	EPA 8270E
1,2-Dichlorobenzene, Semi-volatile	EPA 8270E
1,3-Dichlorobenzene, Semi-volatile	EPA 8270E
1,4-Dichlorobenzene, Semi-volatile	EPA 8270E
2-Methylnaphthalene	EPA 8270E

Serial No.: 68853

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. JOHN TRIMBLE
ALPHA ANALYTICAL
320 FORBES BOULEVARD
MANSFIELD, MA 02048

NY Lab Id No: 11627

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Semi-Volatile Organics

Acetophenone	EPA 8270E
Benzaldehyde	EPA 8270E
Benzoic Acid	EPA 8270E
Benzyl alcohol	EPA 8270E
Caprolactam	EPA 8270E
Dibenzofuran	EPA 8270E

Volatile Organics

1,4-Dioxane	EPA 8270E SIM
Ethylene Glycol	EPA 8015D
Isobutyl alcohol	EPA 8015D
tert-butyl alcohol	EPA 8015D

Sample Preparation Methods

	EPA 3570
	EPA 3580A
	EPA 3050B
	EPA 3051A

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MR. MARCO SOARES
ALPHA ANALYTICAL LLC
8 WALKUP DR
WESTBOROUGH, MA 01581-1019

NY Lab Id No: 11148

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES POTABLE WATER
All approved analytes are listed below:

Bacteriology

Coliform, Total / E. coli (Qualitative)	SM 20, 21-23 9223B (-04) (Colilert)
E. coli (Enumeration)	SM 20, 21-23 9223B (-04) (Colilert)
Heterotrophic Plate Count	SM 20, 21-23 9215B (-04)

Fuel Additives

Methyl tert-butyl ether	EPA 524.2
Naphthalene	EPA 524.2

Microextractables

1,2,3-Trichloropropane, Low Level	EPA 504.1
1,2-Dibromo-3-chloropropane, Low Le	EPA 504.1
1,2-Dibromoethane, Low Level	EPA 504.1

Miscellaneous

Odor	SM 21-23 2150 B (-97)
Organic Carbon, Dissolved	SM 21-23 5310C (-00)
Organic Carbon, Total	SM 21-23 5310C (-00)
Turbidity	SM 21-23 2130 B (-01)
	EPA 180.1 Rev. 2.0

Non-Metals

Alkalinity	SM 21-23 2320B (-97)
Chloride	EPA 300.0 Rev. 2.1
Color	SM 21-23 2120B (-01)
Cyanide	SM 20, 21-23 4500-CN E
Fluoride, Total	EPA 300.0 Rev. 2.1
	SM 21-23 4500-F C (-97)
Nitrate (as N)	EPA 353.2 Rev. 2.0
	EPA 300.0 Rev. 2.1
	SM 21-23 4500-NO3 F (-00)

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Non-Metals

Nitrite (as N)	EPA 353.2 Rev. 2.0
	SM 21-23 4500-NO3 F (-00)
	SM 21-23 4500-NO2 B (-00)
Orthophosphate (as P)	SM 19, 21-23 4500-P E (-99)
Solids, Total Dissolved	SM 21-23 2540C (-97)
Specific Conductance	SM 21-23 2510B (-97)
Sulfate (as SO4)	EPA 300.0 Rev. 2.1

Trihalomethanes

Bromodichloromethane	EPA 524.2
Bromoform	EPA 524.2
Chloroform	EPA 524.2
Dibromochloromethane	EPA 524.2
Total Trihalomethanes	EPA 524.2

Volatile Aromatics

1,2,3-Trichlorobenzene	EPA 524.2
1,2,4-Trichlorobenzene	EPA 524.2
1,2,4-Trimethylbenzene	EPA 524.2
1,2-Dichlorobenzene	EPA 524.2
1,3,5-Trimethylbenzene	EPA 524.2
1,3-Dichlorobenzene	EPA 524.2
1,4-Dichlorobenzene	EPA 524.2
2-Chlorotoluene	EPA 524.2
4-Chlorotoluene	EPA 524.2
Benzene	EPA 524.2
Bromobenzene	EPA 524.2
Chlorobenzene	EPA 524.2

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Volatile Aromatics

Ethyl benzene	EPA 524.2
Hexachlorobutadiene	EPA 524.2
Isopropylbenzene	EPA 524.2
n-Butylbenzene	EPA 524.2
n-Propylbenzene	EPA 524.2
p-Isopropyltoluene (P-Cymene)	EPA 524.2
sec-Butylbenzene	EPA 524.2
Styrene	EPA 524.2
tert-Butylbenzene	EPA 524.2
Toluene	EPA 524.2
Total Xylenes	EPA 524.2

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 524.2
1,1,1-Trichloroethane	EPA 524.2
1,1,2,2-Tetrachloroethane	EPA 524.2
1,1,2-Trichloroethane	EPA 524.2
1,1-Dichloroethane	EPA 524.2
1,1-Dichloroethene	EPA 524.2
1,1-Dichloropropene	EPA 524.2
1,2,3-Trichloropropane	EPA 524.2
1,2-Dichloroethane	EPA 524.2
1,2-Dichloropropane	EPA 524.2
1,3-Dichloropropane	EPA 524.2
2,2-Dichloropropane	EPA 524.2
Bromochloromethane	EPA 524.2
Bromomethane	EPA 524.2
Carbon tetrachloride	EPA 524.2

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Volatile Halocarbons

Chloroethane	EPA 524.2
Chloromethane	EPA 524.2
cis-1,2-Dichloroethene	EPA 524.2
cis-1,3-Dichloropropene	EPA 524.2
Dibromomethane	EPA 524.2
Dichlorodifluoromethane	EPA 524.2
Methylene chloride	EPA 524.2
Tetrachloroethene	EPA 524.2
trans-1,2-Dichloroethene	EPA 524.2
trans-1,3-Dichloropropene	EPA 524.2
Trichloroethene	EPA 524.2
Trichlorofluoromethane	EPA 524.2
Vinyl chloride	EPA 524.2

Sample Preparation Methods

SM 20, 21-23 4500-CN C (-99)

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All approved analytes are listed below:

Acrylates

Acrolein (Propenal)	EPA 8260D
	EPA 624.1
Acrylonitrile	EPA 8260D
	EPA 624.1
Ethyl methacrylate	EPA 8260D
Methyl methacrylate	EPA 8260D

Amines

1,2-Diphenylhydrazine	EPA 625.1
	EPA 8270E
2-Naphthylamine	EPA 8270E
2-Nitroaniline	EPA 8270E
3-Nitroaniline	EPA 8270E
4-Chloroaniline	EPA 8270E
4-Nitroaniline	EPA 8270E
Aniline	EPA 625.1
	EPA 8270E
Carbazole	EPA 625.1
	EPA 8270E
Diphenylamine	EPA 8270E
Pyridine	EPA 625.1
	EPA 8270E

Bacteriology

Coliform, Fecal	Colilert-18
E. coli (Enumeration)	SM 9223B-2016
Heterotrophic Plate Count	SM 18-21 9215B

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Benzidines

3,3'-Dichlorobenzidine	EPA 625.1
	EPA 8270E
Benzidine	EPA 625.1
	EPA 8270E

Chlorinated Hydrocarbon Pesticides

4,4'-DDD	EPA 8081B
	EPA 608.3
4,4'-DDE	EPA 8081B
	EPA 608.3
4,4'-DDT	EPA 8081B
	EPA 608.3
Aldrin	EPA 8081B
	EPA 608.3
alpha-BHC	EPA 8081B
	EPA 608.3
alpha-Chlordane	EPA 8081B
	EPA 608.3
beta-BHC	EPA 8081B
	EPA 608.3
Chlordane Total	EPA 8081B
	EPA 608.3
delta-BHC	EPA 8081B
	EPA 608.3
Dieldrin	EPA 8081B
	EPA 608.3
Endosulfan I	EPA 8081B
	EPA 608.3

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Chlorinated Hydrocarbon Pesticides

Endosulfan II	EPA 8081B
	EPA 608.3
Endosulfan sulfate	EPA 8081B
	EPA 608.3
Endrin	EPA 8081B
	EPA 608.3
Endrin aldehyde	EPA 8081B
	EPA 608.3
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
	EPA 608.3
Heptachlor	EPA 8081B
	EPA 608.3
Heptachlor epoxide	EPA 8081B
	EPA 608.3
Lindane	EPA 8081B
	EPA 608.3
Methoxychlor	EPA 8081B
	EPA 608.3
Mirex	EPA 608.3
PCNB	EPA 8270E
Toxaphene	EPA 8081B
	EPA 608.3

Chlorinated Hydrocarbons

1,2,3-Trichlorobenzene	EPA 8260D
1,2,4,5-Tetrachlorobenzene	EPA 8270E
1,2,4-Trichlorobenzene	EPA 625.1

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Chlorinated Hydrocarbons

1,2,4-Trichlorobenzene	EPA 8270E
2-Chloronaphthalene	EPA 625.1
	EPA 8270E
Hexachlorobenzene	EPA 625.1
	EPA 8270E
Hexachlorobutadiene	EPA 625.1
	EPA 8270E
Hexachlorocyclopentadiene	EPA 625.1
	EPA 8270E
Hexachloroethane	EPA 625.1
	EPA 8270E

Chlorophenoxy Acid Pesticides

2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A
Dichloroprop	EPA 8151A
Dinoseb	EPA 8151A

Demand

Biochemical Oxygen Demand	SM 5210B-2016
Carbonaceous BOD	SM 5210B-2016
Chemical Oxygen Demand	EPA 410.4, Rev. 2.0 (1993)
	SM 5220D-2011

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Fuel Oxygenates

Di-isopropyl ether	EPA 8260D
Ethanol	EPA 8260D
	EPA 624.1
Methyl tert-butyl ether	EPA 8260D
	EPA 624.1
tert-amyl methyl ether (TAME)	EPA 8260D
tert-butyl alcohol	EPA 8260D
	EPA 624.1
tert-butyl ethyl ether (ETBE)	EPA 8260D

Haloethers

2,2'-Oxybis(1-chloropropane)	EPA 625.1
	EPA 8270E
4-Bromophenylphenyl ether	EPA 625.1
	EPA 8270E
4-Chlorophenylphenyl ether	EPA 625.1
	EPA 8270E
Bis(2-chloroethoxy)methane	EPA 625.1
	EPA 8270E
Bis(2-chloroethyl)ether	EPA 625.1
	EPA 8270E

Low Level Halocarbons

1,2,3-Trichloropropane, Low Level	EPA 8011
1,2-Dibromo-3-chloropropane, Low Le	EPA 8011
1,2-Dibromoethane, Low Level	EPA 8011

Low Level Polynuclear Aromatics

Acenaphthene Low Level	EPA 8270E SIM

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All approved analytes are listed below:

Low Level Polynuclear Aromatics

Acenaphthylene Low Level	EPA 8270E SIM
Anthracene Low Level	EPA 8270E SIM
Benzo(a)anthracene Low Level	EPA 8270E SIM
Benzo(a)pyrene Low Level	EPA 8270E SIM
Benzo(b)fluoranthene Low Level	EPA 8270E SIM
Benzo(g,h,i)perylene Low Level	EPA 8270E SIM
Benzo(k)fluoranthene Low Level	EPA 8270E SIM
Chrysene Low Level	EPA 8270E SIM
Dibenzo(a,h)anthracene Low Level	EPA 8270E SIM
Fluoranthene Low Level	EPA 8270E SIM
Fluorene Low Level	EPA 8270E SIM
Indeno(1,2,3-cd)pyrene Low Level	EPA 8270E SIM
Naphthalene Low Level	EPA 8270E SIM
Phenanthrene Low Level	EPA 8270E SIM
Pyrene Low Level	EPA 8270E SIM

Metals I

Iron, Total	SM 3500-Fe B-2011

Metals II

Chromium VI	EPA 7196A
	SM 3500-Cr B-2011

Mineral

Acidity	SM 2310B-2011
Alkalinity	SM 2320B-2011
Chloride	EPA 300.0, Rev. 2.1 (1993)
	SM 4500-Cl- E-2011
	EPA 9056A

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All approved analytes are listed below:

Mineral

Fluoride, Total	EPA 300.0, Rev. 2.1 (1993)
	SM 4500-F- C-2011
	EPA 9056A
Sulfate (as SO4)	EPA 300.0, Rev. 2.1 (1993)
	SM 4500-SO4- E-2011
	EPA 9056A

Miscellaneous

Bromide	EPA 300.0, Rev. 2.1 (1993)
Color	SM 2120B-2011
Cyanide, Total	LACHAT QuikChem 10-204-00-1-X
	EPA 9014
	SM 4500-CN E-2016
	EPA 9012B
Formaldehyde	EPA 8315A
non-Polar Extractable Material (TPH)	EPA 1664B
Oil and Grease Total Recoverable	EPA 1664B
Organic Carbon, Total	SM 5310C-2014
	EPA 9060A
Phenols	EPA 420.1 (Rev. 1978)
	EPA 9065
Specific Conductance	EPA 120.1 (Rev. 1982)
	SM 2510B-2011
	EPA 9050A
Sulfide (as S)	SM 4500-S2- D-2011
Surfactant (MBAS)	SM 5540C-2011
Turbidity	SM 2130 B-2011
	EPA 180.1, Rev. 2.0 (1993)

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All approved analytes are listed below:

Nitroaromatics and Isophorone

1,3-Dinitrobenzene	EPA 8270E
2,4-Dinitrotoluene	EPA 625.1
	EPA 8270E
2,6-Dinitrotoluene	EPA 625.1
	EPA 8270E
Isophorone	EPA 625.1
	EPA 8270E
Nitrobenzene	EPA 625.1
	EPA 8270E

Nitrosoamines

N-Nitrosodimethylamine	EPA 625.1
	EPA 8270E
N-Nitrosodi-n-propylamine	EPA 625.1
	EPA 8270E
N-Nitrosodiphenylamine	EPA 625.1
	EPA 8270E

Nutrient

Ammonia (as N)	SM 4500-NH3 H-2011
	EPA 350.1, Rev. 2.0 (1993)
Kjeldahl Nitrogen, Total	EPA 351.1 (Rev. 1978)
	SM 4500-NH3 H-2011
Nitrate (as N)	EPA 353.2, Rev. 2.0 (1993)
	EPA 300.0, Rev. 2.1 (1993)
	SM 4500-NO3 F-2016
	EPA 9056A
Nitrate-Nitrite (as N)	EPA 353.2, Rev. 2.0 (1993)

Serial No.: 68690

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. MARCO SOARES
ALPHA ANALYTICAL LLC
8 WALKUP DR
WESTBOROUGH, MA 01581-1019

NY Lab Id No: 11148

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES NON POTABLE WATER
All approved analytes are listed below:

Nutrient	
Nitrate-Nitrite (as N)	SM 4500-NO3 F-2016
Nitrite (as N)	EPA 353.2, Rev. 2.0 (1993)
	SM 4500-NO3 F-2016
	SM 4500-NO2 B-2011
Orthophosphate (as P)	SM 4500-P E-2011
Phosphorus, Total	SM 4500-P E-2011
Organophosphate Pesticides	
Atrazine	EPA 625.1
	EPA 8270E
Parathion ethyl	EPA 8270E
Thionazin	EPA 8270E
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015D
Gasoline Range Organics	EPA 8015D
Phthalate Esters	
Benzyl butyl phthalate	EPA 625.1
	EPA 8270E
Bis(2-ethylhexyl) phthalate	EPA 625.1
	EPA 8270E
Diethyl phthalate	EPA 625.1
	EPA 8270E
Dimethyl phthalate	EPA 625.1
	EPA 8270E
Di-n-butyl phthalate	EPA 625.1
	EPA 8270E
Di-n-octyl phthalate	EPA 625.1

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Phthalate Esters	
Di-n-octyl phthalate	EPA 8270E
Polychlorinated Biphenyls	
Aroclor 1016 (PCB-1016)	EPA 8082A
	EPA 608.3
Aroclor 1221 (PCB-1221)	EPA 8082A
	EPA 608.3
Aroclor 1232 (PCB-1232)	EPA 8082A
	EPA 608.3
Aroclor 1242 (PCB-1242)	EPA 8082A
	EPA 608.3
Aroclor 1248 (PCB-1248)	EPA 8082A
	EPA 608.3
Aroclor 1254 (PCB-1254)	EPA 8082A
	EPA 608.3
Aroclor 1260 (PCB-1260)	EPA 8082A
	EPA 608.3
Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A
Polynuclear Aromatics	
Acenaphthene	EPA 625.1
	EPA 8270E
Acenaphthylene	EPA 625.1
	EPA 8270E
Anthracene	EPA 625.1
	EPA 8270E
Benzo(a)anthracene	EPA 625.1

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Polynuclear Aromatics

Benzo(a)anthracene	EPA 8270E
Benzo(a)pyrene	EPA 625.1
	EPA 8270E
Benzo(b)fluoranthene	EPA 625.1
	EPA 8270E
Benzo(g,h,i)perylene	EPA 625.1
	EPA 8270E
Benzo(k)fluoranthene	EPA 625.1
	EPA 8270E
Chrysene	EPA 625.1
	EPA 8270E
Dibenzo(a,h)anthracene	EPA 625.1
	EPA 8270E
Fluoranthene	EPA 625.1
	EPA 8270E
Fluorene	EPA 625.1
	EPA 8270E
Indeno(1,2,3-cd)pyrene	EPA 625.1
	EPA 8270E
Naphthalene	EPA 625.1
	EPA 8270E
Phenanthrene	EPA 625.1
	EPA 8270E
Pyrene	EPA 625.1
	EPA 8270E

Priority Pollutant Phenols

2,3,4,6 Tetrachlorophenol	EPA 8270E

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Priority Pollutant Phenols

2,4,5-Trichlorophenol	EPA 625.1
	EPA 8270E
2,4,6-Trichlorophenol	EPA 625.1
	EPA 8270E
2,4-Dichlorophenol	EPA 625.1
	EPA 8270E
2,4-Dimethylphenol	EPA 625.1
	EPA 8270E
2,4-Dinitrophenol	EPA 625.1
	EPA 8270E
2-Chlorophenol	EPA 625.1
	EPA 8270E
2-Methyl-4,6-dinitrophenol	EPA 625.1
	EPA 8270E
2-Methylphenol	EPA 625.1
	EPA 8270E
2-Nitrophenol	EPA 625.1
	EPA 8270E
3-Methylphenol	EPA 625.1
	EPA 8270E
4-Chloro-3-methylphenol	EPA 625.1
	EPA 8270E
4-Methylphenol	EPA 625.1
	EPA 8270E
4-Nitrophenol	EPA 625.1
	EPA 8270E
Cresols, Total	EPA 8270E

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Priority Pollutant Phenols

Pentachlorophenol	EPA 625.1
	EPA 8270E
Phenol	EPA 625.1
	EPA 8270E

Residue

Settleable Solids	SM 2540 F-2015
Solids, Total	SM 2540 B-2015
Solids, Total Dissolved	SM 2540 C-2015
Solids, Total Suspended	SM 2540 D-2015
Solids, Volatile	SM 2540 E-2015

Semi-Volatile Organics

1,1'-Biphenyl	EPA 8270E
1,2-Dichlorobenzene, Semi-volatile	EPA 8270E
1,3-Dichlorobenzene, Semi-volatile	EPA 8270E
1,4-Dichlorobenzene, Semi-volatile	EPA 8270E
2-Methylnaphthalene	EPA 625.1
	EPA 8270E
Acetophenone	EPA 625.1
	EPA 8270E
Benzaldehyde	EPA 8270E
Benzoic Acid	EPA 8270E
Benzyl alcohol	EPA 8270E
Caprolactam	EPA 8270E
Dibenzofuran	EPA 8270E
n-Decane	EPA 625.1
n-Octadecane	EPA 625.1

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Volatile Aromatics

1,2,4-Trichlorobenzene, Volatile	EPA 8260D
1,2,4-Trimethylbenzene	EPA 8260D
1,2-Dichlorobenzene	EPA 8260D
	EPA 624.1
1,3,5-Trimethylbenzene	EPA 8260D
1,3-Dichlorobenzene	EPA 8260D
	EPA 624.1
1,4-Dichlorobenzene	EPA 8260D
	EPA 624.1
2-Chlorotoluene	EPA 8260D
4-Chlorotoluene	EPA 8260D
Benzene	EPA 8260D
	EPA 624.1
Bromobenzene	EPA 8260D
Chlorobenzene	EPA 8260D
	EPA 624.1
Ethyl benzene	EPA 8260D
	EPA 624.1
Isopropylbenzene	EPA 8260D
m/p-Xylenes	EPA 8260D
Naphthalene, Volatile	EPA 8260D
n-Butylbenzene	EPA 8260D
n-Propylbenzene	EPA 8260D
o-Xylene	EPA 8260D
p-Isopropyltoluene (P-Cymene)	EPA 8260D
sec-Butylbenzene	EPA 8260D
Styrene	EPA 8260D

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Volatile Aromatics

Styrene	EPA 624.1
tert-Butylbenzene	EPA 8260D
Toluene	EPA 8260D
	EPA 624.1
Total Xylenes	EPA 8260D
	EPA 624.1

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 8260D
1,1,1-Trichloroethane	EPA 8260D
	EPA 624.1
1,1,2,2-Tetrachloroethane	EPA 8260D
	EPA 624.1
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260D
	EPA 624.1
1,1,2-Trichloroethane	EPA 8260D
	EPA 624.1
1,1-Dichloroethane	EPA 8260D
	EPA 624.1
1,1-Dichloroethene	EPA 8260D
	EPA 624.1
1,1-Dichloropropene	EPA 8260D
1,2,3-Trichloropropane	EPA 8260D
1,2-Dibromo-3-chloropropane	EPA 8260D
1,2-Dibromoethane	EPA 8260D
1,2-Dichloroethane	EPA 8260D
	EPA 624.1
1,2-Dichloropropane	EPA 8260D

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Volatile Halocarbons

1,2-Dichloropropane	EPA 624.1
1,3-Dichloropropane	EPA 8260D
2,2-Dichloropropane	EPA 8260D
2-Chloroethylvinyl ether	EPA 8260D
	EPA 624.1
Bromochloromethane	EPA 8260D
Bromodichloromethane	EPA 8260D
	EPA 624.1
Bromoform	EPA 8260D
	EPA 624.1
Bromomethane	EPA 8260D
	EPA 624.1
Carbon tetrachloride	EPA 8260D
	EPA 624.1
Chloroethane	EPA 8260D
	EPA 624.1
Chloroform	EPA 8260D
	EPA 624.1
Chloromethane	EPA 8260D
	EPA 624.1
cis-1,2-Dichloroethene	EPA 8260D
	EPA 624.1
cis-1,3-Dichloropropene	EPA 8260D
	EPA 624.1
Dibromochloromethane	EPA 8260D
	EPA 624.1
Dibromomethane	EPA 8260D

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Volatile Halocarbons

Dichlorodifluoromethane	EPA 8260D
	EPA 624.1
Hexachlorobutadiene, Volatile	EPA 8260D
Methyl iodide	EPA 8260D
Methylene chloride	EPA 8260D
	EPA 624.1
Tetrachloroethene	EPA 8260D
	EPA 624.1
trans-1,2-Dichloroethene	EPA 8260D
	EPA 624.1
trans-1,3-Dichloropropene	EPA 8260D
	EPA 624.1
trans-1,4-Dichloro-2-butene	EPA 8260D
Trichloroethene	EPA 8260D
	EPA 624.1
Trichlorofluoromethane	EPA 8260D
	EPA 624.1
Vinyl chloride	EPA 8260D
	EPA 624.1

Volatiles Organics

1,4-Dioxane	EPA 8260D
	EPA 8260D SIM
2-Butanone (Methylethyl ketone)	EPA 8260D
2-Hexanone	EPA 8260D
4-Methyl-2-Pentanone	EPA 8260D
	EPA 624.1
Acetone	EPA 8260D

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Volatiles Organics

Acetone	EPA 624.1
Carbon Disulfide	EPA 8260D
Cyclohexane	EPA 8260D
Di-ethyl ether	EPA 8260D
Ethyl Acetate	EPA 8260D
Hexane	EPA 8260D
Isopropanol	EPA 8260D
Methyl acetate	EPA 8260D
Methyl cyclohexane	EPA 8260D
n-Butanol	EPA 8260D
o-Toluidine	EPA 8270E
Tetrahydrofuran	EPA 8260D
Vinyl acetate	EPA 8260D
	EPA 624.1

Sample Preparation Methods

SM 4500-P B(5)-2011
EPA 5030C
SM 4500-CN B-2016 and C-2016
EPA 9030B
EPA 3510C
SM 4500-NH3 B-2011
SM 4500-F B-2011
SM 4500-N Org B-2011 or C-2011
EPA 9010C

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All approved analytes are listed below:

Acrylates

Acrolein (Propenal)	EPA 8260D
Acrylonitrile	EPA 8260D
Ethyl methacrylate	EPA 8260D
Methyl methacrylate	EPA 8260D

Amines

1,2-Diphenylhydrazine	EPA 8270E
2-Nitroaniline	EPA 8270E
3-Nitroaniline	EPA 8270E
4-Chloroaniline	EPA 8270E
4-Nitroaniline	EPA 8270E
Aniline	EPA 8270E
Carbazole	EPA 8270E
Diphenylamine	EPA 8270E

Benzidines

3,3'-Dichlorobenzidine	EPA 8270E
Benzidine	EPA 8270E

Characteristic Testing

Corrosivity (pH)	EPA 9040C
	EPA 9045D
Free Liquids	EPA 9095B
Ignitability	EPA 1030
	EPA 1010A
Synthetic Precipitation Leaching Proc.	EPA 1312
TCLP	EPA 1311

Chlorinated Hydrocarbon Pesticides

4,4'-DDD	EPA 8081B

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Chlorinated Hydrocarbon Pesticides

4,4'-DDE	EPA 8081B
4,4'-DDT	EPA 8081B
Aldrin	EPA 8081B
alpha-BHC	EPA 8081B
alpha-Chlordane	EPA 8081B
Atrazine	EPA 8270E
beta-BHC	EPA 8081B
Chlordane Total	EPA 8081B
delta-BHC	EPA 8081B
Dieldrin	EPA 8081B
Endosulfan I	EPA 8081B
Endosulfan II	EPA 8081B
Endosulfan sulfate	EPA 8081B
Endrin	EPA 8081B
Endrin aldehyde	EPA 8081B
Endrin Ketone	EPA 8081B
gamma-Chlordane	EPA 8081B
Heptachlor	EPA 8081B
Heptachlor epoxide	EPA 8081B
Lindane	EPA 8081B
Methoxychlor	EPA 8081B
Pentachloronitrobenzene	EPA 8270E
Toxaphene	EPA 8081B

Chlorinated Hydrocarbons

1,2,3-Trichlorobenzene	EPA 8260D
1,2,4,5-Tetrachlorobenzene	EPA 8270E
1,2,4-Trichlorobenzene	EPA 8270E

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Chlorinated Hydrocarbons

2-Chloronaphthalene	EPA 8270E
Hexachlorobenzene	EPA 8270E
Hexachlorobutadiene	EPA 8270E
Hexachlorocyclopentadiene	EPA 8270E
Hexachloroethane	EPA 8260D
	EPA 8270E

Chlorophenoxy Acid Pesticides

2,4,5-T	EPA 8151A
2,4,5-TP (Silvex)	EPA 8151A
2,4-D	EPA 8151A
2,4-DB	EPA 8151A
Dalapon	EPA 8151A
Dicamba	EPA 8151A
Dichloroprop	EPA 8151A
MCPA	EPA 8151A
MCPP	EPA 8151A

Haloethers

2,2'-Oxybis(1-chloropropane)	EPA 8270E
4-Bromophenylphenyl ether	EPA 8270E
4-Chlorophenylphenyl ether	EPA 8270E
Bis(2-chloroethoxy)methane	EPA 8270E
Bis(2-chloroethyl)ether	EPA 8270E

Low Level Polynuclear Aromatic Hydrocarbons

Acenaphthene Low Level	EPA 8270E SIM
Acenaphthylene Low Level	EPA 8270E SIM
Anthracene Low Level	EPA 8270E SIM

Serial No.: 68691

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NEW YORK STATE DEPARTMENT OF HEALTH
WADSWORTH CENTER

Expires 12:01 AM April 01, 2025
Issued April 01, 2024

CERTIFICATE OF APPROVAL FOR LABORATORY SERVICE

Issued in accordance with and pursuant to section 502 Public Health Law of New York State

MR. MARCO SOARES
ALPHA ANALYTICAL LLC
8 WALKUP DR
WESTBOROUGH, MA 01581-1019

NY Lab Id No: 11148

is hereby APPROVED as an Environmental Laboratory in conformance with the National Environmental Laboratory Accreditation Conference Standards (2016) for the category ENVIRONMENTAL ANALYSES SOLID AND HAZARDOUS WASTE
All approved analytes are listed below:

Low Level Polynuclear Aromatic Hydrocarbons

Benzo(a)anthracene Low Level	EPA 8270E SIM
Benzo(a)pyrene Low Level	EPA 8270E SIM
Benzo(b)fluoranthene Low Level	EPA 8270E SIM
Benzo(g,h,i)perylene Low Level	EPA 8270E SIM
Benzo(k)fluoranthene Low Level	EPA 8270E SIM
Chrysene Low Level	EPA 8270E SIM
Dibenzo(a,h)anthracene Low Level	EPA 8270E SIM
Fluoranthene Low Level	EPA 8270E SIM
Fluorene Low Level	EPA 8270E SIM
Indeno(1,2,3-cd)pyrene Low Level	EPA 8270E SIM
Naphthalene Low Level	EPA 8270E SIM
Phenanthrene Low Level	EPA 8270E SIM
Pyrene Low Level	EPA 8270E SIM

Metals II

Chromium VI	EPA 7196A

Minerals

Chloride	EPA 9251
Sulfate (as SO4)	EPA 9038

Miscellaneous

Cyanide, Total	EPA 9014
	EPA 9012B
Phenols	EPA 9065
Specific Conductance	EPA 9050A

Nitroaromatics and Isophorone

2,4-Dinitrotoluene	EPA 8270E
2,6-Dinitrotoluene	EPA 8270E

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Nitroaromatics and Isophorone	
Isophorone	EPA 8270E
Nitrobenzene	EPA 8260D
	EPA 8270E
Pyridine	EPA 8270E
Nitrosoamines	
N-Nitrosodimethylamine	EPA 8270E
N-Nitrosodi-n-propylamine	EPA 8270E
N-Nitrosodiphenylamine	EPA 8270E
Organophosphate Pesticides	
Parathion ethyl	EPA 8270E
Petroleum Hydrocarbons	
Diesel Range Organics	EPA 8015D
Gasoline Range Organics	EPA 8015D
Oil and Grease Total Recoverable	EPA 9071B (Solvent:Hexane)
Phthalate Esters	
Benzyl butyl phthalate	EPA 8270E
Bis(2-ethylhexyl) phthalate	EPA 8270E
Diethyl phthalate	EPA 8270E
Dimethyl phthalate	EPA 8270E
Di-n-butyl phthalate	EPA 8270E
Di-n-octyl phthalate	EPA 8270E
Polychlorinated Biphenyls	
Aroclor 1016 (PCB-1016)	EPA 8082A
Aroclor 1016 (PCB-1016) in Oil	EPA 8082A
Aroclor 1221 (PCB-1221)	EPA 8082A

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Polychlorinated Biphenyls

Aroclor 1221 (PCB-1221) in Oil	EPA 8082A
Aroclor 1232 (PCB-1232)	EPA 8082A
Aroclor 1232 (PCB-1232) in Oil	EPA 8082A
Aroclor 1242 (PCB-1242)	EPA 8082A
Aroclor 1242 (PCB-1242) in Oil	EPA 8082A
Aroclor 1248 (PCB-1248)	EPA 8082A
Aroclor 1248 (PCB-1248) in Oil	EPA 8082A
Aroclor 1254 (PCB-1254)	EPA 8082A
Aroclor 1254 (PCB-1254) in Oil	EPA 8082A
Aroclor 1260 (PCB-1260)	EPA 8082A
Aroclor 1260 (PCB-1260) in Oil	EPA 8082A
Aroclor 1262 (PCB-1262)	EPA 8082A
Aroclor 1262 (PCB-1262) in Oil	EPA 8082A
Aroclor 1268 (PCB-1268)	EPA 8082A
Aroclor 1268 (PCB-1268) in Oil	EPA 8082A

Polynuclear Aromatic Hydrocarbons

Acenaphthene	EPA 8270E
Acenaphthylene	EPA 8270E
Anthracene	EPA 8270E
Benzo(a)anthracene	EPA 8270E
Benzo(a)pyrene	EPA 8270E
Benzo(b)fluoranthene	EPA 8270E
Benzo(g,h,i)perylene	EPA 8270E
Benzo(k)fluoranthene	EPA 8270E
Chrysene	EPA 8270E
Dibenzo(a,h)anthracene	EPA 8270E
Fluoranthene	EPA 8270E

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Polynuclear Aromatic Hydrocarbons

Fluorene	EPA 8270E
Indeno(1,2,3-cd)pyrene	EPA 8270E
Naphthalene	EPA 8270E
Phenanthrene	EPA 8270E
Pyrene	EPA 8270E

Priority Pollutant Phenols

2,3,4,6 Tetrachlorophenol	EPA 8270E
2,4,5-Trichlorophenol	EPA 8270E
2,4,6-Trichlorophenol	EPA 8270E
2,4-Dichlorophenol	EPA 8270E
2,4-Dimethylphenol	EPA 8270E
2,4-Dinitrophenol	EPA 8270E
2-Chlorophenol	EPA 8270E
2-Methyl-4,6-dinitrophenol	EPA 8270E
2-Methylphenol	EPA 8270E
2-Nitrophenol	EPA 8270E
3-Methylphenol	EPA 8270E
4-Chloro-3-methylphenol	EPA 8270E
4-Methylphenol	EPA 8270E
4-Nitrophenol	EPA 8270E
Pentachlorophenol	EPA 8270E
Phenol	EPA 8270E

Semi-Volatile Organics

1,1'-Biphenyl	EPA 8270E
1,2-Dichlorobenzene, Semi-volatile	EPA 8270E
1,3-Dichlorobenzene, Semi-volatile	EPA 8270E

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Semi-Volatile Organics

1,4-Dichlorobenzene, Semi-volatile	EPA 8270E
2-Methylnaphthalene	EPA 8270E
Acetophenone	EPA 8270E
Benzaldehyde	EPA 8270E
Benzoic Acid	EPA 8270E
Benzyl alcohol	EPA 8270E
Caprolactam	EPA 8270E
Dibenzofuran	EPA 8270E

Volatile Aromatics

1,2,4-Trichlorobenzene, Volatile	EPA 8260D
1,2,4-Trimethylbenzene	EPA 8260D
1,2-Dichlorobenzene	EPA 8260D
1,3,5-Trimethylbenzene	EPA 8260D
1,3-Dichlorobenzene	EPA 8260D
1,4-Dichlorobenzene	EPA 8260D
2-Chlorotoluene	EPA 8260D
4-Chlorotoluene	EPA 8260D
Benzene	EPA 8260D
Bromobenzene	EPA 8260D
Chlorobenzene	EPA 8260D
Ethyl benzene	EPA 8260D
Isopropylbenzene	EPA 8260D
m/p-Xylenes	EPA 8260D
Naphthalene, Volatile	EPA 8260D
n-Butylbenzene	EPA 8260D
n-Propylbenzene	EPA 8260D
o-Xylene	EPA 8260D

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Volatile Aromatics

p-Isopropyltoluene (P-Cymene)	EPA 8260D
sec-Butylbenzene	EPA 8260D
Styrene	EPA 8260D
tert-Butylbenzene	EPA 8260D
Toluene	EPA 8260D
Total Xylenes	EPA 8260D

Volatile Halocarbons

1,1,1,2-Tetrachloroethane	EPA 8260D
1,1,1-Trichloroethane	EPA 8260D
1,1,2,2-Tetrachloroethane	EPA 8260D
1,1,2-Trichloro-1,2,2-Trifluoroethane	EPA 8260D
1,1,2-Trichloroethane	EPA 8260D
1,1-Dichloroethane	EPA 8260D
1,1-Dichloroethene	EPA 8260D
1,1-Dichloropropene	EPA 8260D
1,2,3-Trichloropropane	EPA 8260D
1,2-Dibromo-3-chloropropane	EPA 8260D
1,2-Dibromoethane	EPA 8260D
1,2-Dichloroethane	EPA 8260D
1,2-Dichloropropane	EPA 8260D
1,3-Dichloropropane	EPA 8260D
2,2-Dichloropropane	EPA 8260D
2-Chloroethylvinyl ether	EPA 8260D
3-Chloropropene (Allyl chloride)	EPA 8260D
Bromochloromethane	EPA 8260D
Bromodichloromethane	EPA 8260D
Bromoform	EPA 8260D

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Volatile Halocarbons

Bromomethane	EPA 8260D
Carbon tetrachloride	EPA 8260D
Chloroethane	EPA 8260D
Chloroform	EPA 8260D
Chloromethane	EPA 8260D
cis-1,2-Dichloroethene	EPA 8260D
cis-1,3-Dichloropropene	EPA 8260D
Dibromochloromethane	EPA 8260D
Dibromomethane	EPA 8260D
Dichlorodifluoromethane	EPA 8260D
Hexachlorobutadiene, Volatile	EPA 8260D
Methyl iodide	EPA 8260D
Methylene chloride	EPA 8260D
Tetrachloroethene	EPA 8260D
trans-1,2-Dichloroethene	EPA 8260D
trans-1,3-Dichloropropene	EPA 8260D
trans-1,4-Dichloro-2-butene	EPA 8260D
Trichloroethene	EPA 8260D
Trichlorofluoromethane	EPA 8260D
Vinyl chloride	EPA 8260D

Volatile Organics

1,4-Dioxane	EPA 8260D
	EPA 8270E
2-Butanone (Methylethyl ketone)	EPA 8260D
2-Hexanone	EPA 8260D
2-Nitropropane	EPA 8260D
4-Methyl-2-Pentanone	EPA 8260D

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Volatile Organics

Acetone	EPA 8260D
Carbon Disulfide	EPA 8260D
Cyclohexane	EPA 8260D
Di-ethyl ether	EPA 8260D
Ethyl Acetate	EPA 8260D
Hexane	EPA 8260D
Methyl acetate	EPA 8260D
Methyl cyclohexane	EPA 8260D
Methyl tert-butyl ether	EPA 8260D
n-Butanol	EPA 8260D
tert-butyl alcohol	EPA 8260D
Tetrahydrofuran	EPA 8260D
Vinyl acetate	EPA 8260D

Sample Preparation Methods

EPA 5035A-L
EPA 5035A-H
EPA 3580A
EPA 3540C
EPA 3546
EPA 3060A
EPA 9010C

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Felicia Grogan Approved on 3/8/2023 1:18:34 PM
Rusty Vicinie Approved on 3/19/2023 6:25:14 PM
Stephanie Atkins Approved on 3/30/2023 11:41:31 AM
Jacob Cottrell Approved on 3/30/2023 2:40:44 PM

Title Page

Quality Manual
Pace Analytical Services, LLC

Prepared for:

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Pace® Analytical Services, LLC
816 Durst Avenue
Greenwood, SC 29649
Phone: 864-229-4413

Signatory Attestation: I attest the application of my electronic signature on this title page affirms my management commitment and responsibility to uphold the requirements of the PAS Quality Management System (QMS) described in this Quality Manual (manual) at each location for which this manual is prepared.

Refer to the Quality Manual Signatory Page to view the job title and physical address for each signatory.

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Quality Manual Approval Signatories - Asheville

The following individuals represent the PAS corporate and local management team responsible for implementing the PAS Quality Management System (QMS) and upholding the requirements of this manual at the location(s) for which this manual was prepared, at the time this version of the manual was made effective, and that correlate with the electronic signatures shown on the title page of this manual.

If these persons(s) change positions, leave the company, or are on extended leave of absence, the approval of this manual automatically transfers to the person replacing the signatory or to the signatory's primary or alternate deputy until the manager is replaced and/or the manager returns to work. The individual replacing the signatory automatically accepts the responsibilities associated with the original signatory's attestation. Refer to Section 4.1.5.1.1 of this manual for the deputies assigned to key personnel job titles.

The manual is not revised and released under an updated version for the sole purpose of updating personnel change(s). Personnel information is updated when the next revision of the manual is released. See manual Sections 1.2.1 and 1.2.2 for more information about how this manual is maintained.

Name	Job Title	Address, City, State, ZIP	Phone
Rusty Vicinie	Vice President of Operations	1638 Roseytown Rd, Suite 2, 3, 4 Greensburg, PA 15601	724-850-5600
Felicia Grogan	Director of Lab Operations	9800 Kincey Avenue, Suite 100 Huntersville, NC 28078	704-977-0955
Stephanie Atkins	Quality Program Manager	106 Vantage Point Drive, West Columbia, SC 29172	612-723-8459
Jacob Cottrell	Quality Manager	2225 Riverside Drive, Asheville, NC 28804	828-417-6052

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Quality Manual Approval Signatories - Eden

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Jacob Cottrell	Quality Manager	2225 Riverside Drive, Asheville, NC 28804	828-417-6052

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Quality Manual Approval Signatories - Raleigh

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Quality Manual Approval Signatories - Greenwood

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Jacob Cottrell	Quality Manager	2225 Riverside Drive, Asheville, NC 28804	828-417-6052

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1.0 PURPOSE AND SCOPE

1.1 Purpose

This quality manual (manual) outlines the quality management system (QMS) and management structure of Pace® Analytical Services, LLC. Pace® Analytical Services, LLC is referred to by brand name Pace® Analytical Services and by the acronyms PAS or ENV. The acronyms PAS and ENV are interchangeable.

The PAS QMS is also referred to as the quality program throughout this manual and other PAS documents. The phrases "quality management system" and "quality program" are synonymous and are referred to by the acronym QMS.

The QMS is the collection of policies and processes established by the senior leaders of PAS (top management) to ensure the service and products provided by PAS consistently meet relevant requirements and achieves the goal of Pace® to provide customers with high quality, cost-effective, analytical measurements, and services.

The QMS is also planned to establish conformance[1] and compliance with the current published versions of the following international and national quality system standards:

- ISO/IEC 17025: *General requirements for the competence of testing and calibration laboratories*
- NELAC/TNI Standard Volume 1: *Management and Technical Requirements for Laboratories Performing Environmental Analysis*

[1]The statement of conformity to these Standards pertains only to testing and sampling activities carried out by the laboratory at its physical address, in temporary or mobile facilities, in-network, or by laboratory personnel at a customer's facility.

In addition to the international and national standards, the QMS is planned to achieve regulatory compliance with the various federal and state programs for which PAS locations provide compliance testing and/or holds certification or accreditation. Federal or state requirements that do not apply to all PAS locations, are provided in addendum to this manual or in other documents that supplement the manual. Customer-specific project and program requirements are not included in the manual in order to maintain client confidentiality.

- A list of accreditation and certifications held by each location associated with this manual is provided in Appendix A.
- A list of analytical testing capabilities offered by each location associated with this manual is provided in Appendix B.

1.2 Scope and Application

This manual applies to each location listed on the Title Page of this manual, including PAS laboratories, satellite laboratories, service centers, and supporting business functions.

For purposes of the PAS QMS:

- The term "location" used in this manual refers to laboratories and/or service centers.
- The term "laboratory" refers to any PAS location, however named by Pace® that provides testing, collects samples (sampling), or conducts field measurement services in a fixed building, mobile unit, or in-situ (field).

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- The phrase "service center" refers to any PAS location, however named by Pace® that does not perform any testing, sampling, or field measurements.
- The phrase "satellite laboratory" refers to a limited-service laboratory affiliated to a larger business unit or location. Some PAS business groups, such as accounting, may refer to a satellite laboratory as a "service center." Irrespective of internal jargon or reference by any group, any PAS location that generates a test result for external use is a "laboratory" and must comply with the requirements specified in this manual for all analytical testing services.

PAS locations are defined by physical address. Laboratories are defined by physical address and certification/accreditation ID except mobile units which may be defined by the address of the location to which they are assigned, by VIN (vehicle identification number), or by certification/accreditation ID. Laboratories that provide sampling and field testing are defined by the physical address of the PAS location to which they are affiliated and that manages these activities.

1.2.1 Quality Manual Template

This manual was prepared using the PAS Quality Manual Template (template) created by the PAS Corporate Quality Director (CQD).

The template, known as document ID ENV-TMP-CORQ-0007, specifies the minimum requirements that every PAS location must abide by, regardless of scope of services or number of personnel, to maintain a quality program that achieves the objectives of the PAS Quality Policy (See Section 4.2.2).

The template is the mechanism used by top management to communicate to PAS personnel their commitment to continuously develop and improve the QMS for effectiveness, to meet customer expectations, and to comply with any statutory and regulatory requirements. Their signature of approval on this template is the mechanism used to document this responsibility.

"Top Management" is the phrase used by the TNI Standard to refer to the leaders of an organization that develop and/or release the PAS Quality Policy Statement and QMS under their authority

For PAS, these managers include the Chief Executive Officer (CEO) and Chief Compliance Officer (CCO) of Pace® and the President, CQD, Senior Vice President of Operations (Sr. VPO), and the Chief Technical Officer (CTO) of PAS.

The template and instructions for use of the template are released by corporate quality personnel to local quality managers responsible for each location (Local QM). The local QM uses the template to prepare the location manual by following the instructions provided to them. The local QM may not alter the font, structure, or content of the template, except where specified by instruction to do so. As previously stated, program specific requirements unique to each location are provided in addendum or in documents that supplement the manual.

The template is reviewed by corporate quality personnel annually and updated, if needed. More frequent review and revision may occur to manage change, to maintain conformance and compliance to relevant standards or to improve the QMS.

See standard operating procedure (SOP) ENV-SOP-CORQ-00015 *Document Management and Control* for more information

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1.2.2 Quality Manual

The quality manual is created from template ENV-TMP-CORQ-0007 by local quality personnel, who are also responsible for maintenance and management of the document.

- PAS locations are not permitted to alter content of the template when preparing their manual, except where specified in the template. Control of content in the manual is necessary to ensure consistency of implementation of the PAS quality program across the network.
- If additions or changes to the manual are needed to maintain regulatory compliance or conformance to relevant standards and these changes cannot be covered by addendum to the manual, the need for change must be raised to the PAS Corporate Quality Director, who will decide how to resolve the need.

The manual is approved for release by the management team listed on the Quality Manual Approval Signatory Page. The manager's electronic signature on the Title Page of the manual affirms their commitment to implement and uphold the requirements, processes, and procedures of the PAS QMS at each location for which the manual was prepared.

The manual is reviewed annually and updated with each release of a new version of the template, and as needed to update appendices and addendum. More frequent review and revision may be necessary when there are significant changes to the capabilities, and resources of the laboratory during the calendar year

See SOP ENV-SOP-CORQ-00015 *Document Management and Control* for more information.

1.2.3 References to Supporting Documents

The template and the manual include references to other organization documents that support the QMS such as policies and standard operating procedures (SOPs).

These references may include the document's document control number (DC#) and the document title. This information is subject to change at the discretion of PAS. The manual and/or template are updated to reflect the editorial change during the manual's next scheduled review/revision cycle or the next time a version of the manual is released, whichever is sooner.

Each location maintains a current list of documents used by the location to support the QMS. This list, known as the "Master List", is readily available to personnel for their use and it provides a cross reference to the legacy document ID, where applicable. Parties external to PAS may contact the location of interest to obtain the most current version of the Master List for their use as needed.

2.0 REFERENCES

References used to prepare this manual include:

- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136, most current version.
- "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846.
- "Methods for Chemical Analysis of Water and Wastes," EPA 600-4-79-020, 1979 Revised 1983, U.S. EPA.

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- U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis, current version.
- U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis, current version.
- "Standard Methods for the Examination of Water and Wastewater." Current Edition APHA-AWWA-WPCF.
- "Annual Book of ASTM Standards," Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society of Testing and Materials.
- "Annual Book of ASTM Standards," Section 11: Water and Environmental Technology, American Society of Testing and Materials.
- "NIOSH Manual of Analytical Methods," U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health, most current version.
- "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water," U.S. EPA, Environmental Monitoring and Support Laboratory – Cincinnati (Sep 1986).
- Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.
- Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079C.
- Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February 1992.
- Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July 1990.
- Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, most current version.
- National Environmental Laboratory Accreditation Conference (NELAC) Standard- most current version.
- ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories, 2nd Edition 2005-05-15; 3rd Edition 2017-11

  The following are implemented by normative reference to ISO/IEC 17025:

  - ISO/IEC Guide 99, *International vocabulary of metrology –Basic and general concepts and associated terms*
  - ISO/IEC 17000, *Conformity assessment – Vocabulary and general principles*
- Department of Defense Quality Systems Manual (QSM), most current version.
- TNI (The NELAC Institute) Standard, 2009 and 2016 versions.
- UCMR Laboratory Approval Requirements and Information Document, most current version.
- US EPA Drinking Water Manual, most current version.

3.0 TERMS AND DEFINITIONS

Refer to Appendix C for terms, acronyms, and definitions used in this manual and in other documents used by PAS to support the QMS.

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4.0 MANAGEMENT REQUIREMENTS

4.1 Organization

4.1.1 Legal Identity

Pace® Analytical Services, LLC (Pace® Analytical Services) is the responsible entity authorized by the State of Minnesota to do business as a limited liability company, under the parent company, PAS Parent, Inc.

4.1.1.1 Change of Ownership

If there is a change of ownership, if a location goes out of business, or if the entire organization ceases to exist, PAS management is responsible to notify regulatory authorities of the change within the timeframe required by each state agency for which the location is certified or accredited.

Requirements for records and other business information are addressed in the ownership transfer agreement or in accordance with appropriate regulatory requirements, whichever takes precedence.

4.1.2 Compliance Responsibility

PAS management has the responsibility and authority to establish and implement procedures and to maintain resources necessary to assure its activities are carried out in such a way to meet the federal and statutory requirements in addition to the requirements of the PAS QMS. Also See Section 1.1.

4.1.3 Scope of the Quality Management System

The QMS applies to work carried out at each location covered by this manual including permanent facilities, at sites away from its permanent facilities, or in associated temporary or mobile facilities.

The permanent and mobile facilities to which this manual applies are listed on the Title Page of this manual.

4.1.4 Organization History and Information

Founded in 1978, Pace® Analytical Services, LLC (PAS) is a privately held scientific services firm operating one of the largest full-service contract laboratory and service center networks in the United States.

The business purpose of PAS is to deliver the highest standard of testing and scientific services in the market. We offer the most advanced solutions in the industry, backed by transparent data, a highly trained team, and the service and support that comes from over four decades of experience.

4.1.4.1 Organization Structure

Each PAS location is led by a management team referred to as local management[1]. Local management is responsible for making day-to-day decisions regarding the operations of the facility and implementing, and sustaining the requirements, policies, and procedures of the PAS quality program.

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The roles that make up the local management team include a Vice President of Operations (VPO), a General Manager (GM) or Director of Laboratory Operations (DLO), a Quality Program Manager (QPM), and the Quality Manager (QM).

[1] The term "local management" does not mean "on-site" management. Some of the roles that make up the local management team, work off site or from a different PAS location. Refer to the Quality Manual Approval page at the beginning of this manual for the physical address of each manager that comprises the local management team.

The local management team is supported by department specific supervisors and in some PAS locations, a site supervisor or operations manager.

Local management and supervisors are supported by personnel from functional groups that support the division, such as HR, IT, Sales & Marketing, Finance, and EHS (Environmental Health & Safety).

Technical oversight for each location is provided by local personnel with support and guidance from the PAS Chief Technical Officer (CTO), PAS corporate quality personnel, and the Pace® compliance team. Locations that hold TNI accreditation, also have personnel appointed to serve as the "acting technical manager for TNI, however named" to perform the duties and responsibilities of this designation per the TNI Standard. See Section 4.1.5.2.1 for more information on this TNI requirement.

The reporting relationships and responsibilities of quality personnel are independent of operations in order to safeguard impartiality. See Section 4.1.5.2 for more information.

Refer to the organization charts provided in Appendix D to view the organization structure, reporting relationships, and the interrelationships between positions.

4.1.5 Management Requirements

4.1.5.1 Personnel

Each PAS location is staffed with administrative and/or technical personnel who perform and verify work under the supervision of their direct line supervisor.

All personnel are expected to perform their duties in accordance with the policies and processes outlined in this manual and in accordance with standard operating procedures (SOPs) and other quality system documents. PAS policies and procedures are designed for impartiality and integrity. When these procedures are fully implemented, personnel remain free from undue pressure and other influences that adversely impact the quality of their work or data.

4.1.5.1.1 Key Personnel

Key personnel are management positions that have the authority and responsibility to plan, direct, and control activities related to the QMS for the entire division (PAS Corporate), or for one or more PAS locations (Local).

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PAS Key Personnel Positions & Deputy Assignments by Role

Job Title	Acronym	Primary / Alternate Deputy
Chief Executive Officer	CEO	President
Chief Compliance Officer	CCO	CQD
President	NA	CEO / Sr. VPO
Corporate Quality Director	CQD	CCO
Quality Program Manager	QPM	CQD / Peer QPM
Chief Technical Officer	CTO	CQD / CCO
Sr. VP of Operations	Sr. VPO	President / VPO
Vice President of Operations	VPO	Sr. VPO / Peer VPO
Director of Lab Operations[1]	DLO	VPO / Peer GM or Sr. VPO
Health and Safety Director	NA	CCO
IT Director	NA	CTO
Quality Manager	QM	Direct QPM / Peer QPM
General Manager[1]	GM	VPO / Sr. VPO or Peer GM
Operations Manager[1]	OM	GM / DL or VPO
Technical Manager[1]	TM	CTO / Peer TM
TNI Approved TM[2]	TNI TM	Another Qualified Employee

[1.] Position is not in place at all locations.
[2.] The TNI TM is not a PAS position. See Section 4.1.5.2.1 for more information.

Some certification and accreditation programs require notification when there is a change in key personnel. Notification requirements and timeframes by agency, are tracked and upheld by the local QM, when these requirements apply.

4.1.5.2 Roles and Responsibilities

The qualifications, duties, and responsibilities for each position at Pace® are detailed in job descriptions maintained by the Pace® Human Resource personnel (HR).

The following sections provide a general overview of various management and supervisory roles and are presented in no particular order.

Chief Executive Officer (CEO): Provides leadership for overall operations; oversight of regulatory and compliance standards; development of growth strategies; and long-range capital and strategic planning for Pace®.

Chief Compliance Officer (CCO): Has overall responsibility for statutory and regulatory compliance and the environmental health and safety programs (EHS) for Pace®.

President: Provides leadership for overall operations; oversight of regulatory and compliance standards; development of growth strategies; and long-range capital and strategic planning for PAS.

Chief Technical Officer (CTO): Provides technical oversight and leadership to all PAS locations. Responsible for innovation and standardization of technical activities.

Corporate Director of Quality (CQD): Responsible for developing the PAS quality program and the policies and procedures that support the QMS. The CQD leads the

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quality team, establishing functions, responsibilities, duties, and organization structure for PAS.

Corporate Quality Program Manager (QPM): Responsible for helping local management implement, monitor, maintain and improve the PAS quality program for one or more locations in the network and for direct supervision of Quality Manager(s).

Director of Information Technology: Oversees and delivers the systems and processes of information technology used by PAS. These systems include Laboratory Information Management Systems (LIMS); data acquisition, reduction, and reporting software; virus-protection, communication tools, and ensuring the integrity, security of electronic data, and associated policies and procedures.

Sr. Vice President of Operations (Sr VPO): Provides leadership, direction, and insight necessary to achieve strategic initiatives. Develops and improves processes, structure, and allocation of resources for operations for all of PAS.

Vice-President of Operations (VPO): Provides leadership, guidance, and resources, including allocation of personnel, necessary to achieve the strategic goals of the organization and the PAS quality program to one or more PAS locations.

Director of Laboratory Operations (DLO): See description for General Manager.

General Manager (GM): The GM is responsible for overall administration and operation of one or more PAS locations and service centers. Although task duties associated with this responsibility may be delegated, the GM is responsible for ensuring all duties and activities of the locations they oversee comply with the PAS QMS, the PAS EHS program, and with any applicable statutory, regulatory requirements or program requirements.

Any GM of a NELAC/TNI Accredited laboratory is also responsible for the designation of technical personnel to serve as acting technical managers for TNI for the fields of accreditation held by the laboratory (See Section 4.1.5.2.1) and for notifying the accreditation body (AB) of any extended absence or reassignment of these designations.

Quality Manager (QM): The QM oversees and monitors the implementation, compliance, and improvement of the QMS and communicates gaps, deviations, and opportunities for improvement to local and corporate laboratory management. The QM is independent of the operation and analytical activities for which they provide oversight and has the authority to carry out the roles and responsibilities of their position without outside influence.

The QM:

- serves as the focal point for QA/QC protocol decisions and oversees review of QC data for trend analysis;
- evaluates data objectively and performs assessments without outside influence;

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- has documented training and experience in QA/QC procedures and the PAS quality system;
- has a general knowledge of the analytical methods offered by the laboratory;
- coordinates and conducts internal systems and technical audits;
- notifies laboratory management of deficiencies in the quality system;
- monitors corrective actions;
- provides support to technical personnel and may serve as the primary deputy for the acting TNI Technical Manager(s).

Manager-Client Services (CSM): This position is responsible for the training and supervision of project manager(s) and/or shipping, receiving and courier personnel. The primary responsibility of the CSM is to ensure projects are successfully managed to meet the expectations and needs of PAS customers.

Department Managers / Supervisors / Team Lead: These positions are responsible for administrative and operations management and implementation of the QMS in the work area he/she oversees. These responsibilities include but are not limited to: training and supervision of personnel, monitoring work activity to maintain compliance with this manual, SOPs, policies and other instructional documents that support the QMS; method development, validation and the establishment and implementation of SOPs to assure regulatory compliance and suitability for the intended purpose; monitoring QA/QC performance, proper handling and reporting of nonconforming work, purchasing of supplies and equipment adequate for use, maintaining instrumentation and equipment in proper working order and calibration, and general maintenance of administrative and technical processes and procedures established by the laboratory.

Operations Manager (OM): The OM is responsible for management of production and/or other duties assigned by the GM.

4.1.5.2.1 Approved Technical Manager (TNI Accreditation Only):

The requirements in this subsection apply to only to PAS locations that are NELAC/TNI accredited.

The TNI Standard specifies requirements for the qualification and duties of technical personnel. The TNI Standard lists these duties under the reference "technical manager(s), however named."

At PAS, these duties closely correlate with the responsibilities and duties outlined in the PAS job descriptions for managers, supervisors, team leads, and/or scientist. However, these duties do not need to be associated with any specific job title and can be assigned to any one or more PAS employees that meets the qualifications specified in the TNI Standard.

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Refer to the applicable version of the TNI Standard to view the required qualifications for each discipline.

PAS locations that are TNI accredited must designate one or more employees to perform these duties and submit these qualifications to the TNI accreditation body (AB) for approval.

Employees approved by the TNI AB, to perform these duties retain their Pace® assigned job title.

When TNI Accreditation Bodies (AB) refer to these employees as 'technical manager' or 'technical director' on the official certificate or the scope of accreditation, this reference is referring to their approval to perform duties of the 'technical manager, however named' as specified in the TNI Standard and not to a PAS job title.

The duties of any approved technical manager for TNI, however named, can be completed in person or remotely. If an employee that is an approved technical manager for TNI is completely absent from work or on a leave of absence for more than 15 calendar days, the duties and responsibilities specified in the TNI Standard are temporarily reassigned to another employee that meets the qualifications for the technology or field of accreditation. If the employee's absence exceeds 35 calendar days, the local QM must formally notify the TNI primary AB of the absence and the details of reassignment of duties in writing.

4.1.5.3 Conflict of Interest

A conflict of interest is a situation where a person has competing interests that may affect impartiality. It is the policy of Pace® to ensure business relationships, decisions and transactions do not place personal interest ahead of the organization, customers, colleagues, job responsibilities or the public we serve. Conflict of interest is avoided by making personnel aware of circumstances that conflict or appear to conflict with impartiality and/or designing process and procedures to include checks and balances to prevent conflict and ensure impartiality.

See the current version of policy COR-POL-0004 *Code of Ethics and Professional Conduct* for more information.

4.1.5.4 Confidentiality

PAS management is committed to preserving the confidentiality of Pace® customers and confidentiality of Pace® business information.

Client information obtained or created during work activities is considered confidential and is protected from intentional release to any person or entity other than the client or the client's authorized representative, except when Pace® is required by law to release confidential information to another party, such as a regulatory agency or for litigation purposes. In which case, Pace® will notify the client

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of the release of information and the information provided, unless notification is prohibited by law.

When Pace® obtains information about the customer from a source other than the customer, Pace® will keep the source of the information confidential unless disclosure is agreed upon by the source.

The terms of client confidentiality are included in PAS Standard Terms and Conditions (T&C). With the acceptance of the T&C and/or the implicit contract for analytical services that occurs when the client sends samples to PAS for testing, the client authorizes Pace® to release confidential information when required. Other procedures used by PAS to maintain confidentiality include:

- A Code of Ethics and Professional Conduct policy that covers this topic (COR-POL-0004):
- A Confidentiality Agreement which supervisory and sales personnel and other positions are required to sign at the time of employment and abide by the conditions of throughout employment;
- Record retention and disposal procedures that assure confidentiality is maintained; and
- Physical access controls and encryption of electronic data; and

See policy COR-POL-0004 *Code of Ethics and Professional Conduct* for more information.

4.1.5.5 Communication

Communication is defined as the imparting or exchanging of news and information. Effective (good) communication occurs when the people included in the communication gets the point and understands it.

4.1.5.5.1 Workplace Communication

Effective communication in the workplace is necessary to assure work is performed correctly, efficiently, and in accordance with client specifications.

Instructions for how to conduct testing and other work activities are communicated to personnel via written policies, standard operating procedures, and other work instructions.

Information about PAS performance (positive and negative) and ideas for improvement are communicated to personnel using various communication channels such as face to face meetings, video conferencing, conference calls, email, memoranda, written reports, and posters.

4.1.5.5.2 External Communication

Communication with external parties such as customers, vendors, business partners, and regulatory agencies takes place every day.

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PAS management is responsible for training personnel to communicate in professional and respectful ways to build strong relationships and to avoid misunderstanding.

4.2 Quality Management System

4.2.1 Quality Management System Objectives

The objectives of the PAS QMS are to provide clients with consistent, exemplary professional service, and objective work product that is of known and documented quality that meets their requirements for data usability and regulatory compliance.

Objective work product is analytical services, data, test results, and information that is not influenced by personal feeling or opinions. The quality of being objective is also known as 'impartiality.'

4.2.1.1 Impartiality

PAS achieves and maintains impartiality by establishing an organizational structure that safeguards impartiality (See 4.1.4.1) and implementing and adhering to the policies and processes of the QMS outlined in this manual, which are based on industry accepted standards and methodologies.

PAS procedures for handling nonconforming work (See 4.9), corrective and preventive actions (See 4.11, 4.12) and management review (See 4.15) are the primary mechanisms used to identify risk to impartiality and to prompt actions necessary to eliminate or reduce the threat when risk to impartiality is suspected or confirmed.

4.2.1.2 Risk and Opportunity Assessment

Risks are variables that make achieving the goals and objectives of the QMS uncertain.

An opportunity is something that has potential positive consequences for the organization.

PAS personnel manage risks and opportunities on a daily basis by following policies, procedures and processes that support the QMS. Some ways in which the QMS is designed to identify, minimize, or eliminate risk on a daily basis include but are not limited to:

- Capability and capacity reviews of each analytical service request to assure the laboratory can meet the customer's requirements;
- Maintenance of accreditation and certification for test methods in multiple states and programs to cover a broad range of jurisdiction for regulatory compliance;
- SOPs and other controlled instructional documents are provided to personnel to eliminate variability in the process. These documents include actions to counter risk factors inherent in the process and are reviewed on a regular basis for on-going suitability and relevancy;
- Participation in proficiency testing programs and auditing activities to verify on-going competency and comparability in performance;

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- Provision of on-the-job training and established protocol for quality control (QC) corrective action for nonconforming events;
- An established program for ethics, and data integrity;
- Tiered data review process;
- Culture of continuous improvement;
- Monitoring activities to assess daily and long-term performance; and
- Annual critical review of the effectiveness of the QMS.

PAS also promotes a continuous improvement culture based on the principles of lean manufacturing. These principles include 3P (Process, Productivity, Performance) and Kaizen. 3P is a platform used by PAS to share best practices and standardization across the network to achieve operational excellence. Kaizen is a team-based process used to implement tools and philosophies of lean to reduce waste and achieve flow with the purpose of improving both external and internal customer satisfaction. The PAS lean program and activities help to mitigate risk because they generate a collective understanding of vulnerabilities and utilize group-effort to develop and implement solutions at all levels.

Risk and opportunities may also be formally identified using specific risk and opportunity assessment methods such as SWOT Analysis (Strength, Weakness, Opportunity, Threats) and 3-Stage Impact/Probability Grids.

4.2.1.3 Communication of the Quality Management System

This manual is the primary mechanism used by PAS management to communicate the QMS to personnel.

To assure personnel understand and implement the quality program outlined in the manual:

- PAS personnel are required to sign a Read and Acknowledgement Statement to confirm the employee has:

  1) been informed of the manual by management,

  2) has access to the manual,

  3) has read the manual

  4) understands the content of the manual, and

  5) agrees to abide by the requirements, policies, and procedures therein.
- Personnel are informed that the manual provides the "what" of the QMS. The "how to" implementation of the QMS is provided in policy, SOPs, standard work instructions, and other instructional documents.
- This manual and supporting policies and procedures are made readily accessible to personnel in the area where the work activity is performed.

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4.2.2 Quality Policy Statement

The quality policy of PAS is to provide customers with data of known and documented quality fit for their intended purpose. PAS achieves this policy by implementing the QMS defined in this manual, by following industry accepted protocol for analytical testing and quality assurance and quality control (QA/QC) activities, by conformance with published and industry accepted testing methodologies, and by compliance with international and national standards for the competency and/or accreditation of testing laboratories.

Intrinsic to this policy statement is each of the following principles:

- PAS will provide customers with reliable, consistent, and professional service. This is accomplished by making sure each PAS location has the resources necessary to maintain capability and capacity; that staff are trained and competent to perform the tasks they are assigned; that client-facing staff are trained and prepared to find solutions to problems and to assist customers with their needs for analytical services. Customer feedback, both positive and negative, is shared with personnel and used to identify opportunities for improvement.
- PAS maintains a quality program that complies with applicable state, federal, and industry standards for analytical testing and competency.
- PAS management provides training to personnel so that all personnel are familiar with the QMS outlined in this manual and that they understand that implementation of the QMS is achieved by adherence to the Pace® and PAS policies and procedures.
- PAS management continuously evaluates and improves the effectiveness of the QMS by responding to customer feedback, and other measures of performance, such as but not limited to the results of internal/external audits, proficiency testing, metrics, trend reports, and annual and periodic management reviews.

4.2.2.1 Ethics Policy / Data Integrity Program

Pace® has established a comprehensive ethics and data integrity program that is communicated to all Pace® employees so that they understand what is expected of them. The program is designed to promote a mindset of ethical behavior and professional conduct that is applied to all work activities.

The key elements of the Pace® Ethics / Data Integrity Program include:

- Ethics Policy (COR-POL-0004);
- Ethics Officer (Chief Compliance Officer);
- Standardized data integrity training course taken by all new employees on hire and a yearly refresher data integrity training course for all existing employees;
- Policy Acknowledgement Statements that all Pace® personnel, including contract and temporary, are required to sign at the time of employment and again during annual refresher training to document the employee's commitment and

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obligation to abide by the company's standards for ethics, data integrity and confidentiality;

- SOPs that provide instructions for how to carry out a test method or process to assure tasks are done correctly and consistently by each employee;
- On the Job Training;
- Data integrity monitoring activities which include, but are not limited to, primary, secondary and completeness data reviews, internal technical and system audits, data audits, data surveillance, and proficiency testing; and
- Confidential reporting process for alleged ethics and data integrity issues.

All PAS managers and supervisors are expected to provide a work environment where personnel feel safe and can report unethical or improper behavior in complete confidence without fear of retaliation. Retaliation against any employee that reports a concern is not tolerated.

Pace® has engaged Lighthouse Services, Inc. to provide personnel with an anonymous reporting process available to them 24 hours per day/7 days per week. The alert line may be used by any employee to report potential violations of the company's ethics and data integrity program. Reports are forwarded to the Pace® Ethics Compliance Officer to investigate and resolve the matter. Investigations concerning data integrity are kept confidential.

See COR-POL-0001 *Compliance Alertline* for more information.

Posters and flyers with the compliance alert line information must be prominently posted in each PAS location for personnel reference.

Compliance Alert Line Information:

English Speaking US & Canada	(844) 940-0003
Spanish Speaking North America	(800) 216-1288
Internet	www/lighthouse-services.com/pacelabs
Email	reports@lighthouse-services.com

4.2.3 Management Commitment: Quality Management System

Evidence of management's commitment for the development, maintenance, and on-going improvement of the QMS is provided by the application of their signature of approval to the template and/or manual. Their signature confirms they understand their responsibility to implement the QMS outlined in this manual, to communicate the quality program to personnel, and to uphold requirements of the program during work activities.

4.2.4 Management Commitment: Customer Service

Management communicates the importance of meeting customer and regulatory requirements to personnel by training personnel on the QMS outlined in this manual, implementing the QMS outlined in this manual, and upholding these requirements for all work activities.

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4.2.5 Supporting Procedures

References to processes and procedures that support the QMS are included throughout this manual. The structure of the document management system is outlined in SOP ENV-SOP-CORQ-0015 *Document Management and Control* and summarized in the following subsections.

4.2.5.1 Quality Management System Document Structure

Documents associated with the QMS are classified into document types that identify the purpose of the document and establish how the document is managed and /or controlled.

Examples: Types of PAS Internally Created Documents

Document Type	Purpose
Quality Manual	Outlines the PAS QMS and structure and how it works for a system including policy, goals, objectives and detailed explanation of the system and the requirements for implementation of system. Includes roles and responsibilities, relationships, procedures, systems, and other information necessary to meet the objectives of the system described.
Policy	Provide requirements and rules for a process and is used to set course of actions and to guide and influence decisions. Policy describes the "what," not the "how".
Standard Operating Procedure	Provide written and consistent set of instructions or steps for execution of a routine process, method, or set of tasks performed. Assures that activities are performed properly in accordance with applicable requirements.
Standard Work Instruction	Provide step by step visual and/or written instruction to perform a specific task to improve competency, minimize variability, reduce work injury and strain, or to boost efficiency and quality of work (performance). SWI are associated with an SOP unless the task described is unrelated to generation of or contribution to environmental data or analytical results.
Template	Pre-formatted document that serves as a starting point for a new document.
Guide	Assists users in using a particular product; or a technical interpretation of a method or process by which PAS locations must abide.
Form	Used for a variety of purposes such as to provide a standardized format to record observations, to provide information to supplement an SOP.
Guidance	Non-binding advice used to explain internal policies, procedures, or practices.

Example: Types of External Documents used by PAS

Certificate	Lists parameters, methods, and matrices for which the location is certified/accredited to perform within the jurisdiction of the issuing regulatory agency or accreditation body.
Reference Document	Provide information, protocol, instructions, and/or requirements. Issued by the specifier. Examples include ISO/IEC, TNI, DoD and published referenced methods such as Standard Methods, ASTM, SW846, EPA, and federal and state regulatory bodies.
Project Document	Provides requirements necessary to meet individual client expectations for intended use of data. Examples include project quality assurance plans (QAPP), client-program technical specifications, contracts, and other agreements.

These document types are ranked to establish which documents takes precedence when there is an actual or perceived conflict between documents and to establish the hierarchal relationships between documents. The ranking system also provides information to document writers and reviewers to assure downline documents agree with documents of higher rank.

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PAS Document Hierarchy

Rank	Document
1	Corporate Manual
2	Corporate Policy
3	Corporate SOP
4	Corporate SWI, Templates, Guides, Forms, Guidance
5	Local Manual
6	Local SOP
7	Local SWI, Templates, Guide, Forms, Guidance

Information and requirements from project documents are not incorporated into PAS policy or SOPs in order to maintain client confidentiality. These documents are managed as external documents and any requirements for work specified is followed when work for the project is performed. Project Documents are reviewed and maintained as part of the contract/incoming work review process (See Section 4.4). If the project document is less stringent than the PAS QMS, policies, or SOPs, and/or is less stringent than applicable federal or state requirements, PAS locations are still required to meet the minimum requirements of the PAS QMS and any applicable statutory or federal requirements in addition to the requirements specified in the project document.

Reference documents are not ranked because all PAS created documents, processes and procedures must be consistent with the applicable reference document(s) in addition to higher-ranking PAS documents.

See SOP ENV-SOP-CORQ-0015 *Document Management and Control* for more information.

4.2.6 Roles and Responsibilities

The roles and responsibilities for technical management and the quality manager are provided in section 4.1.5.2.

4.2.7 Change Management

When significant changes to the PAS QMS are planned, these changes are managed by corporate quality personnel to assure that the integrity of the QMS is maintained.

4.3 Document Control

4.3.1 General

PAS procedures for document control are provided in SOP ENV-SOP-CORQ-0015 *Document Management and Control.*

PAS locations use electronic document management software (eDMS) to perform the document control procedures of the SOP. This system provides centralized access to all documents used by PAS locations across the network. All PAS locations are required to use the eDMS system established for PAS (presently Qualtrax) unless an exemption has been granted by the PAS Corporate Quality Director.

eDMS automates the process for unique document identification, version control, approval, access, and archival and restricts access to archived documents except to authorized users to prevent the use of obsolete documents.

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The local QM maintains a master list of controlled documents used at each location. The master list minimally includes the document control number, document title, and current revision status and is made available to personnel for their reference.

See SOP ENV-SOP-CORQ-0015 *Document Management and Control* for more information.

4.3.2 Document Approval and Issue

Documents that support the QMS are reviewed by qualified personnel and approved by management prior to release for use.

Only the approved versions of documents are available to personnel for use unless use of a draft document is authorized by management.

The managers responsible for authorization of each document is situation specific.

See SOP ENV-SOP-CORQ-0015 *Document Management and Control* for more information.

4.3.3 Document Review and Change

Unless a more frequent review is required by regulatory, certification or accreditation program documents are reviewed at least every two years to ensure the documents remains current, appropriate, and relevant.

Documents are also informally reviewed every time the document is used. Personnel are expected to refer to and follow instructions in controlled documents when they conduct their work activities. Consequently, any concerns or problems with the document should be caught and brought to the attention of management on an on-going basis.

Documents are revised whenever necessary to ensure the document remains usable and correct. Older document versions and documents no longer needed are made obsolete and archived for historical purposes.

PAS does not allow hand-edits to documents. If an interim change is needed pending re-issue of the document, the interim change is communicated to those that use the document using a formal communication channel, such as change in progress form, email, or memorandum.

The document review, revision, and archival process is managed by quality personnel at the location from which the document was released using the procedures established in SOP ENV-SOP-CORQ-0015 Document Management and Control.

4.4 Analytical Service Request, Tender, and Contract Review

PAS management and/or client service personnel perform thorough reviews of requests and contracts for analytical services to verify the location(s) performing the work has the capability, capacity, and resources necessary to successfully meet the customer's needs. These review procedures are described in SOP ENV-SOP-ASHE-0033 *Review of Analytical Requests.*

The procedures in this SOP(s) are established to ensure that:

- The PAS locations performing the work understand the purpose of data collection in order to ensure the test methods requested are appropriate for the intended use of the data and capable of meeting the client's data quality objectives;

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- PAS locations and any external subcontractor(s) have the capability, capacity, and resources to meet the project requirements and expectations within the requested time frame for delivery of work product;
- Any concerns that arise from review are discussed and resolved with the client;
- Any discrepancies between the PAS QMS, statutory or regulatory requirements and the client request are resolved; and
- The results of review and any correspondence with the client related to this process and/or any changes made to the contract are recorded and retained for historical purposes.

Capability review confirms that the PAS locations contracted to perform the work and any internal or external subcontractors hold required certification/accreditation for the test method, matrix, and analyte and verifies the location can achieve the client's target compound list and data quality objectives (DQOs) for analytical sensitivity and reporting limits, QA/QC protocol, and hardcopy test report and electronic data deliverable (EDD) formats.

Capacity review verifies that the in-network locations and any potential subcontractors are able to manage the sample load and deliver work production within the delivery timeframe requested.

Resource review verifies that the location and any potential subcontractors have adequate qualified personnel with the skills and competency to perform the test methods and services requested and sufficient and proper equipment and instrumentation needed to perform the services requested.

4.5 Subcontracting (Internal and External)

The terms 'subcontract' and 'subcontracting' refers to analytical work done by an organization external to Pace® (External Subcontracting) or by a Pace® location with an address different than the address listed on the cover page of the test report (Internal Subcontracting).

The PAS network offers comprehensive analytical capability and capacity to ensure Pace® can meet a diverse range of client needs for any type of project. If a PAS laboratory receives a request for analytical services and it cannot fulfill the project specifications, the location's client services team will collaborate with the client to place the work within the PAS network.

When it is not possible to place the work within network, the location will, with documented client approval, subcontract the work to a subcontractor that has the capabilities to meet the project specifications and can meet the same commitment agreed on between the location and the client.

Whenever work is subcontracted, the PAS location responsible for management of the project verifies each of these qualifications:

- The internal or external subcontractor has the proper accreditation/certifications required for the project and these are current; and
- The use of the internal or external subcontractor is approved by the client and/or regulatory agency when such approval is required by the customer. Record of customer approval is retained in the project record.

External subcontractors selected by Pace® must be pre-qualified by quality personnel to verify their QMS is similar to Pace® and complies with all relevant Standards such, as ISO/IEC 17025 and the TNI Standard(s) and/or federal and state regulatory requirements. The list of approved

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subcontractors for each location is maintained by local quality personnel. Pre-qualification of a subcontractor does not eliminate the requirement for the PAS location placing work to verify the subcontractor has the certifications, capability, capacity, and resources to perform work on behalf of Pace® on a project-specific basis.

For all subcontracted work, the PAS location placing the work internally or externally is responsible to ensure project specifications are always communicated to and understood by the subcontractor.

4.6 Purchasing Services and Supplies

Vendors that provide services and supplies to PAS are qualified to meet the needs of Pace®. These needs include but are not limited to competitive pricing, capacity to fill purchase orders, quality of product, customer service, and business reputation and stability. Evidence of this qualification is the availability to purchase services and supplies from the vendor in the corporate purchasing system.

PAS locations may purchase goods and services from any supplier in the purchasing system.

The specifications (type, class, grade, tolerance, purity, etc.) of supplies, equipment, reagents, standard reference materials and other consumables used in the testing process are specified in SOPs. The SOP specifications are based on the governing requirements of the approved reference methods and any additional program driven regulatory specification, such as drinking water compliance.

All requisitions for materials and consumables are approved by local management who is responsible to ensure the services and supplies procured and received are fit for intended use.

4.7 Customer Service

Project details and management is managed by PAS client services personnel.

4.7.1 Commitment to Meet Customer Expectations

PAS personnel collaborate closely with our customers to ensure their needs are met and to establish their confidence in the capability of PAS to meet their needs for analytical services and expectations for service.

The project manager (PM) is the customer's primary point of contact for each analytical service request (work order). The PM gathers information from the customer to ensure the details of their request are understood. After samples are received, the PM monitors the progress of the project and alerts the customer of any delays or excursions that may adversely impact data usability. Supervisors are expected to keep the PM informed of project status and any delays or key issues, so that the PM can keep the client informed.

PAS encourages customers to visit our locations to learn more about the capabilities, observe performance and to meet personnel.

PAS customers expect confidentiality. Personnel will not divulge or release information to a third party without proper authorization unless the information is required for litigation purposes. See Section 4.1.5.4 of this manual and policy COR-POL-0004 *Code of Ethics and Professional Conduct* for more information on the policy for client confidentiality.

4.7.2 Customer Feedback

PAS actively seeks positive and negative feedback from customers through surveys and direct communication. Information from the client about their experience working with PAS and

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their satisfaction with work product is used to enhance processes and practices and to improve decision making. Customer feedback is reviewed to identify risk and opportunity. Corrective, preventive, or continuous improvement actions are taken based on nature of and/or feedback trends.

Also see sections 4.9, 4.10, 4.11, 4.12, 4.14, and 4.15 for more information about how customer feedback is managed by PAS and used to enhance the QMS.

4.8 Complaints

A complaint is a formal expression of dissatisfaction with the performance of a service or product originating from a party external to Pace®. Complaints provide opportunities to improve processes and/or build stronger working relationships with clients.

The PAS complaint resolution process depends on the situation and the nature of the complaint.

Each complaint received is reviewed to determine if it is valid. If the complaint is valid, it is either addressed immediately by the person receiving the complaint or the nature of the complaint is further reviewed and investigated prior to resolution and follow up with the customer.

Complaints (and compliments) are recorded and reviewed during Annual Management Review (See Section 4.15).

4.9 Nonconforming Work

4.9.1 Definition of Nonconforming Work

Nonconforming work is work that does not conform to customer requirements, standard specifications, policies, and procedures, or that does not meet acceptance criteria.

The discovery of non-conforming work comes from various sources which include, but are not limited to:

- results of quality control samples and instrument calibrations;
- quality checks on consumables and materials;
- general observations of personnel;
- data review;
- proficiency testing;
- internal and external audits;
- complaints and feedback;
- management review and reports; and
- regulatory and certification and accreditation actions.

The way in which the laboratory or service center manages nonconforming work depends on the significance and impact (risk) of the issue. Some issues may simply require correction, others may require investigation, corrective action (See 4.11) and/or data recall (See 4.16). When the location releases data and test results associated with nonconforming QC and acceptance criteria, test results are qualified, or non-conformances are noted in the final analytical report to apprise the data user of the situation. (See 5.10)

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Nonconforming work also includes unauthorized departure from policies, procedures, and test methods. Authorized departures are explained in the following subsections. Situations that do not conform to these conditions are considered unauthorized departure(s).

4.9.1.1 Authorized Departure from SOPs

Departures from an SOP may sometimes be necessary to correct for an error in an SOP or to resolve a complex problem. For example, to mitigate a complex matrix interference.

An authorized departure from a test method SOP is one that has been reviewed and approved by the department leader, however named, of the work area in which the test method is performed. The leader, when authorizing a departure from an SOP, accepts full responsibility to ensure the departure does not conflict with Pace® or PAS policy or procedure, does not affect statutory, regulatory or program compliance and does not adversely affect data integrity or usability.

Departure from administrative or process-oriented SOPs must be approved by the local QM.

Documentation of the reason for the SOP departures must be retained with management approval. Approved departures from test method SOPs should be noted in the final test report to advise the data user.

See SOP ENV-SOP-CORQ-0016 *SOP for SOPs and SWI*, for more information.

4.9.1.2 Authorized Departure from Test Methods (Method Modifications)

When test results are associated to a published reference test method, the location's test method SOP must be consistent with the test method. If the test method is mandated for use by a specific regulatory program such as drinking water, wastewater or a certification or accreditation program, such as TNI/NELAC, the SOP must comply with or include these requirements, or the resulting data and test results cannot be used for regulatory compliance purposes.

If the procedures in the SOP are modified from the test method, these modifications must be clearly identified in the SOP. The conditions under which the location may establish an SOP that is modified from these reference method or regulatory program and what is considered a modification are specified in ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification*.

Client requests to deviate from the test method are managed as client requests to depart from the test method SOP since it is the SOP that the location follows when performing work.

4.9.1.3 Stop Work Authority

Stop Work Authority provides PAS personnel with capability to stop work when there is a perceived unsafe condition or behavior that may result in an unwanted event.

All personnel have the authority to request a stop work order when necessary to preserve data integrity or safety of workers.

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The need for the stop work order and resolution of the problem must be confirmed by subject matter experts and resumption of work must be approved as follows:

- For stop work orders related to environmental health and safety (EHS) and/or waste management, the decision to stop work may be made by any employee. These decisions must often be made in real-time to protect the safety of the worker. The decision to correct the problem, how, and/or to resume work after stop work has been initiated may be made by the Chief Compliance Officer or the EHS Director, or the deputies assigned to these positions.

- Any employee may recommend a stop work order for concerns related to data integrity. The need to stop work must be reviewed and affirmed by quality personnel to confirm the concern is valid. The decision to uphold the stop work order must minimally include the local QM, the QPM, and the Corporate Quality Director. The President, the Sr. VPO, the VPO, the Chief Compliance Officer and Chief Technical Officer may also be included in the decision making and resolution process depending on the situation and/or needs for correction to ensure protocols for investigation are followed. Resumption of work after correction may be made by the Corporate Quality Director, or the Quality Program Manager assigned to the location for which the stop work order was issued or by the deputies assigned to these positions.

4.10 Continuous Improvement

The PAS QMS is designed to achieve continuous improvement through the implementation of the quality policy and objectives outlined in this manual. Information about laboratory and service center activities and performance is gained from sources such as customer feedback, audits, QC, trend analysis, business analytics, management reports, proficiency testing, and management systems review. This information is subsequently used during the corrective action (see section 4.11) and preventive action (see section 4.12) processes and during annual review of the management system (see section 4.15) to establish goals and objectives for improvement.

PAS also promotes a continuous improvement culture based on the principles of lean manufacturing. These principles include 3P (Process, Productivity, Performance) and Kaizen. 3P is a platform used by Pace to share best practices and standardization across the network to achieve operational excellence. Kaizen is a team-based process used to implement tools and philosophies of lean to reduce waste and achieve flow with the purpose of improving both external and internal customer satisfaction. All activities of 3P and Lean must conform with the requirements of this quality manual and supporting policies and procedures.

4.11 Corrective Action

Corrective action is a process used to eliminate the cause of a detected nonconformity. It is different from a correction. A correction is an action taken to fix an immediate problem but that does not resolve the underlying cause of why the problem occurred. The objective of corrective action is to find the underlying cause(s) of the problem and to put in place fixes to prevent the problem from happening again. The corrective action process, referred to as CAPA, is one of the most effective tools used by PAS to prevent nonconforming work, identify risk and opportunity, and improve service to our customers.

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PAS has two general processes for corrective action, the application of which process is used depends on the type of nonconformity:

Quality control (QC) exceptions (nonconformance) that occur during routine testing are investigated through troubleshooting and required actions for correction is specified in policies and SOPs. When action is not taken, cannot be taken, or is not successful, test results associated with the nonconforming work are qualified in the final test report. Documentation of the nonconformance and corrective action taken is documented in the analytical record.

A 7-stage corrective action process is used when there is a recurring problem. These problems are identified through various activities such as but not limited to quality control trends, internal and external audits, management review, customer feedback, and general observation.

The 7 Stage CAPA Process for PAS includes:

1) Identification and Containment
2) Evaluation
3) Investigation
4) Cause Analysis
5) Action Plan
6) Implementation
7) Follow Up and Effectiveness Review

PAS procedures for corrective action, are specified in corporate SOP ENV-SOP-CORQ-0018 *Procedure for Corrective and Preventive Action.* Some key concepts and activities related to the PAS corrective action process is provided in the next three subsections.

4.11.1 Cause Analysis (AKA Root Cause Analysis)

Cause analysis is the process of investigation used to identify the underlying cause(s) of the problem. After causal factors are identified, ways to mitigate the causal factors are identified and action(s) most likely to eliminate these factors are taken.

PAS uses different methods to conduct cause analysis. The most common approach is 5-Why, 4M, Fishbone Diagrams, or brainstorming may be appropriate depending on the situation. The method used is case specific and is documented in the CAPA record.

4.11.2 Effectiveness Review

Monitoring corrective actions taken for effectiveness is an essential part of the corrective action process. Effectiveness means the actions taken were appropriate and sustainable. Appropriate means the action(s) taken prevented recurrence of the problem since the time corrective action was taken and sustainable means the actions taken are still in place.

The data from CAPA records are used by PAS to identify opportunities for preventive action or to gain lessons learned when actions taken were not adequate to solve the problem. See Section 4.12 (Preventive Action) and 4.15 (Management Review) for more information.

4.11.3 Additional Audits

When cause analysis and investigation of a problem casts doubt on compliance with PAS policies, procedures, or to regulatory requirements; a special audit of the area of activity may be performed as part of the corrective action process. These special audits are used to

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determine the scope of the problem and to provide information for the CAPA process. Additional full-scale audits are done when a grave issue or risk to the business is identified.

4.12 Preventive Action

Preventive action(s) are actions taken to eliminate the cause of a potential nonconformity before it happens.

Some examples of preventative action include, but are not limited to:

- Routine instrument maintenance (Preventative maintenance)
- Addition of Staff and Equipment
- Professional Development Activities
- Implementation of New Technology

PAS looks for opportunities for preventive action from a variety of sources including employee idea's, customer feedback, business partners input, trend analysis, business analytics, management reviews, proficiency testing results, and risk-benefit analysis.

PAS management evaluates the success of preventive actions taken in any given year during annual management review. See Section 4.15 for more information.

4.12.1 Change Management

Preventive actions may sometimes result in significant changes to processes and procedures used by PAS locations. PAS management evaluates the risks and benefits of change and includes in its implementation of change process, actions to minimize or eliminate any risk. The types of changes for which risk are considered and managed include infrastructure change, change in analytical service offerings, certification or accreditation status, instrumentation, LIMS changes, and changes in key personnel.

4.13 Control of Records

A record is a piece of evidence about the past, especially an account of an act or occurrence kept in writing or another permanent form. PAS records document activities and provide evidence of conformity to the requirements established in the QMS. These records may be hardcopy or electronic on any form of media.

4.13.1 General Requirements

4.13.1.1 Procedure

PAS requirements for control of records are specified in corporate policy ENV-POL-CORQ-0013 *Record Managemen*t.

The policy is established to assure quality and technical records are identified, retained, indexed, and filed to allow for retrieval during the entire retention timeframe. During storage, records are kept secure and protected from deterioration. At the end of the retention time, the records are disposed of properly in order to maintain client confidentiality and to protect the interests of the company.

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In general, records fall into three categories: quality, technical, and administrative.

Examples of each are provided in the following table:

Record Type	Includes Records of:
Quality	Document Types listed in SOP ENV-SOP-CORQ-0015 Audits: Internal and External Certificates and Scopes of Accreditation Corrective & Preventive Action Management Review Data Investigations Method Validation Instrument Verification Training Records
Technical	Raw Data Logbooks Certificates of Traceability Analytical Record Test Reports & Project Information Technical Training Records & Demonstration of Capability
Administrative	Personnel Records Finance/Business

4.13.1.2 Record Legibility and Storage

Records are designed to be legible and to clearly identify the information recorded. Manual entries are made in indelible ink; automated entries are in a typeface and of sufficient resolution to be read. The records identify personnel that performed the activity or entered the information. Records are archived and stored in a way that they are retrievable. Access to archived records is controlled and managed.

For records stored electronically, the capability to restore or retrieve the electronic record is maintained for the entire retention period. Hardcopy records are filed and stored in a suitable environment to protect from damage, deterioration, or loss. Hardcopy records may be scanned to PDF for retention. Scanned records must be checked against the hardcopy to verify the scan is complete and legible.

Administrative records are kept for a minimum of 5 years and technical and quality records are kept for 10 years unless otherwise specified by the client or regulatory program.

The date from which retention time is calculated depends on the record. In general, the retention time of technical records of original observation and measurement is calculated from the date the record is created. If the technical record is kept in a chronological logbook, the date of retention may be calculated from the date the logbook is archived. The retention time of test reports and project records, which are considered technical records, is calculated from the date the test report was issued. The retention time of quality records is usually calculated from the date the record is archived.

Refer to the record management policy and the location specific SOP for more information.

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4.13.1.3 Security

PAS locations are secure facilities and access to records is restricted to authorized personnel.

4.13.1.4 Electronic Records

The data systems used to store electronic records is backed up in accordance with SOP ENV-SOP-ASHE-0058 *Data and Records Archival*. Access to archived records stored electronically is maintained by personnel responsible for management of the electronic system.

4.13.1.5 Electronic Signature Policy

Work done by PAS locations include activities that require the application of a signature. Some work product is in electronic format and signatures are applied electronically.

The Electronic Signatures in Global and National Commerce Act (E-Sign Act) clarifies that electronic signatures are legally valid and enforceable under United States law.

The PAS policy for use and application of electronic signatures is specified in corporate policy ENV-POL-CORQ-0014 *Electronic Signature Policy*.

All employees of PAS including temporary and contract personnel, must sign an Electronic Signature Agreement to acknowledge that they understand and accept that work activities performed by them may be authenticated with application of an electronic signature and that electronic signature has the same validity as a handwritten signature. Their signed agreement also confirms the individual has read and understands the policy and agrees to abide by the requirements for use of electronic signature stated in the policy.

4.13.2 Technical Records

In addition to the requirements specified in subsections 4.13.1.1 through 4.13.1.5, the requirements in the following subsections also apply to technical records.

4.13.2.1 Description

Technical records are the accumulation of data and information generated from the analytical process. These records may include forms, worksheets, workbooks, checklists, notes, raw data, calibration records, final test reports, and project record. The accumulated record needs to provide adequate detail to historically reconstruct the process and identify the personnel that performed the tasks associated with a test result.

4.13.2.2 Real Time Recordkeeping

Personnel are instructed and expected to always record observations, data, and calculations at the time they are made. PAS managers are responsible to assure that data entries, whether made electronically or on hardcopy, are identifiable to the task.

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4.13.2.3 Error Correction

Errors in records must never be erased, deleted, or made illegible. Use of correction fluid, such as white-out is prohibited. In hardcopy records, the error is corrected by a single strike through the original entry and the new entry recorded alongside or footnoted to allow for readability. Corrections are initialed and dated by the person making the correction. If the correction is not self-explanatory, a reason for the correction is recorded.

For electronic records, equivalent measures of error correction or traceability of changes made is kept. For example, audit trails provide records of change.

Maintenance of proper practices for error correction is monitored through the tiered data review process described in Section 5.9.3. Records are reviewed throughout the data review process. Individuals performing these reviews flag errors that are not properly corrected and bring these to the attention of the department manager or supervisor of the work area in which the record was generated so that the problem may be addressed and corrected with the individual(s) that did not make the correction properly.

4.14 Audits

Quality personnel, or their designees, perform internal systems and technical audits to assess implementation of the QMS, compliance to this manual, policy, and procedures that make up the QMS. Since the processes in this manual are based on the requirements from relevant and applicable Standards for the operation and management of laboratories when operations are assessed against the PAS QMS, compliance with regulatory program requirements and accreditation/certification program requirements are also assessed.

PAS locations are also audited by external parties such as regulatory agencies, customers, consultants, and non-government assessment bodies (NGAB).

Information from internal and external audits is used by local and corporate management to address deficiencies and to identify opportunities to improve customer service and quality of work, including reliability and usability of data and test results.

Deficiencies, observations, and recommendations from audits are managed by the local QM using the CAPA process. See Section 4.11 for more information.

4.14.1 Internal Audit

The PAS internal audits are conducted to ensure practice matches what we say we do and what we say we do is compliant with the PAS QMS and relevant standards and requirements.

The internal audit program is managed by the local QM who prepares an audit plan at the beginning of each calendar year. The schedule is prepared to assure that all work areas are reviewed over the course of the year and test methods are audited every two years, unless a more frequent test method audit is required by program. Conformance to the schedule is monitored on a monthly basis.

PAS management is responsible to ensure the audit schedule is maintained. PAS supervisors are expected to cooperate with the quality personnel to provide them with complete access to the work area, personnel, and records needed to conduct the audit.

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Internal audits may be performed by non-quality personnel when the auditor is approved by the local QM. Non-quality personnel may not audit their own work activities unless it can be demonstrated that an effective and objective audit will be conducted. The person conducting the audit should be trained, qualified, and familiar enough with the objectives and policies of the PAS QMS and knowledgeable with process and test method SOPs related to the activities audited. The auditors should be trained in auditing practices in order to perform a thorough and effective evaluation.

Test method audits include reviews of test reports to verify the product is consistent with customer/project requirements, the work was conducted in accordance with policy and SOPs, the SOP complies with the cited reference method, test results are accurate, and of known and documented quality and properly qualified, when necessary.

Special audits are performed as needed to follow up on a specific issue such as a client complaint, negative feedback, concerns of data integrity or ethics, or a problem identified through other audits. Special audits may be scheduled or unscheduled. Unscheduled internal audits are conducted whenever doubts are cast on compliance with regulatory requirements or its own policies and procedures. These unscheduled internal audits may be conducted at any time and may be performed without an announcement to the location or work area audited.

When observations and findings from any audit (internal or external) cast doubt on the validity of testing results, the location takes immediate action to investigate the problem and take corrective action. (Also see 4.11 and 4.16)

4.14.1.1 Corporate Compliance Audit

PAS locations may also be audited by corporate personnel at discretion. The purpose of the corporate compliance audit is to assess whether the location's practices, processes and procedures conform with the PAS QMS and to identify risk and opportunity.

4.15 Management Review

Local management conducts an annual business review of each location under their purview to assess performance and to establish goals, objectives, and action plans for the upcoming year.

The procedure used to conduct this review is specified in corporate SOP ENV-SOP-CORQ-0005 *Management Review*.

At a minimum, the following topics are reviewed and discussed during annual management review:

- Changes in internal and external issues relevant to the location;
- Fulfillment of objectives and initiatives;
- Suitability of policies and procedures, including EHS and waste management;
- Status of actions from previous performance reviews;
- The outcome of recent internal audits;
- Corrective and preventive actions;

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- Assessments by external bodies;
- The results of interlaboratory comparisons or proficiency tests;
- Changes in the volume and type of the work;
- Customer and personnel feedback, including complaints;
- Effectiveness of improvements / preventive actions made since last review;
- Adequacy of resources;
- Results of risk identification;
- Proficiency testing performance and other measures related to the assurance of validity of test results; other relevant factors, such as QC trends and training status.

The discussion and results of this review are documented in a report prepared by local management. This report includes a determination of the effectiveness of the management system and its processes, goals, and objectives for improvements in the coming year with timelines and responsibilities, and any other need for change.

Goals and action items from annual management systems review are shared with local employees and with corporate management to highlight focus areas for improvement in addition to areas in which the location has excelled.

4.16 Data Integrity

PAS procedures for the investigation and response to events that may affect data integrity are described in the corporate SOPs for data inquiries and data recall and corrective and preventive action, however named.

Customers whose data are affected by these events are notified in a timely manner, usually within 30 days after the impact of the problem is understood. Some accreditation programs also require notification to the accreditation body (AB) within a certain timeframe from date of discovery when the underlying cause of the issue impacts accreditation. PAS locations must follow any program or project specific client notification requirements for notification, when applicable.

5.0 TECHNICAL REQUIREMENTS

5.1 General

Multiple factors contribute to the correctness and reliability of the technical work performed by PAS. These factors fall under these broad categories:

- Human Performance
- Facility and Environmental Conditions
- Test Method Performance and Validation
- Measurement Traceability
- Handling of Samples

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The impact of each of these factors varies based on the type of work performed. To minimize negative effects from each of these factors, PAS accounts for the contribution from each of these categories when developing test method and process (administrative) SOPs, evaluating personnel qualifications and competence, and in the selection of equipment and supplies used.

5.2 Personnel

5.2.1 Personnel Qualifications

The PAS program for personnel management is structured to ensure personnel are selected, qualified, and competent to perform the roles and responsibilities of their position based on education, experience, and training.

Qualifications, duties, responsibilities, and authorities of each position are specified in job descriptions maintained by corporate HR (See Section 5.2.4). These job descriptions provide the general basis for the selection of personnel for hire and are used by the location to communicate to personnel the duties, responsibilities, and authorities of their position. Qualification records may include but are not limited to diploma, transcripts, and curriculum vitae (CV).

The term "personnel" refers to individuals employed by PAS directly as full-time, part-time, or temporary, and individuals employed by PAS by contract, such as through an employment agency. The term "personnel" is used interchangeably with the term "employee" throughout this manual. For purposes of this manual, these terms are equivalent.

The personnel management program is structured to establish and maintain records for each of the following:

- Selection of personnel;
- Training of personnel;
- Supervision of personnel;
- Authorization of personnel; and
- Monitoring Competence of personnel.

5.2.1.1 Competence

Competence is the ability to apply a skill or series of skills to complete a task or series of tasks correctly within defined expectations.

Competence for technical personnel authorized by PAS to provide opinion and interpretation of data to customers also includes the demonstrated ability to:

- Apply knowledge, experience, and skills needed to safely and properly use equipment, instrumentation, and materials required to carry out testing and other work activities in accordance with manufacturer specifications and location SOPs;
- Understand and apply knowledge of general regulatory requirements necessary to achieve regulatory compliance in work product; and

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- Understand the significance of departures and deviations from procedure that may occur during the analytical testing process and the capability and initiative to troubleshoot and correct the problem, document the situation and decision-making process, and to properly qualify the data and analytical results.

PAS requirements for the competence of personnel (education, qualification, work experience, technical skills, and responsibilities) are specified in job descriptions created by management and kept by human resources (HR). The job description provides the basis for the selection of personnel for each position.

An employee is considered competent when he/she has completed the required training specified in Section 5.2.2 and documentation of training is complete.

5.2.2 Training (Required)

Pace® training requirements are outlined in Pace® policies COR-POL-0023 *Mandatory Training Policy* and COR-POL-0004 *Code of Ethics and Professional Conduct.*

5.2.2.1 Required Training Requirements

The PAS training program includes these elements:

- Scheduling
- Execution
- Documentation and Tracking
- Evaluation of Effectiveness

Required training is scheduled by corporate training personnel, local quality personnel, and the employee's direct supervisor.

Training on required topics, processes and procedure is delivered using various methods that incorporate techniques that appeal to the main learning styles: visual, aural, linguistic, and kinesthetic. Techniques include, on-the-job, instructor-led, self-study, eLearning, and blended.

The employee's direct supervisor is responsible for oversight of completion of the employee's required training and for providing adequate time to the employee to complete training assignments. The supervisor and employee are responsible to make sure the employee's training status and training records for all required training is current, complete, and documentation of training is available.

Training status is tracked by the local QM, who provides the status to local management at least monthly or more frequently, as necessary, to ensure required training for personnel is complete and up to date.

The following subsections further describe the required PAS training program for new hire training and on-going training.

5.2.2.1.1 New Hire Required Training

New hire training requirements apply to new personnel and to existing employees starting in a new position or different work area.

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Required new hire training includes training on each of the following:

- Ethics and Data Integrity (See 5.2.2.1.3)
- Quality Manual / Quality Management System (See 5.2.2.1.4)
- Safety Manual and any training requirements specified in the manual.
- Policies & SOPs relevant to their job tasks
- Technical personnel that prepare and test samples must also successfully complete an initial demonstration of capability (IDOC) for the test methods performed before independently testing customer samples. (See 5.2.2.1.5). Independent testing means without direct supervision of the work activity by the supervisor or a qualified trainer.

All required training must be documented and verified complete by the local QM before the employee is authorized to work independently on client samples. Until then, the employee's direct supervisor is responsible for all work produced by the new employee under their supervision.

5.2.2.1.2 On-Going Required Training

Personnel receive on-going training in each of the following topics:

- Ethics and Data Integrity (See 5.2.2.1.3)
- Quality Manual / Quality Management System (See 5.2.2.1.4)
- Safety
- Changes to Policies & SOPs, relevant to their job activities.
- New Policies & SOPS, relevant to their job activities.
- Technical personnel must also successfully complete on-going demonstration of capability (CDOC) for all test methods performed on an annual basis. (See 5.2.2.1.5)

All required training must be documented and verified complete by the local QM with training records readily accessible in accordance with the corporate policy for Record Management (ENV-POL-CORQ-0013).

5.2.2.1.3 Ethics and Data Integrity Training

Data integrity training is provided to all new personnel and refresher data integrity training is provided to all employees on an annual basis. Personnel are required to acknowledge they understand that any infractions of the PAS data integrity procedures will result in a detailed investigation that could lead to profound consequences

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including immediate termination, debarment, or civil/criminal prosecution.

Completion of data integrity training is documented using the mechanism established by Pace® to provide evidence that the employee has participated in training on this topic and understand their obligations related to data integrity.

The following topics and activities are covered:

- Policy for honesty and full disclosure in all analytical reporting;
- Prohibited Practices;
- How and when to report data integrity issues;
- Record keeping. The training emphasizes the importance of proper written documentation on the part of the analyst with respect to those cases where analytical data may be useful, but are in one sense or another partially nonconforming;
- Training Program, including discussion regarding all data integrity procedures;
- Data integrity training documentation;
- In-depth procedures for data monitoring; and
- Specific examples of breaches of ethical behavior such as improper data manipulations, adjustments of instrument time clocks, and inappropriate changes in concentrations of standards.

All PAS personnel, including contract and temporary, are required to sign an "Attestation of Ethics and Confidentiality" at the time of hire and/or during annual refresher training or as specified in the ethics policy. This document clearly identifies inappropriate and questionable behavior. Violations of this document result in profound consequences, including prosecution and termination, if necessary.

Also see SOP-ENV-COR-POL-0004 *Code of Ethics and Professional Conduct* for more information.

5.2.2.1.4 Management System Documents Training

The Quality Manual policies, and SOPs are the documents used by regulatory bodies and Pace® customers to verify capability, competency, and compliance with their requirements and expectations.

In addition to on-the-job training, employees must have a signed Read and Acknowledgement Statement (R&A) on record for the quality manual, and the policies and SOPs relating to his/her job

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responsibilities. This statement, whether signed by the employee electronically or by wet signature, confirms that the employee has received, read, and understands the content of the document, that the employee agrees to follow the document when carrying out their work tasks; and the employee understands that unauthorized change to procedures in an SOP is not allowed except in accordance with the SOP departure policy (See 4. 9.1).

See SOP ENV-CORQ-0016 *Standard Operating Procedures and Standard Work Instructions* for more information.

5.2.2.1.5 Demonstration of Capability (DOC) Requirements

An initial demonstration of capability (IDOC) must be completed and validated prior to authorization for the employee to work independently on client samples for the test method. After successful IDOC, the employee must demonstrate continued proficiency (CDOC) for the test method on an annual basis. If more than a year has passed since the employee last performed the method; then capability must be re-established with an IDOC.

Successful DOC is one where the DOC replicate data has been compiled, reviewed, and verified by the employee's supervisor and/or manager to be complete and to have met acceptance criteria and the DOC record has been validated by quality personnel for completeness and compliance, and placed in the employee's training file for accessibility and reference.

Demonstration of capability (DOC) procedures and requirements vary by technology.

For example, a DOC for chemistry test methods where spiking is appropriate, is based on the employee's capability to achieve acceptable precision and accuracy for each analyte reported by the laboratory for the test method using the laboratory's test method SOP.

DOC procedures and requirements must be specified in the laboratory's test method SOP or a stand-alone SOP. Refer to these SOPs for more information.

5.2.2.1.6 Effectiveness of Training

Effectiveness of individual employee training is measured by their demonstrated ability to comprehend the training material and apply knowledge and skills gained to their job task. Measurements include but are not limited to:

- Testing of the employee's knowledge of the QMS, policies, and technical and administrative procedures through various mechanisms, such as quizzes, observation, and interviews.

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- Demonstrated ability to convey information correctly and factually in written and verbal communication to internal and external parties.
- Demonstrated ability to carry out tasks in accordance with SOPs and other work instructions.
- Demonstrated ability to make sound decisions based on guidance and information available.
- Demonstrated initiative to seek help or guidance when the employee is unsure of how to proceed.

5.2.2.2 Supplemental Learning

Supplemental learning objectives may be established for newly hired personnel to aid in their development of administrative and technical skills. These learning objectives and materials, referred to as Learning Plans (LP), are created and maintained by the PAS 3P program and managed by the employee's direct supervisor.

Pace® also offers a wide variety of supplemental learning courses that are made available to all employees for professional development. These learning materials, maintained by Pace® corporate training personnel, are accessed via the company's employee portal, PaceConnect. The learning may be self-initiated based on an employee's interest or may be assigned to the employee at the discretion of management as professional development as part of an employee's annual goals.

Supplemental learning courses and learning plan activities are not prerequisites for competency (Section 5.2.1.1) and are not considered part of the required PAS QMS training program.

5.2.3 Personnel Supervision

Every employee is assigned a direct supervisor, however named, who is responsible for their supervision.

General supervisory responsibilities may include but are not limited to:

- Hiring Employees
- Training Employees
- Performance Management
- Development, oversight, and execution of personnel training plans
- Monitoring personnel work product to assure the work is conducted in accordance with this quality manual, policies, SOPs, and other documents that support the QMS.

5.2.4 Job Descriptions

Job Descriptions that define the required education, qualifications, experience, skills, roles and responsibilities, and reporting relationships for each Pace® position are established by top management and kept by corporate HR. The job descriptions apply to employees who are

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directly employed by Pace®, part-time, temporary, technical, and administrative and by those that are under contract with Pace® through other means.

The job descriptions include the education, expertise, and experience required for the position and the responsibilities and duties, including any supervisory or managerial duties assigned to the position.

5.2.5 Authorization of Technical Personnel

Technical personnel are authorized by local quality personnel to perform the technical aspects of their position after quality personnel have verified that the employee meets the qualifications for the position, has successfully completed required training (Section 5.2.2.1), and the employee has completed initial demonstrated capability (Section 5.2.2.1.5). After initial authorization, technical personnel are expected to maintain a current and complete training record, demonstrate on-going capability at least annually for each test method performed, and produce reliable results through accurate analysis of certified reference materials, proficiency testing samples, and/or routine quality control samples in order to remain authorized to continue to perform their duties.

Records to support authorization including, education, experience, training, and other evaluations are kept by the location where the employee works.

5.3 Accommodations and Facilities

5.3.1 Facilities

PAS laboratories and service centers are designed to support the correct performance of procedures and to not adversely affect measurement integrity or safety. Access to PAS facilities is controlled by various measures, such as card access, locked doors, staffed main entry.

5.3.2 Environmental Conditions

Each location is equipped with energy sources, lighting, heating, and ventilation necessary to facilitate proper performance of calibrations and tests. The location ensures that housekeeping, electromagnetic interference, humidity, line voltage, temperature, sound, and vibration levels are appropriately controlled to ensure the integrity of specific measurement results and to prevent adverse effects on accuracy or increases in the uncertainty of each measurement.

Environmental conditions are monitored, controlled, and recorded as required by the relevant specifications, methods, and procedures. Operations are stopped if it is discovered that the environmental conditions would jeopardize the integrity of analytical results or other work product.

5.3.3 Separation of Incompatible Activities

The layout and infrastructure of each work area including air handling systems, power supplies, and gas supplies of each work area is specifically designed for the type of analytical activity performed. Effective separation between incompatible work activities is maintained. For example, sample storage, preparation, and chemical handling for volatile organic analysis (VOA) is kept separate from semi-volatile organic (SVOA).

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Samples known or suspected to contain high concentration of analytes are separated from other samples to avoid the possibility for cross-contamination. If contamination is found, the source of contamination is investigated and resolved in accordance with applicable SOPs.

5.3.4 Security

Security is maintained by controlled access to the building and by surveillance of work areas by authorized personnel. Access is controlled to each area depending on the required personnel, the sensitivity of the operations performed, and potential safety concerns.

5.3.5 Good Housekeeping

PAS locations must maintain good housekeeping practices in work areas to maintain a standard of cleanliness necessary for analytical integrity and personnel health and safety.

5.4 Test Methods

5.4.1 General Requirements

The laboratory uses test methods and procedures that are appropriate for the scope of analytical services the laboratory offers.

Instructions on the use and operation of equipment and sample handling, preparation, and analysis of samples are provided in SOPs. The instructions in SOPs may be supplemented with other documents including, but not limited to, standard work instructions (SWI), manuals, guides, project documents and reference documents.

These documents are managed using the procedures described in SOP ENV-SOP-CORQ-0015 *Document Management and Control* and SOP ENV-SOP-CORQ-0016 *Standard Operating Procedures and Standard Work Instructions.*

5.4.2 Method Selection

The test methods and protocols used by the laboratory are selected to meet the needs of the customer, are appropriate for the items tested, for the intended use of the data, and to conform with applicable federal, statutory, or program requirements.

The test methods offered by PAS are industry accepted methods published by international, regional, or national standards. Each PAS laboratory bases its procedure on the latest approved edition of a method unless it is not appropriate or possible to do so, or unless regulatory requirements specify otherwise.

The laboratory confirms that it can perform the test method and achieve desired outcome before analyzing samples (see section 5.4.5). If there is a change in the published analytical method, then the confirmation is repeated.

When a customer does not specify the test method(s) to be used, the laboratory may suggest test methods that are appropriate for the intended use of the data and the type of samples to be tested. The laboratory will also inform customers when test methods requested are considered inappropriate for their purpose and/or out of date. This discourse takes place during review of analytical service requests (See Section 4.4).

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5.4.3 PAS Developed Methods

A PAS developed method is a method developed from scratch (no published source method), a procedure that modifies the chemistry from the source method, or a procedure that exceeds the scope and application of the source method.

PAS developed methods must be validated prior to use (see section 5.4.5) and the procedure documented in a test method SOP.

The requirements for non-standard methods (Section 5.4.4) also apply to PAS developed methods.

5.4.4 Non-standard Methods

A non-standard method is a method that is not published or approved for use by conventional industry standards for the intended purpose of the data. Non-standard methods must be validated prior to use (see section 5.4.5) and the procedure developed and documented in a test method SOP.

At a minimum, the following information must be included in the procedure:

- Title / Identification of Method;
- Scope and Application;
- Description of the type of item to be analyzed;
- Parameters or quantities and ranges to be determined;
- Apparatus and equipment, including technical performance requirements;
- Reference standards and reference materials required;
- Environmental conditions required and any stabilization period needed; and
- Description of the procedure, including:
    - Affixing identification marks, handling, transporting, storing, and preparing of items;
    - Checks to be made before the work is started;
    - Verifying equipment function and, where required, calibrating and/or adjusting the equipment before each use;
    - Method of recording the observations and results;
    - Any safety measures to be observed;
    - Criteria and/or requirements for approval/rejection;
    - Data to be recorded and method of analysis and presentation; and
    - Uncertainty or procedure for estimating uncertainty.

Use of a non-standard method for testing must be agreed upon with the customer. The agreement, which is retained by the laboratory in the project record, must include the specifications of the client's requirements, the purpose of testing, and their authorization for use of the non-standard method.

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5.4.5 Method Validation

5.4.5.1 Validation Description

Validation is the process of confirmation and the provision of objective evidence that the stated requirements for a specific method/procedure are fulfilled.

The laboratory's requirements and procedures for method validation are outlined in SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification.*

5.4.5.2 Validation Summary

All test methods offered by the laboratory are validated before use to confirm the procedure works and the data and results achieved meet the goals for the method and repeated when there are major changes to the laboratory procedure.

Results of validation are retained are kept in accordance with method validation SOP and the corporate policy ENV-CORQ-POL-0013 *Record Management*.

5.4.5.3 Validation of Customer Need

The validation process includes review of accuracy, precision, sensitivity, selectivity, linearity, repeatability, reproducibility, robustness, and cross-sensitivity of the procedure against general customer needs to ensure the laboratory's procedure will meet those needs.

The following subsections explain some concepts as they are applied to chemistry. The applications of these same concepts may differ for other technologies such as microbiology, radiochemistry, whole effluent toxicity (WET), and asbestos or other validation concepts may apply to these disciplines. Refer to the laboratory's test method SOPs for more information.

5.4.5.3.1 Accuracy

Accuracy is the degree to which the result of a measurement, calculation, or specification conforms to the correct value or a standard. When the result recovers within a range from the known value (control limit); the result generated using the laboratory's test method SOP is considered accurate.

5.4.5.3.2 Precision

Precision refers to the closeness of two or more measurements to each other. It is measured by calculating the relative percent difference (RPD) or relative standard deviation (RSD) from results of separate analysis of the same sample. Precision provides information about repeatability, reproducibility, and robustness of the laboratory's procedure.

5.4.5.3.3 Limits of Detection (LOD) (Chemistry)

The LOD is the minimum result which can be reliably discriminated from a blank with a predetermined confidence level. The LOD

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establishes the limit of method sensitivity and is also known as the detection limit (DL) or the method detection limit (MDL).

Values below the LOD cannot be reliably measured and are not reported by the laboratory unless otherwise specified by regulatory program or test method.

The LOD is established during method validation and after major changes to the analytical system or procedure that affect sensitivity are made.

The laboratory's procedure for LOD determination is specified in SOP ENV-SOP-ASHE-0045 *Determination of LOD and LOQ*.

For chemistry methodology, the local SOP must comply with the current version of each of the following documents:

- EPA document EPA-821-R-16-006 *Definition and Procedure for the Determination of the Method Detection Limit;*
- 2016 TNI Standard V1M4; and
- TNI GUID-3-109-Rev. 0, *V1M4 2016 Standard Update Guidance on Detection and Quantitation.*

5.4.5.3.4 Limits of Quantitation (LOQ) and Reporting Limit (RL)

This section describes these concepts for chemistry. For non-chemistry technologies, such as microbiology, refer to laboratory SOPs.

The LOQ is the minimum level, concentration, or quantity of a target analyte that can be reported with a specified degree of confidence.

The LLOQ is the value of the lowest calibration standard included in the calibration curve. The LLOQ establishes the lower limit of quantitation; it is not the same concept as the LOQ, however, the LOQ and LLOQ may be the same value.

The LOQ and LLOQ represent quantitative sensitivity of the test method.

- The LOQ must always be equal to or greater than the LLOQ and the LLOQ must always be greater than the LOD.
- Any reported value (detect or non-detect) less than the LLOQ is a qualitative value.

The RL is the value to which the presence of a target analyte is reported as detected or not detected. The RL is project-defined based on project data quality objectives (DQO). In the absence of

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project specific requirements, the RL is usually set to the LOQ or the LLOQ.

The laboratory's procedures for LOQ determination must be specified in the same SOP for LOD determination, (See Section 5.4.5.3.3) The LLOQ for each method must be specified in the test method SOP.

5.4.5.3.5 Linearity

Linearity is a mathematical concept applied to calibration models that employ multiple points to establish a calibration range used for quantitative analysis. Linearity is measured differently based on the calibration model. In general, if linearity is demonstrated then the slope of the response of standards are sufficiently close to one another. The accuracy of the linear regression and non-linear curves is verified by checking percent error or relative standard error (RSE), which is the process of refitting calibration data back to the model to determine if the results are accurate. For linear curves that use average calibration or response factor, error is measured by relative standard difference (RSD).

Linearity also establishes the range of quantitation for the test method used which directly impacts the sensitivity of the test method and uncertainty in measurement results. As previously noted, the LLOQ establishes the lower limit of quantitation. Similarly, the upper range of linearity establishes the upper limit of quantitation. In general, results outside of this range are considered qualitative values. However, inorganic test methods sometimes allow for extension of the linear range above the upper limit of quantitation when accuracy at this value is verified.

Linearity can also be used to establish repeatability, reproducibility, and robustness of the laboratory's test method. When linearity is demonstrated using a specific calibration model during method validation, then use of this same calibration model to achieve linearity on a day-to-day basis confirms the laboratory's method is repeatable, reproducible, and robust.

5.4.5.3.6 Demonstration of Capability (DOC)

The DOC performed during method validation confirms that the procedure demonstrated acceptable precision and accuracy.

5.4.6 Measurement Uncertainty

The location provides an estimate of uncertainty in testing measurements with analytical results on request, or when required. For example, for radiochemistry uncertainty is always reported with the test result

For chemistry methodologies, the uncertainty of the test method is reflected in the control limits used to evaluate QC performance for the test method. (See 5.9.1.1.9). ISO/IEC states

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that when a well-recognized test method specifies limits to the values of the major source of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory has satisfied the requirements on analytical uncertainty by following the test method and reporting instructions.

When measurement uncertainty cannot be satisfied through control limits, the location will provide a reasonable estimation of uncertainty. A reasonable estimation is based on knowledge of method performance and previous experience. When estimating the analytical uncertainty, all uncertainty components which are of importance in the given situation are considered.

5.4.7 Control of Data

PAS has policies and processes in place to assure that reported data is free from calculation and transcription errors, that quality control is reviewed and evaluated before data is reported, and to address manual calculation and integration.

5.4.7.1 Calculations, Data Transfer, Reduction and Review

Whenever possible, calculations, transfer of data, and data reduction are performed using validated software programs (See 5.4.7.2).

If manual calculations are performed, the results of these calculations are verified during the data review process outlined in section 5.9.3.

5.4.7.1.1 Manual Integration

The PAS policy and procedures for manual integration are provided in corporate SOP ENV-SOP-CORQ-0006 *Manual Integration*.

This SOP includes the conditions under which manual integration is allowed and the requirements for documentation.

Required documentation of manual integration includes:

- complete audit trail to permit reconstruction of before and after results;
- identification of the analyst that performed the integration and the reason the integration was performed; and
- identification of the individual(s) that reviewed the integration and verified the integration was done and documented in compliance with the SOP.

5.4.7.2 Use of Computers and Automated Acquisition

Whenever possible, PAS uses software and automation for the acquisition, processing, recording, reporting, storage, and/or retrieval of data.

Software applications developed by PAS are validated by corporate IT for adequacy before release for routine use. Commercial off the shelf software is considered sufficiently validated when the location follows the manufacturer or vendor's manual for set-up and use. Records of validation are kept by the corporate information technology (IT) group or by the group that performed the validation.

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The PAS process for the protection of data stored in electronic systems includes:

- Individual usernames and passwords for Laboratory Information Management Systems (LIMS) and auxiliary systems used to store or process data.
- Employee Training in Computer Security Awareness
- Validation of spreadsheets used for calculations to verify formulas and logic yield correct results and protection of these cells to prevent unauthorized change.
- Operating system and file access safeguards
- Protection from Computer Viruses
- Regular system backup; and testing of retrieved data
- Verification the software application works as expected and is adequate for use and fulfills compliance requirements, such as the need to record date/time of data generation.
- Change control to assure requests for changes are reviewed and approved by management before the change is made.
- Communication channels to assure all staff are aware of changes made.
- Version Control and maintenance of historical records.

5.5 Equipment

5.5.1 Availability of Equipment

Each PAS location is furnished with all equipment and instrumentation necessary to correctly perform the tests offered in compliance with the specifications of the test method and to achieve the accuracy and sensitivity required.

When a regulation, program, or reference test method requires Class A glassware for quantitative measurements, only Class A glassware may be used. Plastic graduated cylinders, even if marketed by the vendor as comparable to Class A glassware, may not be used when Class A glassware is specified because ASTM's definition and tolerances for Class A glass cannot be applied to other materials.

5.5.2 Calibration

Equipment and instrumentation are checked prior to use to verify it performs within tolerance for its intended application.

5.5.2.1 Support Equipment

The location confirms support equipment is in proper working order, uniquely identified, and meets the specifications for use prior to placement in service. Periodic checks are performed to verify tolerance and accuracy are performed thereafter in accordance with a support equipment maintenance scheduled maintained by local quality personnel. Equipment that does not meet specifications is removed from

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service until repaired or replaced. Records of repair and maintenance activities are maintained.

Procedures used to conduct and record these checks are outlined in SOP ENV-SOP-ASHE-0048 *Support Equipment*.

5.5.2.2 Analytical Instruments

Analytical instruments are checked prior to placement in service in accordance with SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification*. After the initial service date, the calibration of instruments and verification calibration is performed in accordance with local test method SOPs.

The calibration procedures in the test method SOPs comply with the requirements for acceptable calibration practices outlined in corporate policy ENV-POL-CORQ-0005 *Acceptable Calibration Practices*, the reference methods, and any applicable regulatory or program requirements.

5.5.3 Equipment Use and Operation

Equipment is operated and maintained by personnel that are trained on the test method SOP. Up-to-date instructions and procedures for the use and maintenance of analytical equipment are included in SOPs and/or supplemental documents such as standard work instructions (SWI) or instrument manuals which are made readily accessible in the work area to all laboratory personnel.

5.5.4 Equipment Identification

Each piece of equipment must be uniquely identified by serial number or any other unique ID system. The identifier is included in the equipment list maintained by the quality department and may not be reused or used interchangeably. New equipment and replacement equipment must be assigned a new unique ID.

5.5.5 Equipment Lists and Records

5.5.5.1 Equipment List

Each PAS location maintains a list of equipment that includes information about the equipment including a description, manufacturer, serial number, date placed in service, condition when received, identity, and the work area where the equipment is used. The date of purchase is tracked by the procurement record. The equipment list(s) for each location covered by this manual is provided in Appendix E.

5.5.5.2 Equipment Records

In addition to the equipment list, the location maintains records of equipment that include:

- Verification that equipment conforms with specifications.
- Calibration records including dates, results, acceptance criteria, and next calibration dates.
- Maintenance plan and records

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- Records of damage, malfunction, or repair

The laboratory follows an equipment maintenance program designed to optimize performance and to prevent instrument failure which is described in SOP ENV-SOP-ASHE-0061 *Preventative, Routine, & Non-routine Maintenance* or in individual test method SOPs.

The maintenance program includes routine maintenance activities which are performed as recommended by the manufacturer at the frequency recommended and non-routine maintenance, which is performed to resolve a specific problem such as degradation of peak resolution, shift in calibration relationship, loss of sensitivity, or repeat failure of instrument performance checks and quality control samples.

Maintenance is performed by PAS personnel or by outside service providers.

All maintenance activities performed by PAS personnel are recorded by the individual(s) that performed the activity at the time the maintenance was performed in an instrument maintenance log.

The maintenance record minimally includes the date of maintenance, the initials of the person(s) performing maintenance, a description of the activity performed, why (when the maintenance is non-routine), and the return to analytical control. When maintenance is performed by an external vendor, the service must be maintained and accessible for easy retrieval. The location must provide personnel with unrestricted access to instrument maintenance logs in order to promote good instrument maintenance and recordkeeping practices.

If an instrument must be moved, the location will use safe practices for handling and transport to minimize damage and contamination.

5.5.6 Out of Service Protocol

Equipment that has been subjected to overloading, mishandling, gives suspect results, has been shown to be defective, or is performing outside of specified limits is taken out of service and either removed from the work area or labeled to prevent accidental use until it has been repaired and verified to perform correctly.

When analytical equipment is taken out of service because it no longer meets tolerance specifications, the potential effect of the nonconformance may have had on previously reported analytical results should be evaluated. (See section 4.9).

5.5.7 Calibration Status

The location labels support equipment to indicate calibration status, whenever practicable or otherwise maintains the calibration status in a visible location in the work area. These procedures are described in SOP ENV-SOP-ASHE-0048 *Support Equipment*.

The calibration status of analytical instruments is documented in the analytical record. Analysts verify on-going acceptability of calibration status prior to use and with instrument performance check standards. These procedures are described in test method SOPs.

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5.5.8 Returned Equipment Checks

When equipment or an instrument is sent out for service, the location using the equipment ensures that the function and calibration status of the equipment is checked and shown to be satisfactory before the equipment is returned to service.

5.5.9 Intermediate Equipment Checks

The location performs intermediate checks on equipment to verify the on-going calibration status. For example, most test methods require some form of continuing calibration verification check, and these procedures are included in the test method SOP. Periodic checks of support equipment are also performed; see SOP ENV-SOP-ASHE-0048 *Support Equipment* for more information.

5.5.10 Safeguarding Equipment Integrity

The location safeguards equipment integrity using a variety of mechanisms that include but are not limited to:

- Adherence to manufacturer's specification for instrument use so that settings do not exceed manufacturer's recommendation or stress the performance of the equipment.
- Established maintenance programs.
- Transparent maintenance records and unrestricted access to maintenance logs.
- Validation and approval of software before use.
- Audits to confirm instrument settings are consistent with SOPs.
- On-the-job training for safe and proper use of laboratory equipment.

5.6 Measurement Traceability

5.6.1 General

Measurement traceability refers to a property of a measurement result whereby the result can be related to a reference through an unbroken chain of calibration, each contributing to the measurement uncertainty. Traceability requires an established calibration hierarchy of equipment (instruments) used during testing including equipment used for subsidiary measurements. The location assures this equipment is calibrated prior to being put into service and that the reference standard and materials used for calibration are traceable to the international standard of units (SI) or national measurement standard.

When strict traceability to SI units cannot be made, the location establishes traceability with the use of reference standards and equipment obtained from competent suppliers that provide calibration certificates and/or certificates of analysis (COA).

5.6.2 Equipment Correction Factors

When correction factors are used to adjust results the PAS personnel will assure that results in computer software are also updated.

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5.6.3 Specific Requirements

5.6.3.1 Requirements for Calibration Laboratories

The laboratory does not offer calibration services to customers; therefore, ISO/IEC and TNI requirements for calibration laboratories do not apply.

5.6.3.2 Requirements for Testing Laboratories

The laboratory has procedures in place to verify equipment is calibrated prior to being put into service (See 5.5.2) and ensures the reference standard and materials used for calibration are traceable to the international standard of units (SI) or national measurement standard. When strict traceability to SI units cannot be made, the laboratory establishes traceability with the use of reference standards and equipment obtained from competent suppliers that provide calibration certificates and/or certificates of analysis (COA).

5.6.4 Reference Standards and Reference Materials

5.6.4.1 Reference Standards

The laboratory uses reference standards of measurement to verify adequacy of working weights and thermometers. The working weights are the weight(s) used for daily balance calibration checks and the working thermometers are used for daily temperature measurements.

Working weights and thermometers must be periodically checked to verify on-going adequacy for use between calibrations performed by an external calibration laboratory using reference standards traceable to SI or a national standard and that are used solely for verification purposes.

For example:

- An acceptable reference standard to check working thermometers against include a NIST Certified Thermometer or a NIST Traceable Thermometer that is not used for any other purpose than to check the adequacy of the working thermometer.
- An acceptable reference standard for the working weights is a set of Class S weights that is not used for any other purpose than to verify the weights used daily.

The working weights must be checked against the reference standard annually and all weight sets must be recertified by an ISO accredited calibration body every 5 years. In this application, "annually" means within thirteen (13) months from the date of the last check.

Working thermometers must be checked against the reference thermometer prior to placement in service to establish a correction factor (CF)[1] and then re-checked annually (±13 months from date of last check) or if battery operated, every three (3) months (±100 days from date of last check).

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Exceptions to the 3-month recheck for battery operated sensors are allowed when the sensor is embedded in a unit and the manufacturer/vendor has evidence to show that the accuracy of the sensor is not affected by battery life.

Liquid in Glass NIST Certified reference thermometers must be recertified by an ISO/IEC accredited calibration laboratory every 5 years. If the reference thermometer is NIST Traceable or is a digital NIST Certified thermometer, the reference thermometer must be recertified annually by an ISO/IEC 17025 accredited calibration laboratory or service provider that provides traceability to a national standard.

If criteria for the intermediate checks or recertification is not acceptable, the impact on previously reported results is evaluated using the process for evaluation of nonconforming work (See 4.9).

See SOP ENV-SOP-ASHE-0048 *Support Equipment* for more information.

5.6.4.2 Reference Materials

The location purchases chemical reference materials (also known as stock standards) from vendors that are accredited to ISO 17034 or Guide 34. Purchased reference materials must be received with a Certificate of Analysis (COA) where available. If a reference material cannot be purchased with a COA, it must be verified by analysis and comparison to a certified reference material and/or there must be a demonstration of capability for characterization. COA are reviewed for adequacy and retained by the laboratory for future reference.

All prepared standards, reference materials, and reagents are verified to meet the requirements of the test method through routine analyses of quality control samples.

The laboratory procedure for traceability and use of these materials is provided in SOP ENV-SOP-ASHE-0059 *Laboratory Supply Procedure*.

This SOP includes each of the following requirements:

- Procedures for documentation of receipt and tracking. The record of entry includes name of the material, the lot number, receipt date, and expiration date.
- Storage conditions and requirements. Reference materials must be stored separately from samples, extracts, and digestates.
- Requirements to assure that preparations of intermediate or working solutions are recorded and assigned a unique identification number for tracking. Records of preparation include the lot number of the stock standard(s) used, the type and lot number of the solvent, the formulation, date, expiration date, and the preparer's initials. The lot number of the working standards is recorded in the analytical record to provide traceability to the standard preparation record. The preparation record provides traceability to the COA, which is traceable to SI or the national measurement standard.
- A requirement that the expiration dates of prepared standards may not exceed the expiration date of the parent standard. Standards, reference materials, and

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reagents are not used after their expiration dates unless it is not possible to procure a new standard and the reliability of the expired material is verified and documented by the location using a procedure approved by corporate quality personnel. Otherwise, the expired material is promptly removed from the work area or clearly labeled as acceptable for qualitative/troubleshooting purposes only.

- The second source materials used for verification of instrument calibration are obtained from a different manufacturer or may be a different lot from the same manufacturer.
- Procedures to check reference materials for degradation and replacement of material if degradation or evaporation is suspected.
- Procedures for labeling. At a minimum, the container must identify the material, the ID of the material and the expiration date. Original containers should also be labeled with date opened.

5.6.4.3 Intermediate Checks

Checks to confirm the calibration status of reference standards and materials must be included in test method SOPs. These checks include use of second source standards and reference materials reserved only for the purpose of calibration checks.

5.6.4.4 Transport and Storage

The location handles and transports reference standards and materials in a manner that protects the integrity of the materials. Reference standard and material integrity is protected by separation from incompatible materials and/or minimizing exposure to degrading environments or materials. Standards and reference materials are stored separately from samples, extracts, and digestates. All standards are stored according to the manufacturer's recommended conditions. Temperatures colder than the manufacturer's recommendation are acceptable if it does not compromise the integrity of the material (e.g., remains in liquid state and does not freeze solid). In the event a standard is made from more than a single source with different storage conditions, the standard will be stored according to the conditions specified in the analytical method.

See the applicable analytical SOPs for specific reference material storage and transport protocols.

5.7 Sampling

Sampling refers to the field collection of samples and to subsamples taken by the laboratory for analysis from the field collected sample.

Subsampling procedures are included in each test method SOP or a stand-alone SOP to assure the aliquot used for testing is representative of the field collected sample.

The requirements in the following subsections apply when field sampling is performed by PAS.

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5.7.1 Sampling Plans and SOPs

When PAS performs field collection of samples, sampling is carried out in accordance with a written sampling plan and sampling SOPs. These documents are made readily accessible at the sampling location. Sampling plans and SOPs are, whenever reasonable, based on appropriate governing methods and address the factors to be controlled to ensure the validity of the analytical results.

5.7.2 Customer Requested Deviations

When the customer requires deviations, additions, or exclusions from the documented sampling plan and/or procedure, the laboratory records the client's change request in detail with the sampling record, communicates the change to sampling personnel, and includes this information in the final test report.

5.7.3 Recordkeeping

PAS assures the sampling record includes the sampling procedure used, any deviations from the procedure, the date and time of sampling, the identification of the sampler, environmental conditions (if relevant), and the sampling location.

5.8 Sample Management & Handling

5.8.1 Procedures

The location's procedures for sample management and handling are outlined in SOP ENV-SOP-ASHE-0030 S*ample Management*.

The procedures in these SOPs are established to maintain the safe handling and integrity of samples from transport, storage, to disposal and during all processing steps to maintain client confidentiality, and to protect the interests of PAS and its customers.

5.8.1.1 Chain of Custody

All samples received by the location must be accompanied with a Chain of Custody (COC) record. The COC provides information about the samples collected and submitted for testing and documents the possession of samples from time of collection to receipt by the location.

The COC record must minimally include the following information:

- Client name, address, phone number;
- Project Reference;
- Client Sample Identification (Client ID);
- Date, Time, and Location of Sampling;
- Sampler's Name or Initials;
- Matrix;
- Type of container, and total number collected for each sample;
- Preservatives;

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- Analyses Requested;
- Mode of collection;
- Any special instructions; and
- The date and time and signature of each sample transfer from time of collection to receipt in the location. When the signature field on CoC includes company. Personnel relinquishing and/or receiving samples are expected to record this information. When the COC is transported inside the cooler, independent couriers do not sign the COC and the shipping manifests and/or air bills are the records of possession during transport. The shipping manifest must be retained as part of the COC record and included in the test report when required (See Section 5.10.3).

A complete and legible COC is required. If the location observes that the COC is incomplete or illegible, the client is contacted for resolution. The COC must be filled out in indelible ink. Personnel correct errors by drawing a single line through the initial entry, so the entry is not obscured, entering the correct information, and initialing, and dating the change.

5.8.1.2 Legal Chain of Custody

Legal chain of custody is a chain of custody protocol used for evidentiary or legal purposes. The protocol is followed by the location when requested by customer or when mandated by a regulatory program.

Legal chain of custody (COC) protocol establishes an intact, continuous record of the physical possession*, storage, and disposal of "samples" which includes sample aliquots, and sample extracts/digestates/distillates.

Legal COC records account for all time periods associated with the samples and identifies all individuals who physically handled individual samples. Legal COC begins at the point established by legal authority, which is usually at the time the sample containers are provided by the location for sample collect or when sample collection begins.

*A sample is in someone's custody if:

- It is in one's physical possession;
- It is in one's view after being in one's physical possession;
- It has been in one's physical possession and then locked or sealed so that no one can tamper with it; and/or
- It is kept in a secure area, restricted to authorized personnel only.

Refer to SOP ENV-SOP-ASHE-0030 S*ample Management* for more information.

5.8.2 Unique Identification

Each sample is assigned a unique identification number (Lab ID) after the sample has been checked and accepted by PAS in accordance with the PAS sample acceptance policy (See 5.8.3). The Lab ID is affixed to the sample container using a durable label.

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The unique identification of samples also applies to subsamples, and prepared samples.

The lab ID is linked to the field ID (client ID) in the receipt and log-in record. Both IDs are linked to the testing activities performed on the sample and the documentation records of the test.

Also see 5.8.4.

5.8.3 Sample Receipt Checks and Sample Acceptance Policy

The location checks the condition and integrity of samples on receipt and compares the labels on the sample containers to the COC record. Any problem or discrepancy is recorded. If the problem impacts the suitability of the sample for analysis or if the documentation is incomplete, the client is notified for resolution. Decisions and instructions from the client are maintained in the project record.

5.8.3.1 Sample Receipt Checks

The following checks are performed:

- Verification that the COC is complete and legible.
- Verification that each sample's container label includes the client sample ID, the date and time of collection and the preservative in indelible ink.
- The container type and preservative are appropriate for each test requested.
- Adequate volume is received for each test requested.
- Visual inspection for damage or evidence of tampering.
- Visual inspection for presence of headspace in VOA vials. (VOA = volatile organic analysis).
- Thermal Preservation: For chemical testing methods for which thermal preservation is required, temperature on receipt is typically considered acceptable if the measurement is above freezing but <6°C unless otherwise specified by federal, statutory, program or test method requirements. Refer to the location's SOP for sample receipt for specific thermal preservation requirements.

  For samples that are hand-delivered to the location immediately after sample collection, there must be evidence that the chilling process began immediately after sample collection and prior to delivery of the samples to the laboratory or service center, such as arrival of the samples on ice.
- Chemical Preservation
- Holding Time: Sample receiving personnel are trained to recognize tests where the holding time is 48 hours or less and to expedite the log-in of these samples. Except for tests with immediate holding times (15 minutes from time of collection or less), when samples are received out of hold, the location will notify the client and request instruction. If the decision is made to proceed with analysis, the final test report will include notation of this instruction.

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5.8.3.2 Sample Acceptance Policy

PAS maintains a sample acceptance policy in accordance with regulatory guidelines to clearly establish the circumstances in which sample receipt is accepted or rejected.

When receipt does not meet criteria for any one of these conditions, the location must document the noncompliance, contact the customer, and either reject the samples or fully document any decisions to proceed with testing. In accordance with regulatory specifications, test results associated with receipt conditions that do not meet criteria are qualified in the final test report.

All samples received must meet each of the following criteria:

- Be listed on a complete and legible COC;
- Be received in properly labeled sample containers;
- Be received in appropriate containers that identify preservative;
- The COC must include the date and time of collection for each sample;
- The COC must include the test method requested for each sample;
- Be in appropriate sample containers with clear documentation of the preservatives used;
- Be received within holding time. Any samples received beyond the holding time will not be processed without prior customer approval;
- Have sufficient sample volume to proceed with the analytical testing. If insufficient sample volume is received, analysis will not proceed without customer approval; and
- Be received within appropriate temperature ranges unless program requirements or customer contractual obligations mandate otherwise.

  Samples that are delivered to the location immediately after collection are considered acceptable if there is evidence that the chilling process has been started. For example, by the arrival of the samples on ice. If samples arrive that are not compliant with these temperature requirements, the customer will be notified. The analysis will NOT proceed unless otherwise directed by the customer. If less than 72 hours remain in the hold time for the analysis, the analysis may be started while the customer is contacted to avoid missing the hold time. Data associated with any deviations from the above sample acceptance policy requirements will be appropriately qualified.

5.8.4 Sample Control and Tracking

The samples are controlled and tracked using the Laboratory Information Management System (LIMS). The LIMS stores information about the samples and project. The process of entering information into the LIMS is called log-in and these procedures are described in SOP ENV-SOP-ASHE-0030 S*ample Management*. After log-in, a label is generated and affixed to

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each sample container. Information on this label, such as the lab ID, links the sample container to the information in LIMS.

At a minimum, the following information is entered during log-in:

- Client Name and Contact Information;
- The laboratory ID linked to the client ID;
- Date and time of sample collection;
- Date and time of sample receipt;
- Matrix; and
- Tests Requested.

5.8.5 Sample Storage, Handling, and Disposal

The location procedures for sample storage, handling and disposal are detailed in SOPs ENV-SOP-ASHE-0030 S*ample Management.*

5.8.5.1 Sample Storage

The samples are stored according to method and regulatory requirements as per test method SOPs. Samples are stored away from all standards, reagents, or other potential sources of contamination and stored in a manner that prevents cross contamination. Volatile samples are stored separately from other samples. All sample fractions, extracts, leachates, and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

Refrigerated storage areas are maintained at ≤6°C (but not frozen) and freezer storage areas are maintained at <-10°C, unless otherwise required per method or program. The temperature of each storage area is checked and documented at least once for each day of use. If the temperature falls outside the acceptable limits, then corrective actions are taken and appropriately documented.

The location is operated under controlled access protocols to ensure sample and data integrity. Visitors must register at the front desk and be properly escorted while on-site. Samples are taken to the appropriate storage location immediately after sample receipt and log-in procedures are completed. All sample storage areas have limited access. Samples are removed from storage areas by designated personnel and returned to the storage areas as soon as possible after the required sample quantity has been taken.

5.8.5.2 Sample Retention and Disposal

The procedures used by the location for sample retention and disposal are detailed in SOP ENV-SOP-ASHE-0030 S*ample Management* and ENV-SOP-ASHE-0014 *Waste Handling and Management.*

In general, unused sample volume and prepared samples such as extracts, digestates, distillates and leachates (samples) are retained by the location for the timeframe necessary to protect the interests of the location and the customer.

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Samples may be stored at ambient temperature when all analyses are complete, the hold time is expired, the report has been delivered, and/or when allowed by the customer or program. Samples requiring storage beyond the minimum sample retention time due to special requests or contractual obligations may be stored at ambient temperature unless the location has a capacity, and their presence does not compromise the integrity of other samples.

After this period expires, non-hazardous samples are properly disposed of as non-hazardous waste. The preferred method for disposition of hazardous samples is to return the excess sample to the customer.

5.9 Assuring the Quality of Test Results

5.9.1 Quality Control (QC) Procedures

The location monitors the validity and reliability of test results using quality control (QC) samples that are prepared and analyzed concurrently with field samples in the same manner as field samples. QC results are always associated to and reported with the field samples they were prepared and analyzed with from the same preparation or analytical batch. See the glossary for definition of preparation and analytical batch.

The results of QC performed during the testing process are used by the location to assure the results of analysis are consistent, comparable, accurate, and/or precise within a specified limit. When the results are not within acceptance criteria or expectations for method performance, correction and corrective action(s) are taken. These actions may include retesting or reporting of data with qualification to alert the end user of the situation.

Other QC measures performed include the use of certified reference materials (see 5.6.4), participation in interlaboratory proficiency testing (see 5.9.1.2), verification that formulae used for reduction of data and calculation of results is accurate (see 5.9.3), on-going monitoring of environmental conditions that could impact test results (see 5.3.2), and evaluation and verification of method selectivity and sensitivity (see 5.4.5).

QC results are also used by the location to monitor performance statistical trends over time and to establish acceptance criteria when no method or regulatory criteria exist. (See 5.9.1.1.9)).

5.9.1.1 Essential QC

Although the general principles of QC for the testing process apply to all testing, the QC protocol used for each test depends on the type of test performed.

QC protocol used by the location to monitor the validity of the test are specified in test method SOPs. The SOP includes QC type, frequency, acceptance criteria, corrective actions, and procedures for reporting of nonconforming work.

These requirements in the SOP conform to the reference method and any applicable regulations or certification and accreditation program requirement for which results of the test are used. When a project requires more stringent QC protocol than specified in the SOP, project specification is followed. When the project requires less stringent QC protocol, the project specification may be followed as an authorized departure from the SOP when the project specifications meet the requirements in the

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mandated method and any regulatory compliance requirements for which the data will be used.

The following are examples of essential QC for chemistry. These concepts may not apply to other technologies and disciplines such as microbiology, radiochemistry, whole effluent toxicity, and/or asbestos. For essential QC for these disciplines, refer to test method SOPs.

5.9.1.1.1 Second Source Standard (ICV/QCS)

The second source standard is a standard obtained from a different vendor than the vendor of the standards used for calibration, or from a different lot from the same vendor, when only one vendor is available. It is a positive control used to verify the accuracy of instrument calibration relative to the purity of the standards used for calibration. This check may be referred to in published test methods and quality system standards as the initial calibration verification (ICV) or a quality control sample (QCS). The second source standard is analyzed immediately after the calibration and before analysis of any samples. When the ICV is not within acceptance criteria, a problem with the purity or preparation of the standards may be indicated. The source of the problem should be investigated and corrected prior to further use of the calibration/instrument for sample analysis.

5.9.1.1.2 Continuing Calibration Verification (CCV)

The CCV is used to determine if the analytical response has significantly changed since calibration. If the response of the CCV is within criteria, the calibration is considered valid. If not, there is a problem that requires further investigation and correction. Actions taken are technology and method specific.

5.9.1.1.3 Method Blank (MB) / Other Blanks

The MB is a negative control used to assess for contamination during the prep/analysis process. The MB consists of a clean matrix, similar to the associated samples that is known to be free of analytes of interest. The MB, unless otherwise specified by the test method, is processed with, and carried through all preparation and analytical steps as the associated samples.

The criteria used to assess for contamination depends on the intended use of data. In general, detections in the MB above the RL or ½ the RL indicate contamination. When contamination is evident, the source is investigated, and corrections are taken to reduce or eliminate it. Analytical results associated with MB that does not meet criteria are qualified in the final test report.

Other types of blanks that serve as negative controls in the process may include:

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- Trip Blanks (VOA)
- Storage Blanks
- Equipment Blanks
- Field Blanks
- Calibration Blanks
- Cleanup Blanks
- Instrument Blanks

5.9.1.1.4 Laboratory Control Sample (LCS)

The LCS is a positive control used to measure the accuracy of process in a blank matrix. The LCS is spiked by the laboratory with a known amount of analyte. The spike is a standard solution that is pre-made or prepared from a certified reference standard. Like the MB, unless otherwise specified in the test method, the LCS is processed with and carried through all preparation and analytical steps as the associated samples.

When the percent recovery (%R) of the LCS is within the established control limit, sufficient accuracy has been achieved. If not, the source of the problem is investigated and corrected, and the procedure may be repeated. Analytical results associated with LCS that does not meet criteria are qualified in the final test report.

5.9.1.1.5 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

The MS and MSD are replicates of a client sample that is spiked with known amount of target analyte. Matrix spikes measure the effect the sample matrix has on precision and accuracy of test results.

Matrix spike results mostly provide information on the effect of the matrix to the client whose sample was used and on samples of the same matrix from the same sampling site, during the same sampling event. Consequently, matrix spikes should be client designated. When there is not a client-specified MS for any sample in the batch, the location randomly selects a sample from the batch; the sample selected at random is called a "batch" matrix spike.

The MS/MSD results for percent recovery and relative percent difference are checked against control limits. However, because the performance of matrix spikes is matrix-dependent and specific to the customer whose sample was used as the MS/MSD, the results of matrix spikes are not used for quality control on the batch.

5.9.1.1.6 Sample Duplicate (SD)

A sample duplicate is a second replicate of sample that is used to measure precision.

The relative percent difference between replicates are evaluated against the established acceptance criteria for relative percent

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difference (RPD) when this criterion is applicable. If RPD is not met, associated test results are reported with qualification.

5.9.1.1.7 Surrogates

Surrogates are compounds that mimic the chemistry of target analytes but are not expected to occur naturally in real world samples. Surrogates are added to each sample and matrix QC samples (MS, MSD, SD) at known concentration to measure the impact of the matrix on the accuracy of method performance. Surrogates are also added to the positive and negative control samples (MB, LCS) to evaluate performance in a clean matrix, and included in the calibration standards and calibration check standards.

The percent recovery of surrogates is evaluated against method-specified limits or statistically derived in-house limits. Project-specific limits and/or program-specific limits are used when required. Results with surrogate recovery out of limits in samples are reported with qualification. Samples with surrogate failures can also be re-extracted and/or re-analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systematic error.

5.9.1.1.8 Internal Standards

Internal Standards are compounds not expected to occur naturally in field samples. They are added to every standard and sample at a known concentration prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. The location follows specific guidelines for the treatment of internal standard recoveries and further information can be found in the applicable test method SOP.

5.9.1.1.9 QC Acceptance Criteria and Control Limits

The QC acceptance criteria are specified in test method SOPs. The criteria in the SOP are based on the requirements in the published test method or regulatory program. When there are no established acceptance criteria, the location develops acceptance criteria in accordance with recognized industry standards.

Some methods and programs require the location to establish control limits for LCS, MS/MSD, and surrogate evaluation using historical data. PAS developed limits are referred to as "in-house" control limits. In-house control limits represent ± 3 Standard Deviations (99% confidence level) from the average recovery of at least 20 data points generated using the same preparation and analytical procedure in a similar matrix.

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See SOP ENV-SOP-ASHE-0054 *Control Chart Generation and Analysis* for more information about the procedures used to establish in-house control limits.

5.9.1.2 Proficiency Testing (PT)

PAS locations participate in interlaboratory proficiency testing (PT) studies to measure performance of the test method and to identify or solve analytical problems. PT samples measure location performance through the analysis of unknown samples provided by an external source.

The frequency of PT participation is based on the certification and accreditation requirements held by the laboratory. The PT samples are obtained from accredited proficiency testing providers (PTP) and treated as field samples which means they are included in the location's normal analytical processes and do not receive extraordinary attention due to their nature.

PAS locations do not share PT samples with other PAS locations, does not communicate with other PAS locations regarding current PT sample results during the duration of the study, and does not attempt to obtain the assigned value of any PT sample from the PT provider.

PT results scored unacceptable are investigated and correction action taken, when necessary.

Refer to corporate policy ENV-POL-CORQ-0002 *PT Policy* for more information.

5.9.2 QC Corrective Action

When the results of QC are not within acceptance criteria or expectations for method performance, correction and corrective action(s) are taken per the specifications in the test method SOP. These actions may include retesting or reporting of data with qualification to alert the end user of the situation.

5.9.3 Data Review

PAS locations use a tiered system for data review. The tiered process provides sequential checks to verify data transfer is complete; manual calculations, if performed, are correct, manual integrations are appropriate and documented, calibration and QC requirements are met, appropriate corrective action was taken when required, test results are properly qualified, process and test method SOPs were followed, project specific requirements were met, when applicable, and the test report is complete.

The sequential process includes three tiers referred to as primary review, secondary review, and administrative/completeness review.

Detailed procedures for the data review process are described in SOP ENV-SOP-ASHE-0056 *Data Review Process*. The general expectations for the tiered review process are described in the following sections:

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5.9.3.1 Primary Review

Primary review is performed by the individual that performed the task. All PAS personnel are responsible for review of their work product to assure it is complete, accurate, documented, and consistent with policy and SOPs.

Checks performed during primary review include but are not limited to:

- Verification that data transfer and acquisition is complete
- Manual calculations, if performed, are documented and accurate
- Manual integrations, if performed, are documented, and comply with SOP ENV-SOP-CORQ-006 *Manual Integration*
- Calibration and QC criteria were met, and/or proper correction and corrective actions were taken, and data and test results associated with QC and criteria exceptions are properly qualified
- Work is consistent with SOPs and any other relevant instructional document such as SWI, program requirements, or project QAPP

5.9.3.2 Secondary Review

Secondary review is performed by a qualified peer or supervisor. Secondary review is a repeat of the checks performed during primary review by another person. In addition to the checks of primary review, secondary review includes chromatography review to check the accuracy of quantitative analyte identification.

5.9.3.3 Completeness Review

Completeness review is an administrative review performed prior to release of the test report to the customer. Completeness review verifies that the final test report is complete and meets project specification. This review also assures that information necessary for the client's interpretation of results are explained in the case narrative or footnoted in the test report.

5.9.3.4 Data Audits

Test reports may be audited by local quality personnel to verify compliance with SOPs and to check for data integrity, technical accuracy, and compliance with the PAS QMS and any applicable federal, statutory, and program requirements. The reports chosen for the data audits are selected at random and these audits are not usually done prior to issuance of the test report to the customer.

If any problems with the data or test results are found during the data audit, the impact of the nonconforming work is evaluated using the process described in Section 4.9.

Also see Section 4.14 for internal audits.

5.9.4 Calibration Certificates

PAS does not perform calibration activities for its customers and calibration certificates are not offered or issued.

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5.9.5 Opinions and Interpretations

The location provides objective data and information to its customers of sufficient detail for their interpretation and decision making. Objective data and information are based solely on fact and does not attempt to explain the meaning (interpret) or offer a view or judgement (opinion). Sometimes the customer may request the location provide opinion or interpretation to assist them with their decisions about the data.

When opinions and interpretations are included in the test report, the location will document the basis upon which the opinions and interpretations have been made and clearly identify this content as opinion or interpretation in the test report.

Examples of opinion and interpretation include but are not limited to:

- A viewpoint on how a nonconformance impacts the quality of the data or usability of results.
- Recommendations for how the customer should use the test results and information.
- Suggestions or guidance to the customer for improvement.

5.9.6 Subcontractor Reports

When analytical work has been subcontracted to an organization external to PAS, the test report from the subcontractor is included in its entirety as an amendment to the final test report.

Test results performed by multiple locations within the PAS network (internal subcontracting) may be merged into a single test report so long as the test report issued clearly identifies the location and address of each network location that performed testing, and which tests each PAS location performed. (See 5.10.2)

5.9.7 Electronic Transmission of Results

When test results and/or reports are submitted to the customer through electronic transmission, the procedures established in this manual for confidentiality and protection of data apply.

5.9.8 Format of Test Reports

The test formats offered by PAS are designed to accommodate each type of analytical test method performed and to minimize the possibility of misunderstanding or misuse of analytical results. The format of electronic data deliverables (EDD) follows the specifications for the EDD.

5.9.9 Amendments to Test Reports

Test reports that are revised or amended by the location after date of release of the original final test report to the customer are issued as a new test report that is clearly identified as an amendment or revision and that includes a reference to the originally issued final test report.

The customer is the organization doing business with PAS external to PAS.

Changes made to test results and data before the final test report is issued to the customer are not amendments or revisions, these are corrections to errors found during the location's data verification and review process.

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The procedure for report amendments and revision are outlined in SOP ENV-SOP-ASHE-0035 *Final Report and Data Deliverable Contents*.

5.10 Reporting

5.10.1 General Requirements

PAS offers a wide variety of test report formats to meet project needs of Pace® customers and that comply with federal and state regulatory programs.

The type and level of deliverable, including the electronic data deliverable (EDD) format are established between PAS and the customer during the contracting process. The report specifications include the test report format, protocol for the reporting limit (RL), conventions for the reporting of results less than the limit of quantitation (LOQ), and specification for the use of project or program specific data qualifiers. Information about review of analytical service requests is provided in Section 4.4.

5.10.2 Test Reports: Required Items

Regardless of deliverable or report requested, every test report issued by the location includes each of the following items:

a) A Title

b) The name and address of the location issuing the test report and for each location where testing was performed if different than address of the location issuing the report. When testing is done at multiple PAS locations, the report must clearly identify which PAS location performed each test method;

c) Unique identification of the test report and on each page an identification number to link each page to the test report, and clear identification of the end of the report.

d) The name and address of the customer

e) Identification of test methods used

f) Cross reference between client sample identification number (Sample ID) and the identification number for the sample (Lab ID) to provide unambiguous identification of samples.

g) The date of receipt of samples, condition of samples on receipt, and identification of any instance where receipt of the samples did not meet sample acceptance criteria.

h) Date and times of sample collection, receipt, preparation, and analysis.

i) Test results and units of measurement, and qualification of results associated with QC criteria exceptions, and identification of reported results outside of the calibration range.

j) All chains of custody (COC) including records of internal transfer between locations within PAS,

k) Name, title, signature of the person(s) authorizing release of the test report and date of release.

l) A statement that the results in the test report relate only to the items tested.

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m) Statement that the test report may not be reproduced except in full without written approval from PAS.

5.10.3 Test Reports: Supplemental Items

5.10.3.1 Supplemental Requirements

The following items are included in the test report when required or relevant:

a) Shipping manifests / bill of ladings as applicable when common couriers are utilized for shipment of samples,

b) Explanation of departure from test method SOPs including, what the departure was and why it was necessary.

c) Statistical methods used. (Required for Whole Effluent Toxicity)

d) For solid samples, specification that results are reported on a dry weight or wet weight basis.

e) Signed Affidavit, when required by client or regulatory agency.

f) A statement of compliance / non-compliance with requirements or specifications (client, program, or standard) that includes identification of test results that did not meet acceptance criteria.

g) When requested by the client, statement of estimated measurement uncertainty. In general, for environmental testing, estimated uncertainty of measurement is extrapolated from LCS control limits. Control limits incorporate the expected variation of the data derived from the laboratory's procedure. When the control limits are specified by the test method or regulatory program, the control limits represent the expected variation of the test method and/or matrices for which the test method was designed.

h) Opinions and Interpretations

i) If a claim of accreditation/certification is included in the test report, identification of any test methods or analytes for which accreditation/certification is not held by the location if the accrediting body offers accreditation/certification for the test method/analyte. The fields of accreditation/certification vary between agencies, and it cannot be presumed that because accreditation/certification is not held that it is offered or required.

j) Certification Information, including certificate number and issuing body.

For PAS locations accredited to ISO/IEC 17025:2017:

- Data included in the test report provided by a customer should be clearly identified. The test report should also include a statement that the test results apply only to the samples as received.

5.10.3.2 Test Reports: Sampling Information

The following items are included in the test report when samples are collected by PAS or when this information is necessary for the interpretation of test results:

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a) Date of Sampling.

b) Unambiguous identification of material samples.

c) Location of sampling including diagrams, sketches, or photographs.

d) Reference to the sampling plan and procedures used.

e) Details of environmental conditions at time of sample that may impact test results.

f) Any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

6.0 REVISION HISTORY

This Version (Version 2):

Section	Description of Change
Header / All	Added registered trademark after Pace as required by branding guidelines
Header	Updated the years associated with the copyright.
Signature Page	Removed Cover Page applied by MasterControl eDMS
Approval Signatory	Changed name of this page to "Management Personnel" and updated Job Titles.
All	Changed references to "laboratory" with PAS or location, where appropriate.
All	Replaced stand-alone acronym "ENV" with "PAS" except where "ENV" is embedded in document control numbers.
All	Corrected spelling, typographical, and format errors.
Various	Added language to clarify the examples in the manual are provided for chemistry, these examples may not apply in the same way to other disciplines such as radiochemistry, microbiology, asbestos, or whole effluent toxicity (WET).
1.0	Corrected Parent Company Information.
1.2	Added definitions for "location," "laboratory" and "service center" for QMS and compliance purposes.
1.2.1	Updated job titles to match current structure.
1.2.2	Revised language for clarity.
1.2.3	Removed specificity to allow for more options
4.1.4	Updated to describe current scope of organization
4.1.4.1	Updated to describe current organization structure
4.1.5.1.1	Updated to match new organization structure and job titles
4.1.5.2	Updated to match new organization structure and job titles
4.1.5.2.1	Updated to clarify qualifications and meaning of "absent"
4.1.5.3	Updated to clarify impartiality
4.1.5.4	Reorganized section for clarity
4.2.1.1	Added statement that the organization structure is designed to safeguard impartiality
4.2.2.1	Added requirement to post compliance alertline posters in work area.
4.2.1.3	Added requirement for policies and procedures to be available in work area (previously implied but not explicitly stated)
4.2.5.1	Clarified hierarchy and application of project documents
4.5	Updated requirements for internal and external subcontracting

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4.8	Updated complaint handling requirements to clarify that only valid complaints are acted on with corrective action.
4.9.1.3	Added roles responsible for authorizing return to work after stop work order.
4.11	Main and subsections updated for clarity
4.14	Main and subsections updated for clarity
5.2.2 Subsections	Content reorganized and language related to documentation of training and authorization of personnel revised to clarify expectations. Requirements of DOCs modified to clarify procedure described in manual pertains to chemistry methodology; other approaches to DOC acceptable for other disciplines such as microbiology, radiochemistry, asbestos, whole effluent toxicity.
5.4.5.3.3	Added reference documents for which the local SOP for LOD must comply with.
5.5	Added language to clarify existing requirements.
5.6.4	Clarified requirements for reference standards for working weights and thermometers and defined meaning of terms "annual" and "quarterly." Included examples of acceptable reference standards for adequacy checks.
5.8.1	Added recommendation for Pace® personnel to add "Pace®" next to their signature on the CoC when receiving samples since the CoC form has signature/company, implying the company affiliation must be added.
5.10.3.1	Included ISO/IEC 17025:2017 to add disclaimer to test reports (applies to laboratories accredited to ISO/IEC 17025:2017 only).

This document supersedes the following documents:

Document Number	Title	Version
ENV-TMP-CORQ-0007	Quality Manual Template	01
ENV-MAN-CORQ-0001	Quality Manual	00

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7.0 APPENDICES

7.1 Appendix A: Certification / Accreditation Listing

Disclaimer: The certifications / accreditation lists provided in this Appendix are those that were held by the PAS location on the effective date of this manual. This information is subject to change without notice and must not be considered valid proof of certification or accreditation status. This manual is not updated with each change made. Current certificates are accessible via the eDMS Portal for PAS employees. External parties should contact the location for the most current information.

7.1.1 PAS-Asheville

Authority	ID
Commonwealth of Virginia	460222
NC DPHH	37712
NC DEQ	40
SC DHEC	99030001

7.1.2 PAS-Eden

Authority	ID
Commonwealth of Virginia	460025
NC DPHH	37738
NC DEQ	633

7.1.3 PAS-Greenwood

Authority	ID
SC DHEC	24562001

7.1.4 PAS-Raleigh

Authority	ID
NC DPHH	37731
NC DEQ	67

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7.2 Appendix B: Capability Listing

The capabilities listed in this Appendix were held by the location referenced on the effective date of this manual. This information is subject to change without notice. External parties should contact the location for the most current information.

Table Legend:

- Air = Air
- DW = Drinking Water
- NPW = Non-Potable Water
- SCM = Solid and Chemical Materials
- Waste = Non-Aqueous Phase Liquid (NAPL), Oil
- Tissue = Biota and Tissue

7.2.1 PAS-Asheville

Analyte	Method	Matrices		
		DW	NPW	SCM
Alkalinity As CaCO3	SM 2320 B-2011	x	x	
Ammonia As N	EPA 350.1 REV 2		x	
Biochemical Oxygen Demand (BOD)	SM 5210 B-2011		x	
Bromide	EPA 300.0 REV 2.1		x	
Bromide	EPA 9056 A		x	
Carbonaceous Bod (CBOD)	SM 5210 B-2011		x	
Chemical Oxygen Demand (COD)	SM 5220 D-2011		x	
Chloride	EPA 300.0 REV 2.1		x	
Chloride	EPA 9056 A		x	
Chloride	SM 4500-CL⁻ E-2011		x	
Chlorine, Total Residual	SM 4500 CL G-2011 (Aqueous)		x[1]	
Chromium Vi	EPA 218.6 REV 3.3		x	
Chromium Vi	EPA 218.7, Rev. 1.0, 2011 [NCAC .02L Variance]		x[1]	
Chromium Vi	EPA 7196 A		x	
Chromium Vi	SM 3500-CR B-2011		x	
Chromium Vi	SW-846 7196 A (Non-Aqueous)		x[1]	
Color	SM 2120 B-2011	x		
Conductivity	EPA 120.1		x	
Cyanide	LACHAT QUIKCHEM 10-204-00-1-X		x	
Cyanide	SM 4500-CN⁻ E-2011		x	
Cyanide, Total	EPA 9012 B		x	
Escherichia Coli	SM 9223 COLILERT MPN	x		

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Escherichia Coli	SM 9223 COLILERT-18 P/A	x		
Escherichia Coli	SM 9223 COLISURE®	x		
Escherichia Coli	SM 9223 COLISURE®	x		
Flashpoint	EPA 1010 B		x	x
Fluoride	EPA 300.0 REV 2.1		x	
Fluoride	EPA 9056 A		x	
Free Liquid	EPA 9095 B		x	x
Kjeldahl Nitrogen - Total (TKN)	EPA 351.2 REV 2 (AS LACHAT 10-107-06-2-D)		x	
Lithium	EPA 6010 D		x	
Mercury	EPA 1631 E		x	
Mercury	EPA 245.1 REV 3		x	
Mercury	EPA 7470 A		x	
Mercury	EPA 7471 B			x
Metals	EPA 200.7 REV 4.4	x[1]	x	
Metals	EPA 200.8 REV 5.4	x	x	
Metals	EPA 6010 D		x	x
Metals	EPA 6020 B		x	
Metals Digestion of Solids	EPA 3050 B			x
Metals Digestion of Water	EPA 3010 A		x	x
Metals Digestion of Water (Dissolved)	EPA 3005 A		x	
Nitrate As N	EPA 300.0 REV 2.1		x	
Nitrate As N	EPA 353.2 REV 2 (AS LACHAT 10-107-04-1 A + C)	x	x	
Nitrate As N	EPA 9056 A		x	
Nitrate As N	HACH 10206	x		
Nitrate/Nitrite	EPA 300.0 REV 2.1		x	
Nitrate/Nitrite	EPA 353.2 REV 2 (AS LACHAT 10-107-04-1-A)	x	x	
Nitrate/Nitrite	EPA 9056 A - EXTENDED		x	
Nitrite As N	EPA 300.0 REV 2.1		x	
Nitrite As N	EPA 353.2 REV 2 (AS LACHAT 10-107-04-1-A)	x	x	
Nitrite As N	EPA 9056 A		x	
Nitrite As N	SM 4500-$NO2^-$ B-2011	x		
Organic Nitrogen	EPA 351.2 MINUS EPA 350.1		x	
Orthophosphate As P	SM 4500-P E-2011		x	
Ph	EPA 9040 C		x	
Ph	EPA 9045 D			x
Ph	SM 4500 H+B-2011 (Aqueous)	x	x[1]	
Phosphorus, Total	EPA 365.1 REV 2 (AS LACHAT 10-115-01-1-E)		x	

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Prep: Cyanide Distillation	EPA 9010 C		x	
Residue-Filterable (TDS)	SM 2540 C-2011	x	x	
Residue-Nonfilterable (TSS)	SM 2540 D-2011		x	
Residue-Settleable	SM 2540 F-2011		x	
Residue-Total (TS)	SM 2540 B-2011		x	
Residue-Volatile	EPA 160.4		x	
Sulfate	EPA 300.0 REV 2.1		x	
Sulfate	EPA 9056 A		x	
Sulfide	SM 4500-$S2^-$ D-2011		x	
Total Coliforms	SM 9223 COLILERT MPN	x		
Total Coliforms	SM 9223 COLILERT-18 P/A	x		
Total Coliforms	SM 9223 COLISURE®	x		
Total Coliforms	SM 9223 COLISURE®	x		
Total Hardness As Caco3	SM 2340 B-2011		x	
Total Organic Carbon (TOC)	EPA 9060 A		x	
Total Organic Carbon (TOC)	SM 5310 B-2011		x	
Total Phenolics	EPA 420.4 REV 1 (AS LACHAT 10-210-00-1-X)		x	
Turbidity	EPA 180.1 REV 2		x	
Turbidity	SM 2130 B-2011	x	x	
UV 254	SM 5910 B-2011	x		

[1] = Laboratory does not hold TNI Accreditation for this test method.

7.2.2 PAS-Eden

Analyte	**Method**	Matrices		
		DW	NPW	SCM
Cyanide, Free	ASTM D4282-02		x	
Escherichia Coli	COLILERT MPN		x	
Fecal Coliforms	COLILERT-18 MPN		x	
Enterococci	ENTEROLERT®		x	
Ammonia As N	EPA 350.1 REV 2		x	
Nitrate As N	EPA 353.2 REV 2 (AS LACHAT 10-107-04-1 A + C)	x	x	
Nitrate/Nitrite	EPA 353.2 REV 2 (AS LACHAT 10-107-04-1-A)	x	x	
Nitrite As N	EPA 353.2 REV 2 (AS LACHAT 10-107-04-1-A)	x	x	
pH	EPA 9040 C		x	
Nitrate As N	HACH 10206	x	x	
Color	SM 2120 B-2011	x	x	
Turbidity	SM 2130 B-2011	x		
Alkalinity As CaCO3	SM 2320 B-2011	x		

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Residue-Filterable (TDS)	SM 2540 C-2011	x	x	
Residue-Nonfilterable (TSS)	SM 2540 D-2011		x	
Residue-Settleable	SM 2540 F-2011		x	
Chloride	SM 4500-CL^- E-2011		x	
Ph	SM 4500-H+ B-2011	x		
Nitrite As N	SM 4500-$NO2^-$ B-2011	x	x	
Orthophosphate As P	SM 4500-P E-2011		x	
Biochemical Oxygen Demand (BOD)	SM 5210 B-2011		x	
Carbonaceous Bod (CBOD)	SM 5210 B-2011		x	
UV 254	SM 5910 B-2011	x		
Escherichia Coli	SM 9223 COLILERT MPN	x		
Total Coliforms	SM 9223 COLILERT MPN	x		
Escherichia Coli	SM 9223 COLILERT-18 P/A	x		
Total Coliforms	SM 9223 COLILERT-18 P/A	x		
Escherichia Coli	SM 9223 COLISURE®	x		
Total Coliforms	SM 9223 COLISURE®	x		

1 = Laboratory does not hold TNI Accreditation for this test method.

7.2.3 PAS-Raleigh

Analyte	**Method**	Matrices		
		DW	NPW	SCM
Fecal Coliforms	COLILERT-18 MPN		x	
Fecal Coliforms	SM 9222D (MF)		x	
Heterotrophic Bacteria	SIMPLATE	x		
Escherichia Coli	SM 9223B COLILERT-18 P/A	x		
Total Coliforms	SM 9223B COLILERT-18 P/A	x		
Escherichia Coli	SM 9223 COLISURE®	x		
Total Coliforms	SM 9223 COLISURE®	x		

7.2.4 PAS-Greenwood

Analyte	**Method**	Matrices		
		DW	NPW	SCM
Residual Chlorine	SM 4500-CL G-3022	x	x	
Dissolved Oxygen	SM 4500-0 G-2011		x	
Turbidity	SM 2130 B-2011		x	
pH	SM 4500-H B-2011		x	
Specific Conductance	SM 2510B-2011		x	

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Temperature	SM 2550B-2010	x	x	

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7.3 Appendix C: Glossary

This glossary provides common terms and definitions used by PAS. **It is not intended to be a complete list of all terms and definitions used.** The definitions have been compiled mostly from the TNI Standard and DoD QSM. Although this information has been reproduced with care, errors cannot be entirely excluded. Definitions for the same term also vary between sources. When the meaning of a term used in a PAS document is different from this glossary or when the glossary does not include the term, the term and definition is included or defined in context in the laboratory document.

Term	Definition
3P Program	The continuous improvement program used by PAS that focuses on Process, Productivity, and Performance.
Absence	Inability to perform assigned duties due to lack of physical presence and connectivity.
Acceptance Criteria	TNI- Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	TNI- The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. DoD- Refers to accreditation in accordance with the DoD ELAP.
Accreditation Body (AB)	TNI- The organization having responsibility and accountability for environmental laboratory accreditation, and which grants accreditation under this program. DoD- Entities recognized in accordance with the DoD-ELAP that are required to operate in accordance with ISO/IEC 17011, *Conformity assessment: General requirements for accreditation bodies accrediting conformity assessment bodies*. The AB must be a signatory, in good standing, to the International Laboratory Accreditation Cooperation (ILAC) mutual recognition arrangement (MRA) that verifies, by evaluation and peer assessment, that its signatory members are in full compliance with ISO/IEC 17011 and that its accredited laboratories comply with ISO/IEC 17025.
Accuracy	TNI- The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Activity, Absolute	TNI- Rate of nuclear decay occurring in a body of material, equal to the number of nuclear disintegrations per unit time. NOTE: Activity (absolute) may be expressed in becquerels (Bq), curies (Ci), or disintegrations per minute (dpm), and multiples or submultiples of these units.
Activity, Areic	TNI- Quotient of the activity of a body of material and its associated area.
Activity, Massic	TNI- Quotient of the activity of a body of material and its mass; also called specific activity.
Activity, Volumic	TNI- Quotient of the activity of a body of material and its volume; also called activity concentration. NOTE: In this module [TNI Volume 1, Module 6], unless otherwise stated, references to activity shall include absolute activity, areic activity, massic activity, and volumic activity.
Activity Reference Date	TNI- The date (and time, as appropriate to the half-life of the radionuclide) to which a reported activity result is calculated. NOTE: The sample collection date is most frequently used as the Activity Reference Date for environmental measurements, but different programs may specify other points in time for correction of results for decay and ingrowth.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
American Society for Testing and Materials (ASTM)	An international standards organization that develops and publishes voluntary consensus standards for a wide range of materials, products, systems, and services.
Analysis	DoD- A combination of sample preparation and instrument determination.
Analysis Code (Acode)	All the set parameters of a test, such as Analytes, Method, Detection Limits and Price.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyst	TNI- The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

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Analyte	TNI- A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed. DoD- The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family and are analyzed together.
Analytical Method	DoD- A formal process that identifies and quantifies the chemical components of interest (target analytes) in a sample.
Analytical Uncertainty	TNI- A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Aliquot	DoD- A discrete, measured, representative portion of a sample taken for analysis.
Annual (or Annually)	Defined by PAS as every 12 months ± 30 days.
Assessment	TNI - The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation). DoD- An all-inclusive term used to denote any of the following: audit, performance evaluation, peer review, inspection, or surveillance conducted on-site.
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Atomization	A process in which a sample is converted to free atoms.
Audit	TNI- A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.
Batch	TNI- Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same quality systems matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours or the timeframe specified by the regulatory program. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples.
Batch, Radiation Measurements (RMB)	TNI- An RMB is composed of 1 to 20 environmental samples that are counted directly without preliminary physical or chemical processing that affects the outcome of the test (e.g., non-destructive gamma spectrometry, alpha/beta counting of air filters, or swipes on gas proportional detectors). The samples in an RMB share similar physical and chemical parameter, and analytical configurations (e.g., analytes, geometry, calibration, and background corrections). The maximum time between the start of processing of the first and last in an RMB is 14 calendar days.
Bias	TNI- The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	TNI and DoD- A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage, or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results (See Method Blank). DoD- Blank samples are negative control samples, which typically include field blank samples (e.g., trip blank, equipment (rinsate) blank, and temperature blank) and laboratory blank samples (e.g., method blank, reagent blank, instrument blank, calibration blank, and storage blank).
Blind Sample	A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
BNA (Base Neutral Acid compounds)	A list of semi-volatile compounds typically analyzed by mass spectrometry methods. Named for the way they can be extracted out of environmental samples in an acidic, basic, or neutral environment.
BOD (Biochemical Oxygen Demand)	Chemical procedure for determining how fast biological organisms use up oxygen in a body of water.

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Calibration	TNI- A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. 1) In calibration of support equipment, the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI); 2) In calibration according to test methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.
Calibration Curve	TNI- The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	A defined technical procedure for performing a calibration.
Calibration Range	DoD- The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve. For metals analysis with a single-point calibration, the low-level calibration check standard and the high standard establish the linear calibration range, which lies within the linear dynamic range.
Calibration Standard	TNI- A substance or reference material used for calibration.
Certified Reference Material (CRM)	TNI- Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.
Chain of Custody	An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Chain of Custody Form (COC)	TNI- Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and type of containers; the mode of collection, the collector, time of collection; preservation; and requested analyses.
Chemical Oxygen Demand (COD)	A test commonly used to indirectly measure the amount of organic compounds in water.
Client (referred to by ISO as Customer)	Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another. Comparable data are produced through the use of standardized procedures and techniques.
Completeness	The percent of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. The equation for completeness is: % Completeness = (Valid Data Points/Expected Data Points)*100
Confirmation	TNI- Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include but are not limited to second-column confirmation; alternate wavelength; derivatization; mass spectral interpretation; alternative detectors; or additional cleanup procedures. DoD- Includes verification of the identity and quantity of the analyte being measured by another means (e.g., by another determinative method, technology, or column). Additional cleanup procedures alone are not considered confirmation techniques.
Conformance	An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also, the state of meeting the requirements.
Congener	A member of a class of related chemical compounds (e.g., PCBs, PCDDs).
Consensus Standard	DoD- A standard established by a group representing a cross-section of a particular industry or trade, or a part thereof.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods that are used to evaluate an instrument calibration from the standpoint of the integrity of the system. High variability would suggest leaks or active sites on the instrument column.
Continuing Calibration Verification	DoD- The verification of the initial calibration. Required prior to sample analysis and at periodic intervals. Continuing calibration verification applies to both external and internal standard calibration techniques, as well as to linear and non-linear calibration models.
Continuing Calibration Verification (CCV) Standard	Also referred to as a Calibration Verification Standard (CVS) in some methods, it is a standard used to verify the initial calibration of compounds in an analytical method. CCVs are analyzed at a frequency determined by the analytical method.

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Continuous Emission Monitor (CEM)	A flue gas analyzer designed for fixed use in checking for environmental pollutants.
Continuous Improvement Plan (CIP)	The delineation of tasks for a given laboratory department or committee to achieve the goals of that department.
Contract Laboratory Program (CLP)	A national network of EPA personnel, commercial labs, and support contractors whose fundamental mission is to provide data of known and documented quality.
Contract Required Detection Limit (CRDL)	Detection limit that is required for EPA Contract Laboratory Program (CLP) contracts.
Contract Required Quantitation Limit (CRQL)	Quantitation limit (reporting limit) that is required for EPA Contract Laboratory Program (CLP) contracts.
Control Chart	A graphic representation of a series of test results, together with limits within which results are expected when the system is in a state of statistical control (see definition for Control Limit)
Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control. Control limit exceedances may require corrective action or require investigation and flagging of non-conforming data.
Correction	DoD- Action taken to eliminate a detected non-conformity.
Corrective Action	DoD- The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence. A root cause analysis may not be necessary in all cases.
Corrective and Preventative Action (CAPA)	The primary management tools for bringing improvements to the quality system, to the management of the quality system's collective processes, and to the products or services delivered which are an output of established systems and processes.
Critical Value	TNI- Value to which a measurement result is compared to make a detection decision (also known as critical level or decision level). NOTE: The Critical Value is designed to give a specified low probability α of false detection in an analyte-free sample, which implies that a result that exceeds the Critical Value, gives high confidence (1 – α) that the radionuclide is actually present in the material analyzed. For radiometric methods, α is often set at 0.05.
Customer	DoD- Any individual or organization for which products or services are furnished or work performed in response to defined requirements and expectations.
Data Integrity	TNI- The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.
Data Quality Objective (DQO)	Systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	TNI- The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form.
Definitive Data	DoD- Analytical data of known quantity and quality. The levels of data quality on precision and bias meet the requirements for the decision to be made. Data that is suitable for final decision-making.
Demonstration of Capability (DOC)	TNI- A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. DoD- A procedure to establish the ability of the analyst to generate analytical results by a specific method that meet measurement quality objectives (e.g., for precision and bias).
Department of Defense (DoD)	An executive branch department of the federal government of the United States charged with coordinating and supervising all agencies and functions of the government concerned directly with national security.
Detection Limit (DL)	DoD- The smallest analyte concentration that can be demonstrated to be different than zero or a blank concentration with 99% confidence. At the DL, the false positive rate (Type 1 error) is 1%. A DL may be used as the lowest concentration for reliably reporting a detection of a specific analyte in a specific matrix with a specific method with 99% confidence.
Detection Limit (DL) for Safe Drinking Water Act (SDWA) Compliance	TNI- Laboratories that analyze drinking-water samples for SDWA compliance monitoring must use methods that provide sufficient detection capability to meet the detection limit requirements established in 40 CFR 141. The SDWA DL for radioactivity is defined in 40 CFR Part 141.25.c as the radionuclide concentration, which can be counted with a precision of plus or minus 100% at the 95% confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).
Deuterated Monitoring Compounds (DMCs)	DoD- SIM specific surrogates as specified for GC/MS SIM analysis.

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Diesel Range Organics (DRO)	A range of compounds that denote all the characteristic compounds that make up diesel fuel (range can be state or program specific).
Digestion	DoD- A process in which a sample is treated (usually in conjunction with heat and acid) to convert the target analytes in the sample to a more easily measured form.
Document Control	The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.
Documents	DoD- Written components of the laboratory management system (e.g., policies, procedures, and instructions).
Dry Weight	The weight after drying in an oven at a specified temperature.
Duplicate (also known as Replicate or Laboratory Duplicate)	The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results of duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation, or storage internal to the laboratory.
Electron Capture Detector (ECD)	Device used in GC methods to detect compounds that absorb electrons (e.g., PCB compounds).
Electronic Data Deliverable (EDD)	A summary of environmental data (usually in spreadsheet form) which clients request for ease of data review and comparison to historical results.
Eluent	A solvent used to carry the components of a mixture through a stationary phase.
Elute	To extract, specifically, to remove (absorbed material) from an absorbent by means of a solvent.
Elution	A process in which solutes are washed through a stationary phase by movement of a mobile phase.
Environmental Data	DoD- Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology.
Environmental Monitoring	The process of measuring or collecting environmental data.
Environmental Protection Agency (EPA)	An agency of the federal government of the United States which was created for the purpose of protecting human health and the environment by writing and enforcing regulations based on laws passed by Congress.
Environmental Sample	A representative sample of any material (aqueous, non-aqueous, or multimedia) collected from any source for which determination of composition or contamination is requested or required. Environmental samples can generally be classified as follows: • Non-Potable Water (Includes surface water, ground water, effluents, water treatment chemicals, and TCLP leachates or other extracts) • Drinking Water - Delivered (treated or untreated) water designated as potable water • Water/Wastewater - Raw source waters for public drinking water supplies, ground waters, municipal influents/effluents, and industrial influents/effluents • Sludge - Municipal sludges and industrial sludges. • Soil - Predominately inorganic matter ranging in classification from sands to clays. • Waste - Aqueous and non-aqueous liquid wastes, chemical solids, and industrial liquid and solid wastes
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Extracted Internal Standard Analyte	Isotopically labeled analogs of analytes of interest added to all standards, blanks and samples analyzed. Added to samples and batch QC samples prior to the first step of sample extraction and to standards and instrument blanks prior to analysis. Used for isotope dilution methods.
Facility	A distinct location within the company that has unique certifications, personnel, and waste disposal identifications.
False Negative	DoD- A result that fails to identify (detect) an analyte or reporting an analyte to be present at or below a level of interest when the analyte is actually above the level of interest.
False Positive	DoD- A result that erroneously identifies (detects) an analyte or reporting an analyte to be present above a level of interest when the analyte is actually present at or below the level of interest.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Determination of physical, biological, or radiological properties, or chemical constituents that are measured on-site, close in time and sPAS to the matrices being sampled/measured, following accepted test methods. This testing is performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.

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Field of Accreditation	TNI- Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.
Field of Proficiency Testing (FoPT)	TNI- Matrix, technology/method, analyte combinations for which the composition, spike concentration ranges and acceptance criteria have been established by the PTPEC.
Finding	TNI- An assessment conclusion referenced to a laboratory accreditation standard and supported by objective evidence that identifies a deviation from a laboratory accreditation standard requirement. DoD- An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive, negative, or neutral and is normally accompanied by specific examples of the observed condition. The finding must be linked to a specific requirement (e.g., this standard, ISO requirements, analytical methods, contract specifications, or laboratory management systems requirements).
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.
Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify, and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	TNI- The maximum time that can elapse between two specified activities. 40 CFR Part 136- The maximum time that samples may be held prior to preparation and/or analysis as defined by the method and still be considered valid or not compromised. For sample prep purposes, hold times are calculated using the time of the start of the preparation procedure. DoD- The maximum time that may elapse from the time of sampling to the time of preparation or analysis, or from preparation to analysis, as appropriate.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Homologue	One in a series of organic compounds in which each successive member has one more chemical group in its molecule than the next preceding member. For instance, methanol, ethanol, propanol, butanol, etc., form a homologous series.
Improper Actions	DoD- Intentional or unintentional deviations from contract-specified or method-specified analytical practices that have not been authorized by the customer (e.g., DoD or DOE).
Incremental Sampling Method (ISM)	Soil preparation for large volume (1 kg or greater) samples.
In-Depth Data Monitoring	TNI- When used in the context of data integrity activities, a review and evaluation of documentation related to all aspects of the data generation process that includes items such as preparation, equipment, software, calculations, and quality controls. Such monitoring shall determine if the laboratory uses appropriate data handling, data use and data reduction activities to support the laboratory's data integrity policies and procedures.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.

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Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a calibration verification standard do not conform to the requirements of the method in use or at a frequency specified in the method.
Initial Calibration Blank (ICB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method. This blank is specifically run in conjunction with the Initial Calibration Verification (ICV) where applicable.
Initial Calibration Verification (ICV)	DoD- Verifies the initial calibration with a standard obtained or prepared from a source independent of the source of the initial calibration standards to avoid potential bias of the initial calibration.
Injection Internal Standard Analyte	Isotopically labeled analogs of analytes of interest (or similar in physiochemical properties to the target analytes but with a distinct response) to be quantitated. Added to all blanks, standards, samples, and batch QC after extraction and prior to analysis.
Instrument Blank	A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination.
Instrument Detection Limits (IDLs)	Limits determined by analyzing a series of reagent blank analyses to obtain a calculated concentration. IDLs are determined by calculating the average of the standard deviations of three runs on three non-consecutive days from the analysis of a reagent blank solution with seven consecutive measurements per day.
Interference, spectral	Occurs when particulate matter from the atomization scatters incident radiation from the source or when the absorption or emission from an interfering species either overlaps or is so close to the analyte wavelength that resolution becomes impossible.
Interference, chemical	Results from the various chemical processes that occur during atomization and later the absorption characteristics of the analyte.
Internal Standard	TNI and DoD- A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
International Organization for Standardization (ISO)	An international standard-setting body composed of representatives from various national standards organizations.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
International System of Units (SI)	The coherent system of units adopted and recommended by the General Conference on Weights and Measures.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Isomer	One of two or more compounds, radicals, or ions that contain the same number of atoms of the same element but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.
Laboratory	A body that calibrates and/or performs testing.
Laboratory Control Sample (LCS)	TNI- (also known as laboratory fortified blank (LFB), spiked blank, or QC check sample): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes and taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a reference method. It is generally used to establish intra-laboratory or analyst-specific precision and bias or to evaluate the performance of all or a portion of the measurement system.
Laboratory Duplicate	Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently.
Laboratory Information Management System (LIMS)	DoD- The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.
Learning Management System (LMS)	A web-based database used by the laboratories to track and document training activities. The system is administered by the corporate training department and each laboratory's learn centers are maintained by a local administrator.
Legal Chain-of-Custody Protocols	TNI- Procedures employed to record the possession of samples from the time of sampling through the retention time specified by the client or program. These procedures are performed at the special request of the client and include the use of a Chain-of-Custody (COC) Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory.

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Limit(s) of Detection (LOD)	TNI- The minimum result, which can be reliably discriminated from a blank with predetermined confidence level. DoD- The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. At the LOD, the false negative rate (Type II error) is 1%. A LOD may be used as the lowest concentration for reliably reporting a non-detect of a specific analyte in a specific matrix with a specific method at 99% confidence.
Limit(s) of Quantitation (LOQ)	TNI- The minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be reported with a specified degree of confidence. DoD- The smallest concentration that produces a quantitative result with known and recorded precision and bias. For DoD/DOE projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard and within the calibration range.
Linear Dynamic Range	DoD- Concentration range where the instrument provides a linear response.
Liquid chromatography/ tandem mass spectrometry (LC/MS/MS)	Instrumentation that combines the physical separation techniques of liquid chromatography with the mass analysis capabilities of mass spectrometry.
Lot	TNI- A definite amount of material produced during a single manufacturing cycle and intended to have uniform character and quality.
Management	Those individuals directly responsible and accountable for planning, implementing, and assessing work.
Management System	System to establish policy and objectives and to achieve those objectives.
Manager (however named)	The individual designated as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the supervisor and the manager may be the same individual.
Matrix	TNI- The substrate of a test sample.
Matrix Duplicate	TNI- A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS) (spiked sample or fortified sample)	TNI- A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.
Matrix Spike Duplicate (MSD) (spiked sample or fortified sample duplicate)	TNI- A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
Measurement Performance Criteria (MPC)	DoD- Criteria that may be general (such as completion of all tests) or specific (such as QC method acceptance limits) that are used by a project to judge whether a laboratory can perform a specified activity to the defined criteria.
Measurement Quality Objective (MQO)	TNI- The analytical data requirements of the data quality objectives are project- or program-specific and can be quantitative or qualitative. MQOs are measurement performance criteria or objectives of the analytical process. Examples of quantitative MQOs include statements of required analyte detectability and the uncertainty of the analytical protocol at a specified radionuclide activity, such as the action level. Examples of qualitative MQOs include statements of the required specificity of the analytical protocol, e.g., the ability to analyze for the radionuclide of interest given the presence of interferences.
Measurement System	TNI- A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s). DoD- A test method, as implemented at a particular laboratory, and which includes the equipment used to perform the sample preparation and test and the operator(s).
Measurement Uncertainty	DoD- An estimate of the error in a measurement often stated as a range of values that contain the true value within a certain confidence level. The uncertainty generally includes many components which may be evaluated from experimental standard deviations based on repeated observations or by standard deviations evaluated from assumed probability distributions based on experience or other information. For DoD/DOE, a laboratory's Analytical Uncertainty (such as use of LCS control limits) can be reported as the minimum uncertainty.
Method	TNI- A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification), systematically presented in the order in which they are to be executed.

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Method Blank	TNI- A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.
Method Detection Limit (MDL)	TNI- One way to establish a Detection Limit; defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Method of Standard Additions	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration.
Minimum Detectable Activity (MDA)	TNI- Estimate of the smallest true activity that ensures a specified high confidence, 1 – β, of detection above the Critical Value, and a low probability β of false negatives below the Critical Value. For radiometric methods, β is often set at 0.05. NOTE 1: The MDS is a measure of the detection capability of a measurement process and as such, it is an a priori concept. It may be used in the selection of methods to meet specified MQOs. Laboratories may also calculate a "sample specific" MDA, which indicates how well the measurement process is performing under varying real-world measurement conditions, when sample-specific characteristics (e.g., interferences) may affect the detection capability. However, the MDA must never be used instead of the Critical Value as a detection threshold. NOTE 2: For the purpose of this Standard, the terms MDA and minimum detectable concentration (MDC) are equivalent.
Minimum Reporting Limit (MRL)	the lowest concentration of standard used for calibration – Drinking Water Manual
MintMiner	Commercial software program used to scan large amounts of chromatographic data to monitor for errors or data integrity issues.
Mobile Laboratory	TNI- A portable enclosed structure with necessary and appropriate accommodation and environmental conditions for a laboratory, within which testing is performed by analysts. Examples include but are not limited to trailers, vans, and skid-mounted structures configured to house testing equipment and personnel.
National Environmental Laboratory Accreditation Conference (NELAC)	See definition of The NELAC Institute (TNI).
National Institute of Occupational Safety and Health (NIOSH)	National institute charged with the provision of training, consultation, and information in the area of occupational safety and health.
National Institute of Standards and Technology (NIST)	TNI- A federal agency of the US Department of Commerce's Technology Administration that is designed as the United States national metrology institute (or NMI).
National Pollutant Discharge Elimination System (NPDES)	A permit program that controls water pollution by regulating point sources that discharge pollutants into U.S. waters.
Negative Control	Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results.
Nitrogen Phosphorus Detector (NPD)	A detector used in GC analyses that utilizes thermal energy to ionize an analyte. With this detector, nitrogen and phosphorus can be selectively detected with a higher sensitivity than carbon.
Nonconformance	An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also, the state of failing to meet the requirements.
Not Detected (ND)	The result reported for a compound when the detected amount of that compound is less than the method reporting limit.
Operator Aid	DoD- A technical posting (such as poster, operating manual, or notepad) that assists workers in performing routine tasks. All operator aids must be controlled documents (i.e., a part of the laboratory management system).
Performance Based Measurement System (PBMS)	An analytical system wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner.
Physical Parameter	TNI- A measurement of a physical characteristic or property of a sample as distinguished from the concentrations of chemical and biological components.

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Photo-ionization Detector (PID)	An ion detector which uses high-energy photons, typically in the ultraviolet range, to break molecules into positively charged ions.
Polychlorinated Biphenyls (PCB)	A class of organic compounds that were used as coolants and insulating fluids for transformers and capacitors. The production of these compounds was banned in the 1970's due to their high toxicity.
Positive Control	Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects.
Post-Digestion Spike	A sample prepared for metals analyses that has analytes spike added to determine if matrix effects may be a factor in the results.
Power of Hydrogen (pH)	The measure of acidity or alkalinity of a solution.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit. The lowest reportable concentration of a compound based on parameters set up in an analytical method and the laboratory's ability to reproduce those conditions.
Precision	TNI- The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance, or range, in either absolute or relative terms.
Preservation	TNI and DoD- Any conditions under which a sample must be kept in order to maintain chemical, physical, and/or biological integrity prior to analysis.
Primary Accreditation Body (Primary AB)	TNI- The accreditation body responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.
Procedure	TNI- A specified way to carry out an activity or process. Procedures can be documented or not.
Proficiency Testing (PT)	TNI- A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.
Proficiency Testing Program (PT Program)	TNI- The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.
Proficiency Testing Provider (PT Provider)	TNI- A person or organization accredited by a TNI-approved Proficiency Testing Provider Accreditor to operate a TNI-compliant PT Program.
Proficiency Testing Provider Accreditor (PTPA)	TNI- An organization that is approved by TNI to accredit and monitor the performance of proficiency testing providers.
Proficiency Testing Reporting Limit (PTRL)	TNI- A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.
Proficiency Testing Sample (PT)	TNI- A sample, the composition of which is unknown to the laboratory, and is provided to test whether the laboratory can produce analytical results within the specified acceptance criteria.
Proficiency Testing (PT) Study	TNI- a) Scheduled PT Study: A single complete sequence of circulation and scoring of PT samples to all participants in a PT program. The study must have the same pre-defined opening and closing dates for all participants; b) Supplemental PT Study: A PT sample that may be from a lot previously released by a PT Provider that meets the requirements for supplemental PT samples given in Volume 3 of this Standard [TNI] but that does not have a pre-determined opening date and closing date.
Proficiency Testing Study Closing Date	TNI- a) Scheduled PT Study: The calendar date by which all participating laboratories must submit analytical results for a PT sample to a PT Provider; b) Supplemental PT Study: The calendar date a laboratory submits the results for a PT sample to the PT Provider.
Proficiency Testing Study Opening Date	TNI- a) Scheduled PT Study: The calendar date that a PT sample is first made available to all participants of the study by a PT Provider; b) Supplemental PT Study: The calendar date the PT Provider ships the sample to a laboratory.
Protocol	TNI- A detailed written procedure for field and/or laboratory operation (e.g., sampling, analysis) that must be strictly followed.
Qualitative Analysis	DoD- Analysis designed to identify the components of a substance or mixture.
Quality Assurance (QA)	TNI- An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Assurance Manual (QAM)	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality Assurance Project Plan (QAPP)	A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

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Quality Control (QC)	TNI- The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.
Quality Control Sample (QCS)	TNI- A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control.
Quality Manual	TNI- A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality System	TNI and DoD- A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.
Quality System Matrix	TNI and DoD- These matrix definitions shall be used for purposes of batch and quality control requirements and may be different from a field of accreditation matrix: • **Air and Emissions**: Whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device • **Aqueous**: Any aqueous sample excluded from the definition of Drinking Water or Saline/Estuarine. Includes surface water, groundwater effluents, and TCLP or other extracts. • **Biological Tissue**: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin. • **Chemical Waste**: A product or by-product of an industrial process that results in a matrix not previously defined. • **Drinking Water**: Any aqueous sample that has been designated a potable or potentially potable water source. • **Non-aqueous liquid**: Any organic liquid with <15% settleable solids • **Saline/Estuarine**: Any aqueous sample from an ocean or estuary, or other saltwater source such as the Great Salt Lake. • **Solids**: Includes soils, sediments, sludges, and other matrices with >15% settleable solids.
Quantitation Range	DoD- The range of values (concentrations) in a calibration curve between the LOQ and the highest successively analyzed initial calibration standard used to relate instrument response to analyte concentration. The quantitation range (adjusted for initial sample volume/weight, concentration/dilution, and final volume) lies within the calibration range.
Quantitative Analysis	DoD- Analysis designed to determine the amounts or proportions of the components of a substance.
Random Error	The EPA has established that there is a 5% probability that the results obtained for any one analyte will exceed the control limits established for the test due to random error. As the number of compounds measured increases in a given sample, the probability for statistical error also increases.
Raw Data	TNI- The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.
Reagent Blank (method reagent blank)	A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.
Reagent Grade	Analytical reagent (AR) grade, ACS reagent grade, and reagent grade are synonymous terms for reagents that conform to the current specifications of the Committee on Analytical Reagents of the American Chemical Society.
Records	DoD- The output of implementing and following management system documents (e.g., test data in electronic or hand-written forms, files, and logbooks).

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Reference Material	TNI- Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
Reference Method	TNI- A published method issued by an organization generally recognized as competent to do so. (When the ISO language refers to a "standard method," that term is equivalent to "reference method"). When a laboratory is required to analyze by a specified method due to a regulatory requirement, the analyte/method combination is recognized as a reference method. If there is no regulatory requirement for the analyte/method combination, the analyte/method combination is recognized as a reference method if it can be analyzed by another reference method of the same matrix and technology.
Reference Standard	TNI- Standard used for the calibration of working measurement standards in a given organization or at a given location.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.
Reporting Limit (RL)	The level at which method, permit, regulatory and customer-specific objectives are met. The reporting limit may never be lower than the Limit of Detection (i.e., statistically determined MDL). Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified. There must be a sufficient buffer between the Reporting Limit and the MDL. DoD- A customer-specified lowest concentration value that meets project requirements for quantitative data with known precision and bias for a specific analyte in a specific matrix.
Reporting Limit Verification Standard (RLVS)	A standard analyzed at the reporting limit for an analysis to verify the laboratory's ability to report to that level.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed. Sample representativeness is dependent on the sampling techniques specified in the project work plan.
Requirement	Denotes a mandatory specification; often designated by the term "shall."
Retention Time	The time between sample injection and the appearance of a solute peak at the detector.
Revocation	TNI- The total or partial withdrawal of a laboratory's accreditation by an accreditation body.
Sample	Portion of material collected for analysis, identified by a single, unique alphanumeric code. A sample may consist of portions in multiple containers, if a single sample is submitted for multiple or repetitive analysis.
Sample Condition Upon Receipt Form (SCURF)	Form used by sample receiving personnel to document the condition of sample containers upon receipt to the laboratory (used in conjunction with a COC).
Sample Delivery Group (SDG)	A unit within a single project that is used to identify a group of samples for delivery. An SDG is a group of 20 or fewer field samples within a project, received over a period of up to 14 calendar days. Data from all samples in an SDG are reported concurrently.
Sample Receipt Form (SRF)	Letter sent to the client upon login to show the tests requested and pricing.
Sample Tracking	Procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a chain-of-custody form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples.
Sampling	TNI- Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.
Selected Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit. The narrower the range, the more sensitive the detector. DoD- Using GC/MS, characteristic ions specific to target compounds are detected and used to quantify in applications where the normal full scan mass spectrometry results in excessive noise.
Selectivity	TNI- The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system.
Sensitivity	TNI- The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest.
Serial Dilution	The stepwise dilution of a substance in a solution.
Shall	Denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative approaches or methods for implementing the specification as long as the requirement is fulfilled.

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Should	Denotes a guideline or recommendation whenever noncompliance with the specification is permissible.
Signal-to-Noise Ratio (S/N)	DoD- A measure of signal strength relative to background noise. The average strength of the noise of most measurements is constant and independent of the magnitude of the signal. Thus, as the quantity being measured (producing the signal) decreases in magnitude, S/N decreases and the effect of the noise on the relative error of a measurement increases.
Source Water	TNI- When sampled for drinking water compliance, untreated water from streams, rivers, lakes, or underground aquifers, which is used to supply private and public drinking water supplies.
Spike	A known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.
Standard (Document)	TNI- The document describing the elements of a laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies.
Standard (Chemical)	Standard samples are comprised of a known amount of standard reference material in the matrix undergoing analysis. A standard reference material is a certified reference material produced by US NIST and characterized for absolute content, independent of analytical test method.
Standard Blank (or Reagent Blank)	A calibration standard consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.
Standard Method	A test method issued by an organization generally recognized as competent to do so.
Standard Operating Procedure (SOP)	TNI- A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks.
Standard Reference Material (SRM)	A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.
Statement of Qualifications (SOQ)	A document that lists information about a company, typically the qualifications of that company to compete on a bid for services.
Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Storage Blank	DoD- A sample of analyte-free media prepared by the laboratory and retained in the sample storage area of the laboratory. A storage blank is used to record contamination attributable to sample storage at the laboratory.
Supervisor	The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties and ascertaining technical employees have the required balance of education, training, and experience to perform the required analyses.
Surrogate	DoD- A substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them for quality control purposes.
Suspension	TNI- The temporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed 6 months or the period of accreditation, whichever is longer, in order to allow the laboratory time to correct deficiencies or area of non-conformance with the Standard.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Target Analytes	DoD- Analytes or chemicals of primary concern identified by the customer on a project-specific basis.
Technical Director	Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory.
Technology	TNI- A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Test	A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate.
Test Method	DoD- A definitive procedure that determines one or more characteristics of a given substance or product.
Test Methods for Evaluating Solid Waste, Physical/ Chemical (SW-846)	EPA Waste's official compendium of analytical and sampling methods that have been evaluated and approved for use in complying with RCRA regulations.

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Test Source	TNI- A radioactive source that is tested, such as a sample, calibration standard, or performance check source. A Test Source may also be free of radioactivity, such as a Test Source counted to determine the subtraction background, or a short-term background check.
The NELAC Institute (TNI)	A non-profit organization whose mission is to foster the generation of environmental data of known and documented quality through an open, inclusive, and transparent process that is responsive to the needs of the community. Previously known as NELAC (National Environmental Laboratory Accreditation Conference).
Total Petroleum Hydrocarbons (TPH)	A term used to denote a large family of several hundred chemical compounds that originate from crude oil. Compounds may include gasoline components, jet fuel, volatile organics, etc.
Toxicity Characteristic Leaching Procedure (TCLP)	A solid sample extraction method for chemical analysis employed as an analytical method to simulate leaching of compounds through a landfill.
Traceability	TNI- The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project.
Training Document	A training resource that provides detailed instructions to execute a specific method or job function.
Trip Blank	This blank sample is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory reagent water and the blank is stored, shipped, and analyzed with its associated samples.
Tuning	A check and/or adjustment of instrument performance for mass spectrometry as required by the method.
Ultraviolet Spectrophotometer (UV)	Instrument routinely used in quantitative determination of solutions of transition metal ions and highly conjugated organic compounds.
Uncertainty, Counting	TNI- The component of Measurement Uncertainty attributable to the random nature of radioactive decay and radiation counting (often estimated as the square root of observed counts (MARLAP). Older references sometimes refer to this parameter as Error, Counting Error, or Count Error (c.f., Total Uncertainty).
Uncertainty, Expanded	TNI- The product of the Standard Uncertainty and a coverage factor, k, which is chosen to produce an interval about the result that has a high probability of containing the value of the measurand (c.f., Standard Uncertainty). NOTE: Radiochemical results are generally reported in association with the Total Uncertainty. Either if these estimates of uncertainty can be reported as the Standard Uncertainty (one-sigma) or as an Expanded Uncertainty (k-sigma, where $k > 1$).
Uncertainty, Measurement	TNI- Parameter associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand.
Uncertainty, Standard	TNI- An estimate of the Measurement Uncertainty expressed as a standard deviation (c.f., Expanded Uncertainty).
Uncertainty, Total	TNI- An estimate of the Measurement Uncertainty that accounts for contributions from all significant sources of uncertainty associated with the analytical preparation and measurement of a sample. Such estimates are also commonly referred to as Combined Standard Uncertainty or Total Propagated Uncertainty, and in some older references as the Total Propagated Error, among other similar items (c.f., Counting Uncertainty).
Unethical actions	DoD- Deliberate falsification of analytical or quality control results where failed method or contractual requirements are made to appear acceptable.
United States Department of Agriculture (USDA)	A department of the federal government that provides leadership on food, agriculture, natural resources, rural development, nutrition, and related issues based on public policy, the best available science, and effective management.
United States Geological Survey (USGS)	Program of the federal government that develops new methods and tools to supply timely, relevant, and useful information about the Earth and its processes.
Unregulated Contaminant Monitoring Rule (UCMR)	EPA program to monitor unregulated contaminants in drinking water.
Validation	DoD- The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

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Verification	TNI- Confirmation by examination and objective evidence that specified requirements have been met. In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation, or specification peculiar to the management of the measuring equipment.
Voluntary Action Program (VAP)	A program of the Ohio EPA that gives individuals a way to investigate possible environmental contamination, clean it up if necessary and receive a promise from the State of Ohio that no more cleanup is needed.
Whole Effluent Toxicity (WET)	The aggregate toxic effect to aquatic organisms from all pollutants contained in a facility's wastewater (effluent).

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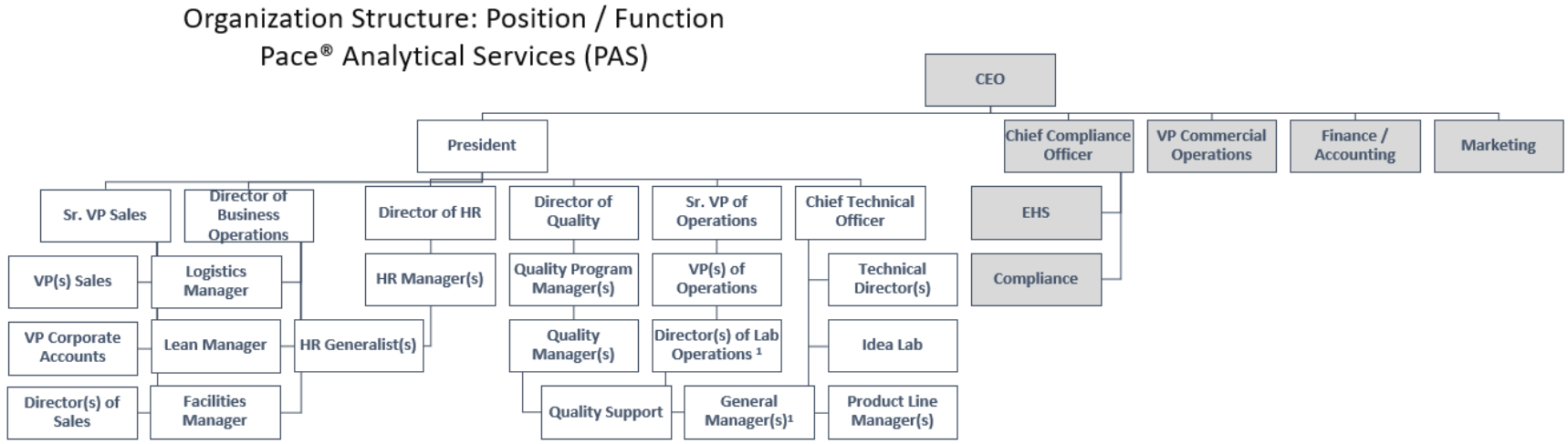
7.4 Appendix D: Organization Chart(s)

7.4.1 PAS Corporate Organization Chart(s)



White Box = PAS Positions / Functions
Grey Box = Pace®Corporate Positions / Functions
1= Positions not Assigned to all Locations – see location specific organization charts

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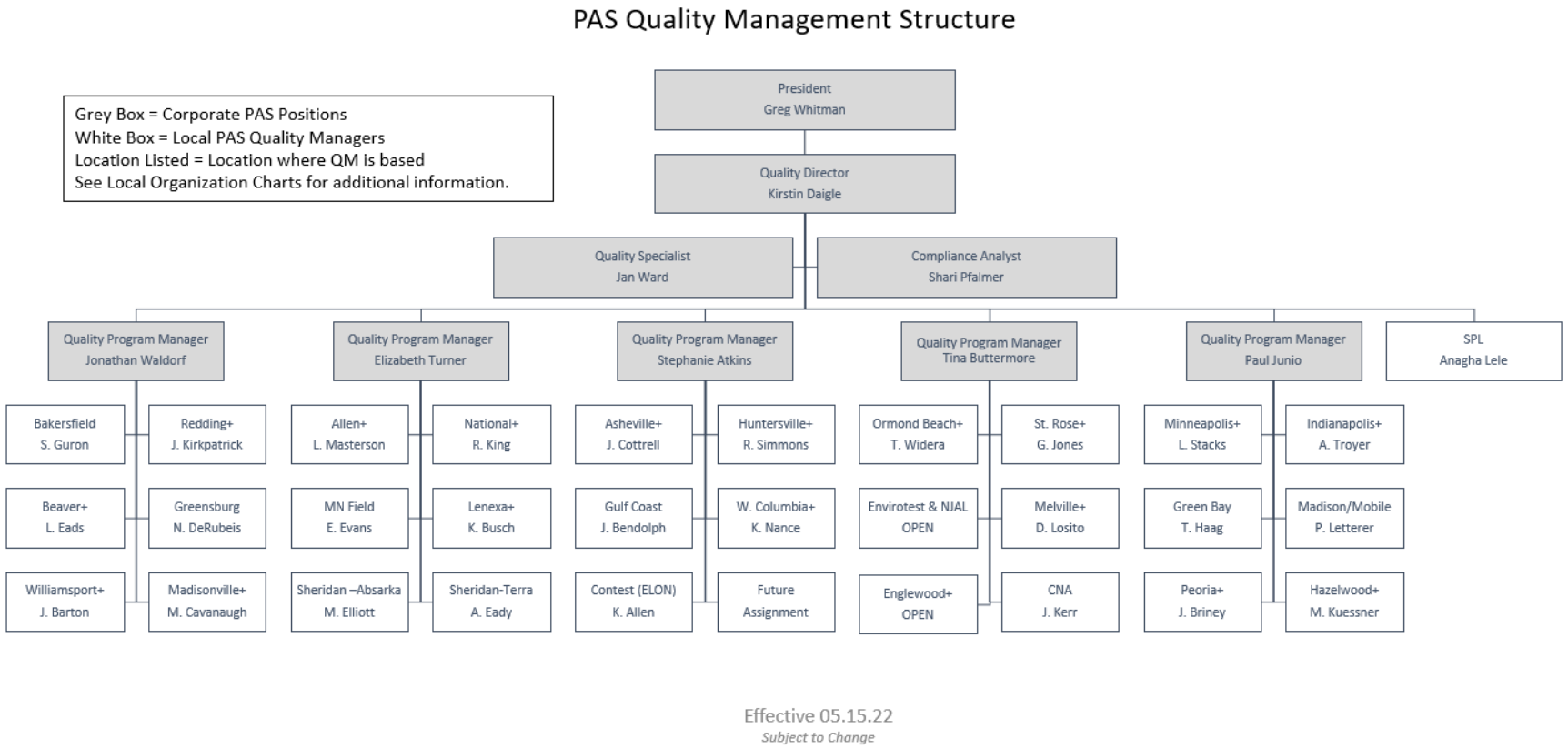
7.4.2 PAS Quality Systems Management Organization Chart



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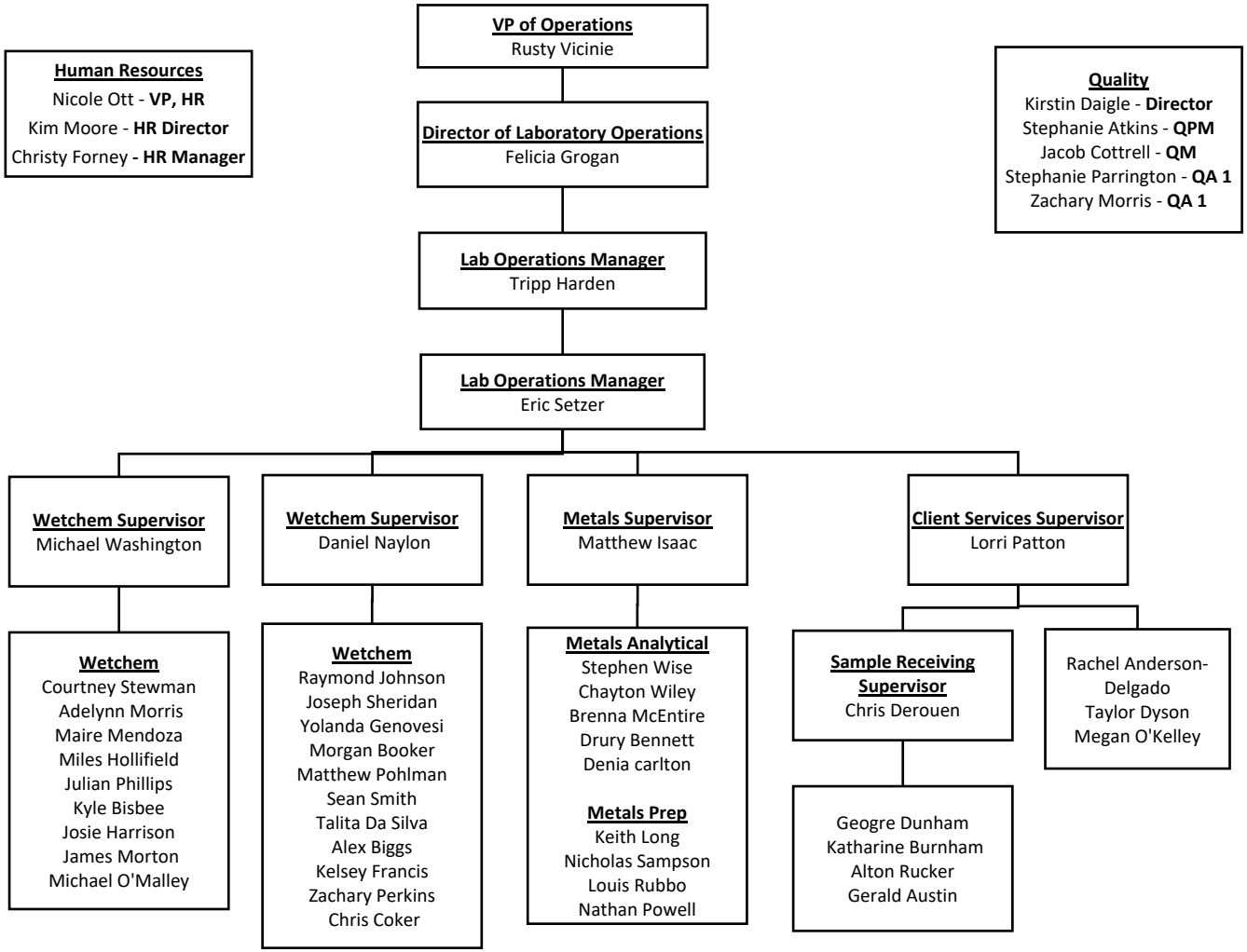
7.4.3 Asheville – Organization Chart



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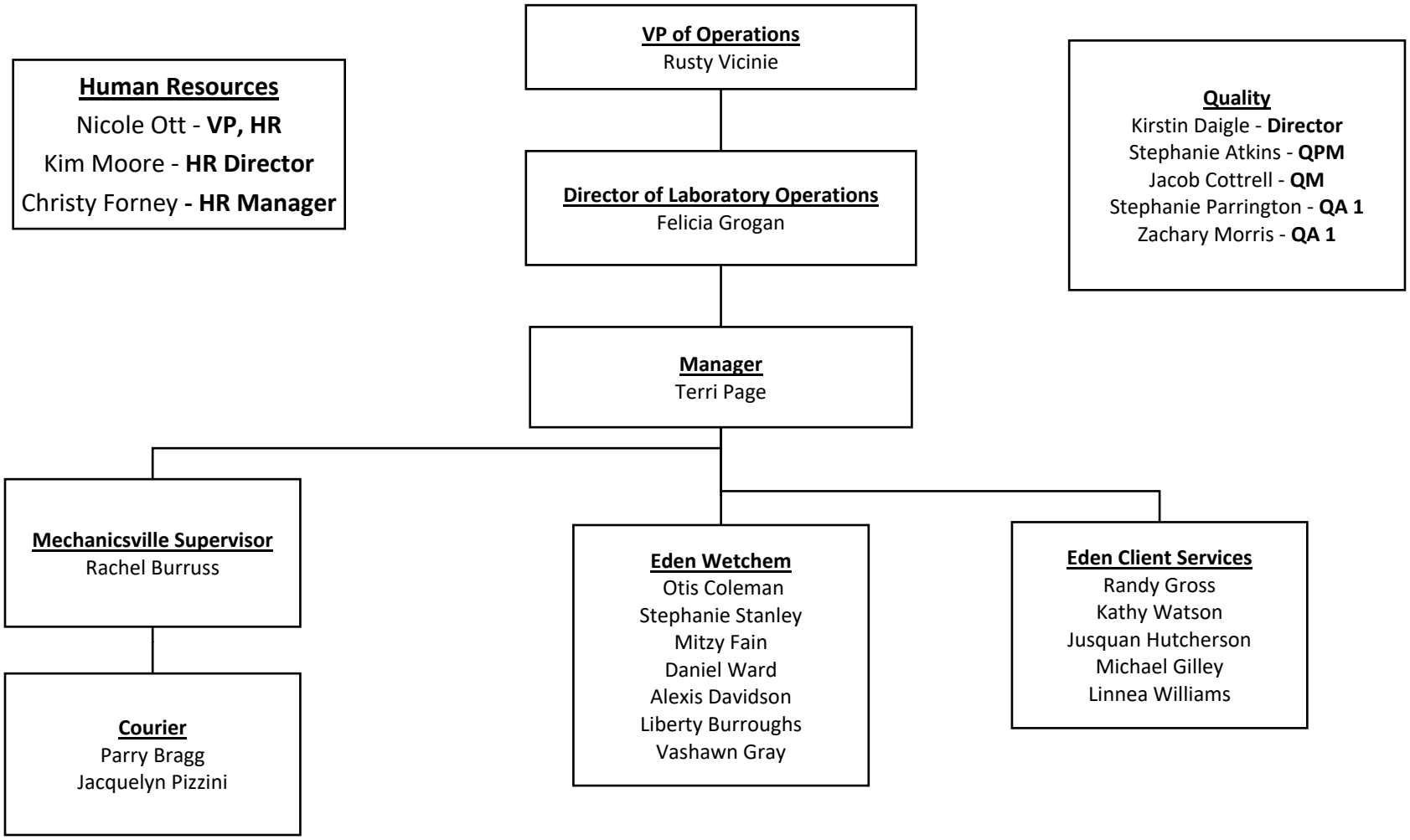
7.4.4 Eden – Organization Chart



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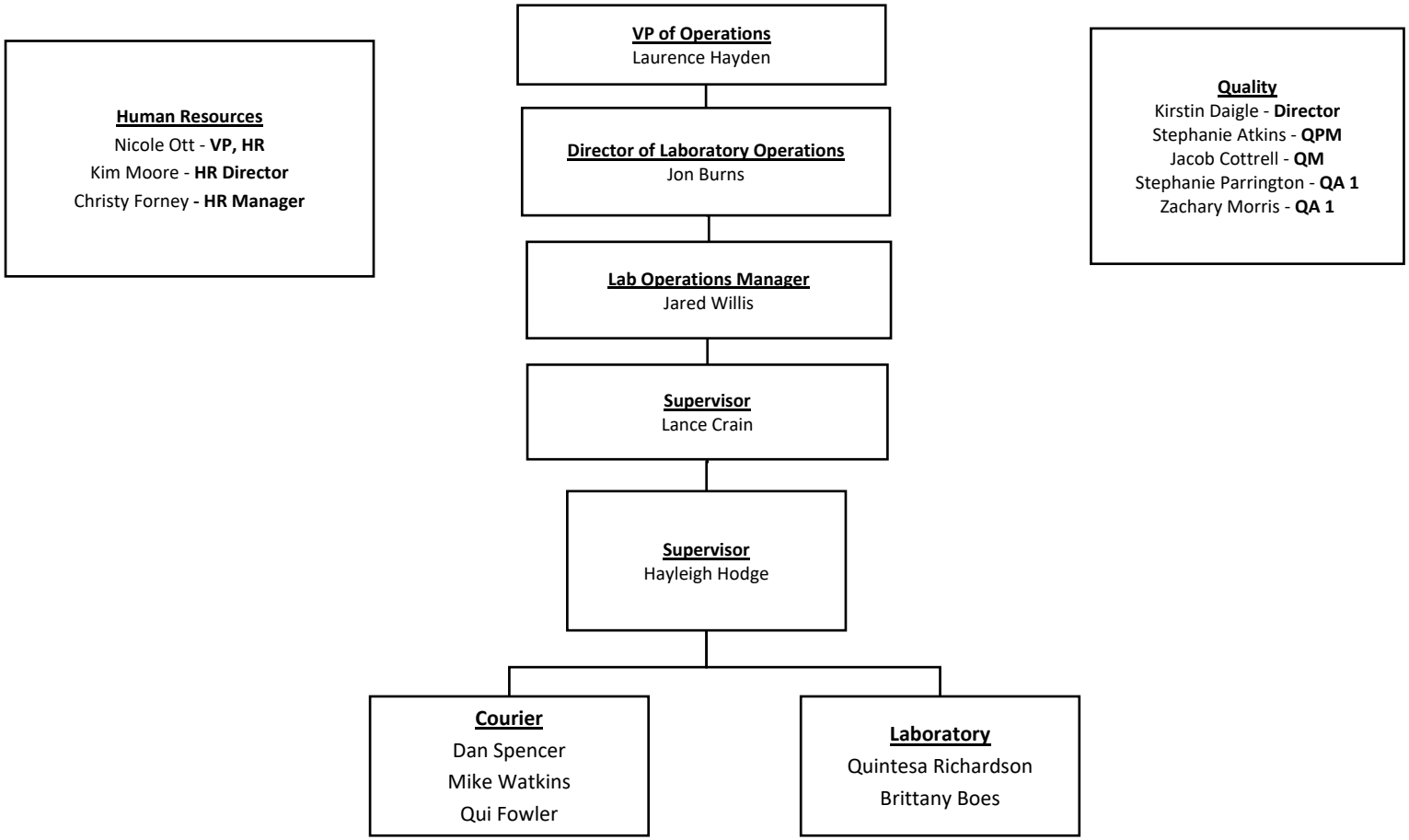
7.4.5 Raleigh - Organization Chart



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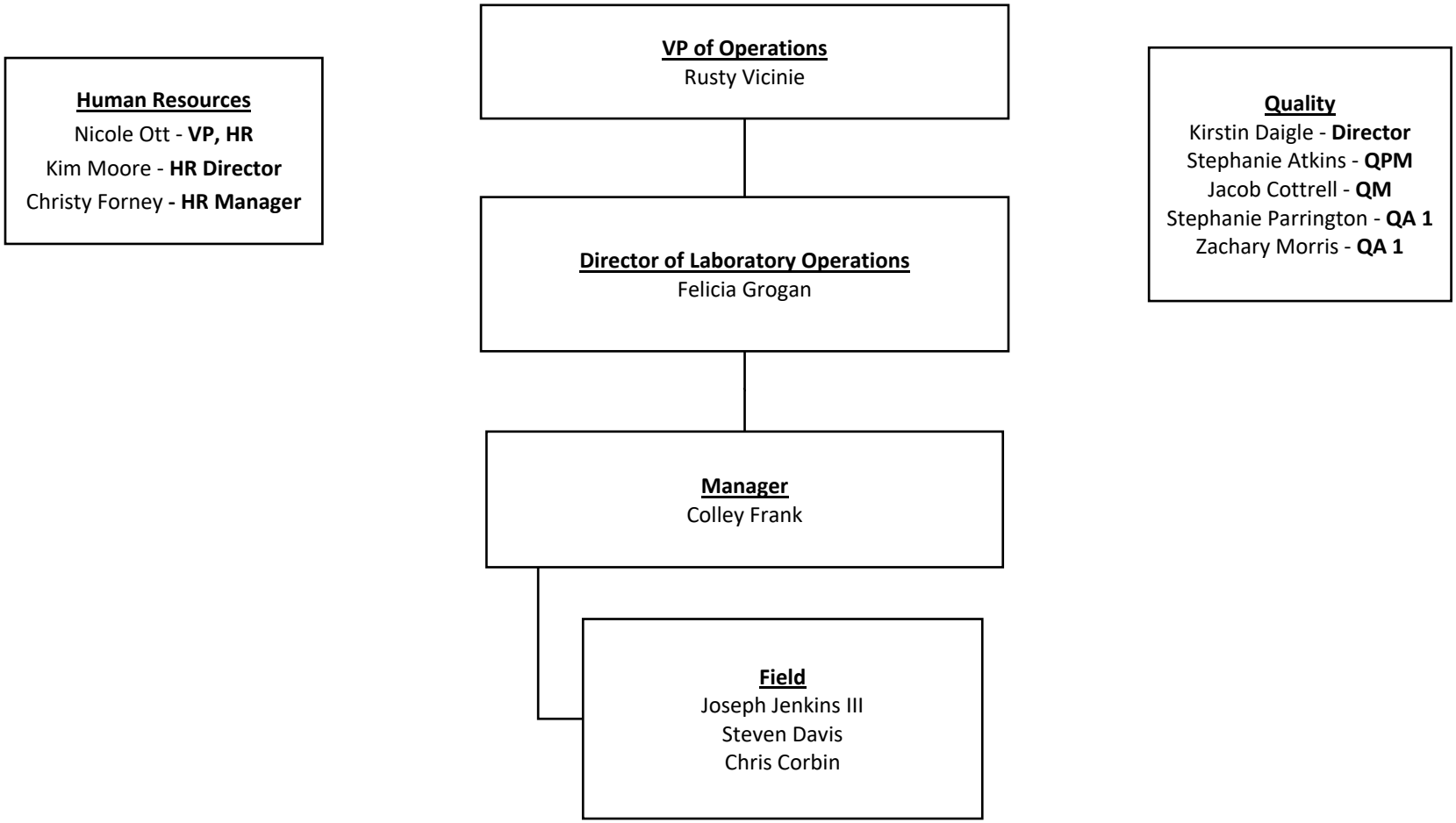
7.4.6 Greenwood – Organization Chart



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7.5 Appendix E: Equipment Listing

The equipment listed represents equipment were held by each location on the effective date of this manual. This information is subject to change without notice. External parties should contact the location for the most current information.

Equipment List: PAS – Asheville, Eden, Greenwood, Raleigh

Lab	Equipment	Manufacturer	Model / Detector	Serial #	Location
ASHE	Top Loader Balance	Sartorius	SECURA 1102-1S	36150095	Metals
ASHE	Top Loader Balance	Sartorius	SECURA 1102-1S	36150096	Metals Prep
ASHE	Analytical Balance	Sartorius	SECURA324-1S	36001910	Wet Chemistry
ASHE	Analytical Balance	Sartorius	SECURA324-1S	36001913	Wastechem
ASHE	Centrifuge	Henttich	Rotina 46 S	33180200	Metals
ASHE	Hg Analyzer	CETAC	M-7600	011502Q76	Metals
ASHE	LL Hg Analyzer	Teledyne	Quicktrace M8000	VS16050009	Metals
ASHE	ICPMS Analyzer	Agilent	7900 ICPMS-3	JP16071303	Metals
ASHE	ICP Analyzer	Thermo Elemental	ICAP 6500	ICP-2010-4901	Metals
ASHE	ICP Analyzer	Agilent Technologies	5110 ICP-OES	MY18010002	Metals
ASHE	DO Meter	YSI	5100	98E0889	Wet Chemistry
ASHE	Chlorine Meter	HACH	DR 2800	1198691	Field
ASHE	Spectrophotometer	HACH	DR 3900	1582033	Wet Chemistry
ASHE	Auto Tritrator	Metrohm	888 Titrando	1789003002162	Wet Chemistry
ASHE	Flashpoint Analyzer	Anton Parr	PMA5	60039989	Wet Chemistry
ASHE	BOD Robot	Skalar	SP2005	16275	Wet Chemistry

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Lab	Equipment	Manufacturer	Model / Detector	Serial #	Location
ASHE	Muffle Furnace	Thermo Scientific	F47900	NA	Wastechem
ASHE	BTU	PARR	6300 Bomb Calorimeter	M9807	Wastechem
ASHE	Sulfur	LECO	S-632	3122	Wastechem
ASHE	Fume Hood Enclosure	Mitsubishi Chemical Analytech	TOX-300	NA	Wastechem
ASHE	TOX	Mitsubishi Chemical Analytech	TOX-300	HOAA0034	Wastechem
ASHE	Lachat	Lachat	QuickChem 8500	10080001233	Wet Chemistry
ASHE	IC Analyzer	Metrohm	761	1761002012193	Wet Chemistry
ASHE	Lachat	Lachat	QuickChem 8500	90300001082	Wet Chemistry
ASHE	IC Analyzer	Metrohm	930	1930100004114	Wet Chemistry
ASHE	Lachat	Lachat	Quick Chem 8500 Series 2	161000001983	Wet Chemistry
ASHE	Autoclave	Market Forge	STM-EL	167625	Micro / WC
ASHE	Analytical Balance	Sartorius	SECURA 124-1S	35150124	Wet Chemistry
ASHE	Top Loader Balance	Sartorius	SECURA 1102-1S	36150094	Wet Chemistry
ASHE	Analytical Balance	Sartorius	CP 124 S	19550801	Quality
ASHE	Top Loader Balance	Sartorius	Secura 1102-1S	0037050197	Hg Room
ASHE	Analytical Balance	Mettler XS204	XS204	B237316232	Quality Office

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Lab	Equipment	Manufacturer	Model / Detector	Serial #	Location
ASHE	Top Loader Balance	Mettler Toledo	PB4002-S/FACT	1129172953	Hg Room
ASHE	Freezer	Whynter	MiniFreezer	NA	Wet Chemistry
ASHE	Metals Hotblock	SCP Science	DigiPrep MS 108 position	MSB1015060047	Metals
ASHE	Metals Hotblock	Environmental Express	SC 151	3994CEC1869	TCLP
ASHE	Hotblock	Environmental Express	SC396	4507CEC2126	Metals Prep
ASHE	TKN Block	Seal	BD50	5146U00930	Wet Chemistry
ASHE	TKN Controller	Seal	BD	5146U00931	Wet Chemistry
ASHE	Hotblock	Environmental Express	C8000	2018MDISW142	Wet Chemistry
ASHE	Hotblock	Environmental Express	C8000	2018MDISW145	Wet Chemistry
ASHE	Hotblock	Environmental Express	SC154	2018CECW4858	Metals Prep
ASHE	TKN Block	SCP Science	Digi Prep HT 100-10 / 010-520-435	HTC1018180568	Wet Chemistry
ASHE	Hotblock	Environmental Express	SC100	145CEC0240	Metals Prep
ASHE	TKN Hotblock	SCP Science	Digi Prep HT 100-40 / 010-525-205	HTC1120450647	Wet Chemistry
ASHE	Metals Hotblock	Environmental Express	36 Position	NA	Hg Room

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Lab	Equipment	Manufacturer	Model / Detector	Serial #	Location
ASHE	Metals Hotblock	Environmental Express	SC Custom 96 position	6815CECW3105	Metals Prep
ASHE	Metals Hotblock	Environmental Express	SC Custom 96 position	8940CECW3899	Metals Prep
ASHE	Metals Hotblock	SCP Science	DigiPrep MS 48 position	MSX0510120717	Metals Prep
ASHE	Hg Analyzer	Teledyne	QuickTrace M-7600	US18036001	Metals
ASHE	Hg Analyzer	Teledyne	M-8000	US18018020	Metals
ASHE	Hg Analyzer	Brooks Rand	Model III 07002	11013801	LL Hg
ASHE	Hg Analyzer	CETAC	M-8000	USZ1351004	LL Hg
ASHE	ICPMS Analyzer	Agilent	7900 ICPMS	SG20034722	Metals
ASHE	ICPMS Analyzer	Agilent	7900 ICPMS	SG21263051	Metals
ASHE	Incubator	Lab Line	302 Imperial III	0390-0143	Micro
ASHE	incubator	Attest	116	128992	Micro
ASHE	Incubator	Shel Lab / Sheldon	SLM722	03038420	Micro
ASHE	Microwave	CEM	Mars 6	MJ6909	Metals Prep
ASHE	Oven	Thermo Fisher	Heratherm OG5400	42541583	Wet Chemistry
ASHE	Oven	Precision	130	9606-003	Wet Chemistry
ASHE	Oven	Thermo Fisher	Heratherm OMH750	41729011	Wet Chemistry
ASHE	Oven	Thermo Scientific	Heratherm OGS750	42248199	Wet Chemistry

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Lab	Equipment	Manufacturer	Model / Detector	Serial #	Location
ASHE	Refrigerator	TRUE	GDM-72	1-4434482	Wet Chemistry
ASHE	Refrigerator	Upright Showcase	GN2	VIC170401	Sample Receiving
ASHE	Refrigerator	Electrolux Home Products	FFBC46L2QS-1	KA82195074	Sample Receiving
ASHE	Refrigerator	AVANTI	RM4436SS	A57770443681620100904	Lachat
ASHE	Refrigerator	Danby	DCR412W	1.0207E+11	Lachat
ASHE	Refrigerator	Sanyo	SR-361X	960972378	Micro
ASHE	Refrigerator	ABSOCOLD	NA	NA	BOD / Spec Standards
ASHE	Refrigerator	Homelabs	HME030065N	000348	BOD/Spec Standards
ASHE	Refrigerator	Home	HME030065N	000448	Micro
ASHE	Refrigerator	Home	MAXBC52SD		Lachat
ASHE	Refrigerator	General Electric	NA	NA	Wet Chemistry
ASHE	Sealer	Idexx	Quanti Tray Plus	QTP1374302995	Micro
ASHE	TCLP Tumbler	Environmental Express	NA	NA	TCLP
ASHE	Water Bath	ThermoScientific	TRCIR35	300180256	Micro
ASHE	Water Bath	ThermoScientific	TSCIR35	300382198	Micro
ASHE	Conductivity Meter	Thermo Scientific / Orion	Orion 3 Star Benchtop Cond Meter	B29683	Wet Chemistry
ASHE	Turbidity Meter	WTW	Turb 550T	201010008	Wet Chemistry
ASHE	TOX	Mitsubishi	TOX-300	HOAA0527	Wet Chemistry
ASHE	pH Meter		Accumet AB150	AB92351556	Wet Chemistry
ASHE	Turbidity Meter	Thermo Scientific	Orion AQ3010	2896604	Metals Prep

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Lab	Equipment	Manufacturer	Model / Detector	Serial #	Location
ASHE	pH Meter	Fisher	AB150	AB92354720	TCLP till April 2021 Wetchem from 11/22/2021 On
ASHE	pH Meter	Fisher	AB150	AB92354774	Wet Chemistry
ASHE	Flashpoint Analyzer	Anton Parr	PMA-500	60096098	Wet Chemistry
ASHE	Refrigerator	Arctic	NA	NA	Sample Receiving
ASHE	Refrigerator	Kysor	KPS-300	N/A	Sample Receiving
ASHE	BTU	Parr	6400 Calorimeter	6400-1702-72642	Wastechem
ASHE	Muffle Furnace	Thermo Fisher	F6010	1126104001180710	Wastechem Wet Chemistry
ASHE	Titrator	OMNIS	Titrator	001000134326	Wet Chemistry
ASHE	IC Analyzer	Metrohm	930	19300009120	Wet Chemistry
ASHE	IC Analyzer	Metrohm	930 Compact IC Flex	86135	Wet Chemistry
ASHE	LL Hex Chrom Analyzer	Metrohm	930	1930200027132	Wet Chemistry
ASHE	Sulfur	Leco Corp	SC832DR (Autosampler) /626-300-800	17171	Wastechem
ASHE	IC Analyzer	Metrohm	930	1000249671	Wet Chemistry
ASHE	LL Hex Chrom Analyzer	Metrohm	930	1930200049105	Wet Chemistry
ASHE	TOC	Shimadzu	TOC-VCSH	H54325732413	Wet Chemistry
ASHE	TOC	Shimadzu	TOC-LCSH	638-91105-22	Wet Chemistry
ASHE	Smartchem Analyzer	Westco Scientific	200	W0811147	Wet Chemistry
ASHE	Lachat	Lachat	Quick Chem 8500 Series 2	200400002277	Wet Chemistry

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Lab	Equipment	Manufacturer	Model / Detector	Serial #	Location
ASHE	Lachat	Lachat	QuickChem 8500 Series 2	110600001330	Wet Chemistry
ASHE	COD Block	SCP Science	DigiPrepMS	MSD101810003	Wet Chemistry
ASHE	Fume Hood	St. Charles	NA	NA	Wet Chemistry
ASHE	Fume Hood	St. Charles	NA	NA	Wet Chemistry
ASHE	Autosampler	Teledyne	ASX-500	031836A560	Metals
ASHE	COD Controler	SCP Science	NA	KPX1019434287	Wet Chemistry
ASHE	DO Meter	YSI	Pro Comm II	160104173	Wet Chemistry
ASHE	DO Meter	YSI	Pro Comm II	16F182651	Wet Chemistry
ASHE	DO Probe	YSI		16J102373	Wet Chemistry
ASHE	DO Probe	YSI		16F103698	Wet Chemistry
ASHE	Hg Autosampler	Brooks Rand	Merx 72	4936A14633	LL Hg
ASHE	IC Analyzer Sampler	Metrohm	858	18580020052470	Wet Chemistry
ASHE	Lachat Pump	Hach			
ASHE	Purging Trap	Brooks Rand	Merx	11023401	LL Hg
ASHE	Automatic Boat Controller	Mitsubishi	ABC-210	D8C20564	Wetchem Lab
ASHE	Colony Counters	Quebec	3325	None	Micro
ASHE	DI Water Purification System	Academie	Mili-Q	F4EN78616	Wet Chemistry
ASHE	Freezer	Roper	NA	NA	Sample Receiving
ASHE	Fume Hood Enclosure	Cetac	NA	NA	Hg Room
ASHE	Fume Hood Enclosure	NA	Canopy	NA	Wet Chemistry

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Lab	Equipment	Manufacturer	Model / Detector	Serial #	Location
ASHE	Fume Hood Enclosure	Environmental Express	Airlite Filter Enclosure	NA	Metals Prep
ASHE	Fume Hood Enclosure	Environmental Express	Airlite Filter Enclosure	NA	Metals Prep
ASHE	Fume Hood Enclosure	Environmental Express	Airlite Filter Enclosure	NA	Metals
ASHE	Fume Hood Enclosure	NA	Canopy	NA	Purge Room
ASHE	Fume Hood Enclosure	CB	Semi Enclosure	NA	Metals Prep
ASHE	Fume Hood Enclosure	CB	Semi Enclosure	NA	Metals Prep
ASHE	Fume Hood Enclosure	CB	Semi Enclosure	NA	Wastechem
ASHE	Fume Hood Enclosure	Cetac	Filter Hood	NA	LL Hg
ASHE	Fume Hood Enclosure	Cetac	ENC500 Filter Hood	NA	LL Hg
ASHE	Fume Hood Enclosure	Cetac	ENC500 Filter Hood	NA	LL Hg
ASHE	Fume Hood Enclosure	Cetac	ENC500 Filter Hood	NA	Metals
ASHE	Fume Hood Enclosure	Cetac	ENC500 Filter Hood	NA	Metals
ASHE	Fume Hood Enclosure	SCP Science	DigiPrep Filter Enclosure	NA	Metals Prep
ASHE	Fume Hood Enclosure	CB	Canopy	NA	Wastechem

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Lab	Equipment	Manufacturer	Model / Detector	Serial #	Location
ASHE	Fume Hood Enclosure	NA	NA	NA	Hg Room
ASHE	Fume Hood Enclosure	Environmental Express	Airlite Filter Enclosure	NA	Metals
ASHE	IC Analyzer Sampler	Metrohm	858	N/A	Wet Chemistry
ASHE	IC Analyzer Sampler	Metrohm	858	N/A	Wet Chemistry
ASHE	IC Analyzer Sampler	Metrohm	858	NA	Wet Chemistry
ASHE	IC Analyzer Sampler	Metrohm	858	1858002006597	Wet Chemistry
ASHE	ICP Autosampler	Agilent Technologies	SPS 4 Auotsampler	AU17253174	Metals
ASHE	Incubator	Heat Craft	LSCO5ZAS	D95M 03829	Wet Chemistry
ASHE	Lachat Autosampler	Lachat	ASX-510	100124ASX	Wet Chemistry
ASHE	Lachat Autosampler	Hach		A81010-670	Wet Chemistry
ASHE	Lachat Efluent Pump		Series III	A28911-852	Wet Chemistry
ASHE	Lachat Pump	Lachat	2200-010	2000-12	Wet Chemistry
ASHE	Lachat Unit	Lachat	QC 8500 Series 2	90300001082	Wet Chemistry
ASHE	Meter	HACH	Pocket Colormeter	960600096027	Field
ASHE	Meter	HF Scientific	Micro TP1 20008	201503159	Field
ASHE	Meter	HACH	DR 1900	15331000 1007	Field
ASHE	Meter	HACH	Pocket Colormeter	011100171841	Field
ASHE	Meter	YSI	YSI Pro 20	JC 01272	Field

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Lab	Equipment	Manufacturer	Model / Detector	Serial #	Location
ASHE	TOC Autosampler	HACH	ASIV	H52104301662	Wet Chemistry
ASHE	TOC Solids Unit	HACH	SSM-5000A	H52504300074	Wet Chemistry
ASHE	ICP Analyzer	Agilent Technologies	Agilent 5900 SVDV ICP-OES	MY2241CP03	Metals
ASHE	Sulfur	LECO	S832 DR / 626-200-400	55738	Wastechem
ASHE	pH Meter	ThemoFisher Scientific	Elite pH Tester	3107508	Sample Rec.
EDN	Autoclave	Market Forge	Sterilmatic	120 208 240	Wet Chemistry
EDN	Balance	Navigator	N14120	119362526	Wet Chemistry
EDN	Balance	Sartorius	ENTRIS224-1SUS	36402931	Wet Chemistry
EDN	Quanti tray Sealer	Idexx	2X	4518	Wet Chemistry
EDN	Meter	YSI	YSI 5100	00K0582	Wet Chemistry
EDN	Spectrophotometer	HACH	DR2800	1207494	Wet Chemistry
EDN	Smartchem Analyzer	Westco	399-Q001-01	W0404051	Wet Chemistry
EDN	UV Meter	Genesys	Genesys 10 UV	2GBC259001	Wet Chemistry
EDN	pH Meter	Fisher Scientific	Accumet AB250	AB92351813	Wet Chemistry
EDN	pH Meter	Fisher Scientific	Accumet AB250	AB92351815	Wet Chemistry
EDN	Lachat	Lachat	QuickChem 8500 Series 2	110500001329	Wet Chemistry
EDN	BOD Robot	Skalar	SP2005	18418	Wet Chemistry
EDN	Meter	YSI	5000 115v	04F9990	Wet Chemistry
EDN	Sealer	IDExx	Quanti Tray Plus	NA	Wet Chemistry
EDN	Meter	YSI	YSI 5100	13L 101224	Wet Chemistry
EDN	Freezer	Hotpoint	CTX17KAB	HV776598	Wet Chemistry

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Lab	Equipment	Manufacturer	Model / Detector	Serial #	Location
EDN	Lachat min dist. Block	Lachat	A2000-835	2000-74	Wet Chemistry
EDN	Hotblock	HACH	COD Reactor	970700016182	Wet Chemistry
EDN	Hotblock	Environmental Express	TDS024L-01	2017TDSW01	Wet Chemistry
EDN	Incubator	VWR Scientific	1545	901391	Wet Chemistry
EDN	Incubator	Precision Scientific	Low Temperature Incubator 815	NA	Micro
EDN	Incubator	FisherScientifc	Isotemp	111N0097	Micro
EDN	Magnifying Glass	ZHANGFEI			Micro
EDN	Oven	Yamato	DX 400	DX401b5200046	Wetchem
EDN	Oven	Barnstead Labline	Imperial V	1820061118035	Wet Chemistry
EDN	Oven	Fisher Scientific	IsoTemp	42116457	Wet Chemistry
EDN	Refrigerator	Hotpoint	CTX17KAB	HV776598	Wet Chemistry
EDN	Refrigerator	Migali	R-55-ST	1192185	Wet Chemistry
EDN	Refrigerator	TRUE	GDM-72	7182682	Wet Chemistry
EDN	UV Lamp	Blak-Ray	UVL-56	399-J	Wet Chemistry
EDN	Water Bath	Thermo Scientific	TSCOL19	300123312	Micro
EDN	Water Bath	Neslab	GP400	NA	Micro
EDN	Water Bath	Thermo Scientific	NA	NA	Micro
EDN	Water Bath	Precision	253	9603464	Wet Chemistry
EDN	Water Bath	VWR Scientific	1275PC	0200400	Wet Chemistry

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Lab	Equipment	Manufacturer	Model / Detector	Serial #	Location
EDN	Water Bath	Precision Scientific	66885	10AZ-11	Micro
EDN	Walkin Refrigerator	NA	NA	NA	Wet Chemistry
EDN	Walkin Refrigerator	Polar King	DT820	A16067782	Wet Chemistry
EDN	Fume Hood	Hamilton	Safeaire	NA	Wet Chemistry
EDN	Meter	HACH	Pocket Colormeter	13120E237627	Field
EDN	Desiccator	Nalgene	5317-0120	NA	Micro
EDN	Desiccator	FisherScientifc	NA	NA	Wet Chemistry
EDN	Turbidity Meter	HACH	2100Q Portable Turbidimeter	22080D000064	Eden Wetchem
GWD	Refrigerator	Avantco	178GDC49HCB	6621315218121870	Field
GWD		ThermoFisher	6936	272308-107	Field
GWD		Troemner	NA	#07621	Greenwood
GWD	Sartorius	L2200P	37040166	Greenwood	TCLP prep
GWD					
RAL	Idexx Quanti Tray Sealer	IDEXX	89-108 94.02	04412-06-0127	Micro
RAL	Analytical Balance	Mettler Toledo AG	xs105DU	1129312116	RAL
RAL	Autoclave	Market Forge	Sterilmatic STME	189107	Micro
RAL	Balance	Mettler Toledo	PB303-S	119010966	Micro
RAL	Incubator	ThermoFisher scientific	Heratherm IGS 180	43077455	Micro
RAL	Combo Meter	Thermo	Orion 5 Star	014144	Wetchem
RAL		Leica	3323	NA	Micro

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Lab	Equipment	Manufacturer	Model / Detector	Serial #	Location
RAL	Ice Machine	Manitowac			Sample Receiving
RAL	Refrigerator	GE	TBX18SIBQLWW	SV538399	Micro
RAL	Refrigerator	TRUE	GDM-37	957271	Sample Receiving
RAL	Water Bath	Precision Scientific	253	3412-103	Micro
RAL	Water Bath	Precision Scientific	51221033	69910439	Micro
RAL		Fisher Scientific	516D	111N0097	Micro
RAL		VWR	2020	303698	BOD Incubator
RAL	Freezer	Whirlpool	Roper	NA	RAL
RAL	Refrigerator	Refrigeration AVANTCO	178GDC49HCB	6098 3152 2109 2418	RAL
RAL	Pouch Sealer	Kapak	Pouch Sealer 9062	LR43737	Micro

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Equipment List: PAS-Field

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Turbidity	HF Scientific	Micro TPI	201502374	3/23/15	New	EDEN	2374	Online
Turbidity	HF Scientific	Micro TPI	201504165	5/1/15	New	RALE	4165	Online
pH/Temp/ DO/SpCon/Redox	YSI	Pro Plus	15F100211	Unknown	New	ASHE	0211	Online
pH/Temp/ DO/SpCond/ORP	YSI	Pro Plus	14K100951	2/1/15	New	GWOO	0951	Online
pH/Temp/ DO/SpCond/ORP	YSI	Pro Plus	16J101768	Unknown	New	GWOO	1768	Online
pH/Temp/ DO/SpCond/ORP	YSI	Pro Plus	14F101975	9/15/14	New	ASHE	1975	Online
pH/Temp/ DO/SpCond/ORP	YSI	Pro Plus	16B103415	Unknown	New	RALE	3415	Online
pH/Temp/ DO/SpCond/ORP	YSI	Pro Plus	16D104485	Unknown	New	GWOO	4485	Online
pH/Temp/ DO/SpCond/ORP	YSI	Pro Plus	16D104486	Unknown	New	ASHE	4486	Online
Turbidity	HF Scientific	Micro TPW	201606086	Unknown	New	RALE	6086	Online
Chlorine	Hach	Pocket Colormeter	960600096027	Unknown	New	ASHE	6027	Online
Sp. Cond	YSI	YSI 30	03K1054	Unknown	New	ASHE	1054	Online
pH/Temp/ DO/ORP	YSI	YSI Pro Plus	14F101975	Unknown	New	ASHE	1975	Online
Chlorine	Hach	1900	153310001007	1/8/16	New	ASHE	1007	Online
pH Meter	HACH	NA	2046907	Unknown	New	EDEN	6907	Online
Chlorine	HACH	NA	1111OE186825	Unknown	New	EDEN	6825	Online
TRC	HACH	Pocket Colorimeter II	040100009468	Unknown	New	GWOO	CLM 7	Online
Specific Conductivity	HACH	SensIONS	09080C852014	9/28/09	New	GWOO	2014	Online

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
pH	YSI	PRO PLUS	15F103906	8/14/15	New	GWOO	YSI-3906	Online
Turbidity	HF Scientific	20000 tpw	201701276	2/24/17	New	GWOO	HFS-1276	Online
Ph/Temp/ DO/SpCond	YSI	YSI Pro Plus	14C102244	10/27/17	New	GWOO	YSI-2244	Online
DO	YSI	YSI 20	146101037	2/1/15	New	GWOO	YSI-9719	Online
Turbidity (backup)	HF Scientific	TPW	201702246	2/16/18	New	GWOO	HFS-2246	Online
Turbidity (primary)	HF Scientific	TPW	201908195	9/9/19	New	GWOO	HFS-8195	Online
High Range Chlorine	Hach	Pocket Colorimeter II	58700-00	Unknown	New	RALE	Lot: A7250	Online
pH/Temp	Accumet	Thermometer	2027212	Unknown	New	RALE	7212	Online
Low Level Chlorine	Hach	Low Level Chlorine	1179255	Unknown	New	RALE	DR2800	Online
Turbidity	HF Scientific	TPW	201702244	Unknown	New	ASHE	2244	Online
Turbidity	HF Scientific	TPW	201702245	Unknown	New	ASHE	2245	Online
DO/Temp	YSI	YSI Pro 20	JC01272	Unknown	New	ASHE	1272	Online
Turbidity	HF Scientific	TPW	20203078	Unknown	New	GWOO	HFS-3078	Online
pH	Oakton	Model 30	2178530	1/4/21	New	KERN	8530	Online
DO	YSI	i20	20cl00349	1/4/21	New	KERN	0349	Online
Cl_2	Hach	2ii	1612OE317523	1/4/21	New	KERN	7523	Online
pH	Oakton	Model 150	2819645	1/4/21	New	KERN	9645	Online
DO	YSI	55	08F100348	1/4/21	New	KERN	0348	Online
Cl_2	Hach	2ii	241429	1/4/21	New	KERN	1429	Online
pH	Oakton	pH11	723955	1/4/21	New	KERN	3955	Online
DO	YSI	55	07C1371	1/4/21	New	KERN	1371	Online
Cl_2	Hach	2ii	17120E346073	1/4/21	New	KERN	6073	Online
pH	Oakton	pH11	2119475	1/4/21	New	KERN	9475	Online
DO	YSI	55	07C1346	1/4/21	New	KERN	1346	Online
Cl_2	Hach	2ii	1290679	1/4/21	New	KERN	0679	Online

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Cl_2	Hach	DR2800	06010D044689	1/4/21	New	KERN	4689	Online
pH	Oakton	pH30	2643422	1/40/1	New	KERN	3422	Online
Cl_2	Hach	2ii	19110A003127	1/4/21	New	KERN	3127	Online
pH	Oakton	pH30	2838793	1/4/21	New	KERN	8793	Online
Cl_2	Hach	2ii	17030E323565	1/4/21	New	KERN	3565	Online
pH	Oakton	pH30	2643396	1/4/21	New	KERN	3396	Online
Cl_2	Hach	2ii	15010E264594	1/4/21	New	KERN	4594	Online
pH	Oakton	pH30	912301	1/4/21	New	KERN	2301	Online

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Andy Rezendes Approved on 7/15/2024 8:07:59 AM
Chris Ouellette Approved on 7/15/2024 7:20:25 AM
Elena Dayn Approved on 7/3/2024 12:51:13 PM
John Trimble Approved on 7/10/2024 2:26:24 PM
Marco Soares Approved on 7/8/2024 1:16:51 PM
Pete Henriksen Approved on 7/3/2024 12:43:28 PM
Rusty Vicinie Approved on 7/3/2024 3:15:49 PM
Stephen Witkowski Approved on 7/15/2024 8:26:29 AM
Jason Hebert Approved on 7/15/2024 10:00:40 AM

Management Approval Page
Local Quality Manual
Pace Analytical Services, LLC (PAS)

Signatory Attestation: The employee's electronic signature on this management approval page affirms the approver's obligation, commitment, and responsibility to uphold the requirements of the PAS Quality Management System (QMS) described in this Quality Manual (manual) at each location for which this manual is prepared.

Refer to the page for Quality Manual Signatories to view the job title and physical address for each approver.

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Title Page
Local Quality Manual
Pace Analytical Services, LLC (PAS)

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Quality Manual Approver Information

The individuals listed below are approvers of this manual and are responsible for implementing the PAS Quality Management System (QMS) and upholding the requirements of this manual at the location(s) for which this manual was prepared, at the time this version of the manual was made effective.

The manual is not revised and released under an updated version when there is a change to personnel. Personnel information is updated during the manual's normal review and revision cycle.

If an approver changes positions, leave the company, or is on extended leave of absence, the responsibility to implement and uphold the PAS QMS automatically transfers to the primary or alternate deputy for the position until the approver is replaced and/or the approver returns to work. The individual replacing the approver automatically accepts the responsibilities associated with the original approver's attestation. Refer to Section 4.1.5.1.1 of this manual to view the deputies assigned to key personnel job titles.

PAS-Westborough

Name	Job Title	Address, City, State, ZIP	Phone
Albert (Rusty) Vicinie	Vice President of Operations	1638 Roseytown Rd #2 Greensburg, PA 15601	724-462-7664
Christopher Ouellette	Director of Lab Operations	8 Walkup Dr. Westborough, MA 01581	508-330-5828
Peter Henriksen	General Manager	8 Walkup Dr. Westborough, MA 01581	617-549-3093
Stephen Witkowski	Quality Program Manager	812 Silvia St. Ewing, NJ 08628	609-659-0268
Jason Hebert	Quality Manager	320 Forbes Blvd. Mansfield, MA 02048	508-898-9220
Marco Soares	Technical Manager (TNI)	8 Walkup Dr. Westborough, MA 01581	508-898-9220
Elena Dayn	Technical Manager (TNI)	8 Walkup Dr. Westborough, MA 01581	508-898-9220

PAS-Mansfield

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Christopher Ouellette	Director of Lab Operations	8 Walkup Dr. Westborough, MA 01581	508-330-5828
Peter Henriksen	General Manager	8 Walkup Dr. Westborough, MA 01581	617-549-3093
Stephen Witkowski	Quality Program Manager	812 Silvia St. Ewing, NJ 08628	609-659-0268
Jason Hebert	Quality Manager	320 Forbes Blvd. Mansfield, MA 02048	508-898-9220
John Trimble	Technical Manager (TNI)	320 Forbes Blvd. Mansfield, MA 02048	508-898-9220

PAS-Mansfield Air Center of Excellence

Name	Job Title	Address, City, State, ZIP	Phone
Albert (Rusty) Vicinie	Vice President of Operations	1638 Roseytown Rd #2 Greensburg, PA 15601	724-462-7664
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Peter Henriksen	General Manager	8 Walkup Dr. Westborough, MA 01581	617-549-3093
Stephen Witkowski	Quality Program Manager	812 Silvia St. Ewing, NJ 08628	609-659-0268
Jason Hebert	Quality Manager	320 Forbes Blvd. Mansfield, MA 02048	508-898-9220
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1.0 PURPOSE AND SCOPE

1.1 Purpose

This quality manual (manual) outlines the quality management system (QMS) and management structure of Pace® Analytical Services, LLC. Throughout this manual, Pace® Analytical Services, LLC is also referred to by the acronyms PAS or ENV. The acronyms PAS and ENV are interchangeable.

The QMS is also called the quality program throughout this manual and other PAS documents. "Quality Management System" and "Quality Program" are synonymous and inferred by the acronym QMS.

The QMS is the collection of policies and processes established by the senior leaders of PAS (top management) to ensure the services and products provided by PAS consistently meet relevant requirements and achieves the goal of Pace® to provide customers with high quality, cost-effective, analytical measurements, and services.

The QMS is planned to establish conformance[1] and compliance with the current published versions of these international and national quality system Standards:

- ISO/IEC 17025: *General requirements for the competence of testing and calibration laboratories*
- NELAC/TNI Standard Volume 1: *Management and Technical Requirements for Laboratories Performing Environmental Analysis*

[1]The statement of conformity to these Standards pertains only to testing and sampling activities carried out by the laboratory at its physical address, in temporary or mobile facilities, in-network, or by laboratory personnel at a customer's facility.

The QMS is also planned to achieve regulatory compliance with the various federal and state programs for which PAS locations provide compliance testing and/or holds certification or accreditation. Federal or state requirements that do not apply to all PAS locations, are provided in addendum to this manual or in other documents that supplement the manual. Customer-specific project and program requirements are not included in the manual in order to maintain client confidentiality.

- A list of accreditation and certifications held by each location associated with this manual is provided in Appendix A.
- A list of analytical testing capabilities offered by each location associated with this manual is provided in Appendix B.

1.2 Scope and Application

This manual applies to each location listed on the Title Page of this manual:

For purposes of the PAS QMS the term "location" refers to laboratories and/or service centers.

- The term "laboratory" refers to any PAS location, however named by Pace® that provides testing, collects samples (sampling), or conducts field measurement services in a fixed building, mobile unit, or in-situ (field).
- The phrase "service center" refers to any PAS location, however named by Pace® that does not perform any testing, sampling, or field measurements.

PAS locations are defined by their physical address and other information:

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- Laboratories are defined by physical address and certification/accreditation ID.
- Mobile units are defined by the address of the location to which they are assigned, by VIN (vehicle identification number), or by certification/accreditation ID.
- The phrase "satellite laboratory" is used by PAS to refer to a limited-service laboratory affiliated with a larger PAS business unit or location. Some PAS business groups refer to satellite laboratories as service centers for accounting purposes. This designation is not consistent with the definitions specified in this manual or throughout the supporting documents associated with the QMS. Regardless of any internal reference or jargon, any PAS location that generates a test result is a "laboratory" and all laboratories must comply with all requirements specified in this manual for analytical testing services.

1.2.1 Quality Manual Template

This manual was prepared from the PAS Quality Manual Template (template) created by the PAS Corporate Quality Director (CQD).

The template, known as document ID ENV-TMP-CORQ-0007, specifies the minimum requirements that every PAS location must abide by, regardless of scope of services or number of personnel, in order to achieve the objectives of the PAS Quality Policy (See Section 4.2.2).

The template is the mechanism used by top management[1] to communicate their commitment to continuously develop and improve the QMS for effectiveness, to meet customer expectations, and to comply with any statutory and regulatory requirements. Their signature of approval for the template is the mechanism used to document their responsibility.

[1]Top Management is the phrase in the TNI Standard that refers to the leaders of an organization that develop and/or release the PAS Quality Policy Statement and manual under their authority. For PAS, top management includes the Pace® Chief Executive Officer (CEO) and Chief Compliance Officer (CCO) and the PAS President, Corporate Quality Director, Senior Vice President of Operations (Sr. VPO), and the Chief Technical Officer (CTO).

The template and instructions for use of the template are released by corporate quality personnel to the local quality managers responsible for each location (Local QM). The local QM uses the template to prepare the location's manual by following the instructions provided to them. The local QM may not alter the font, structure, or content of the template, except where specified by instruction to do so.

The template is reviewed by corporate quality personnel annually and updated, if needed. More frequent review and revision may occur to manage change, to maintain conformance and compliance to relevant standards or to improve the QMS.

See standard operating procedure (SOP) ENV-SOP-CORQ-00015 *Document Management and Control* for more information.

1.2.2 Quality Manual

The quality manual is created from template ENV-TMP-CORQ-0007 by local quality personnel, who are also responsible for maintenance and management of the local manual.

- PAS locations are not permitted to alter content of the template when preparing their manual, except where specified in the template. Control of content in the manual is

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necessary to ensure consistency of implementation of the PAS quality program across the network.

- If additions or changes to the manual are needed to maintain regulatory compliance or conformance to relevant standards and these changes cannot be covered by addendum to the manual, the need for change must be raised to the PAS Corporate Quality Director, who will decide how to resolve the need.

The manual is approved for release by the individuals listed on manual approval page. The person's electronic signature affirms their commitment to implement and uphold the requirements, processes, and procedures of the PAS QMS at each location for which the manual was prepared.

The manual is updated with each release of a new version of the template, and as needed to update appendices and addendum. More frequent review and revision may be necessary when there are significant changes to the capabilities, and resources of the laboratory during the calendar year.

See SOP ENV-SOP-CORQ-00015 *Document Management and Control* for more information.

1.2.3 References to Supporting Documents

The template and the manual include references to other organization documents that support the QMS such as policies and standard operating procedures (SOPs).

These references may include the document's document control number (DC#) and the document title. This information is subject to change at the discretion of PAS. The manual and/or template are updated to reflect the editorial change during the manual's next scheduled review/revision cycle or the next time a version of the manual is released, whichever is sooner.

Each location maintains a current list of documents used by the location to support the QMS. This list, known as the controlled document or master list is readily available to personnel for their use and it provides a cross reference to the legacy document ID, where applicable. Parties external to PAS may contact the location of interest to obtain the most current version of controlled document list if desired.

2.0 REFERENCES

External references used to prepare this manual include:

- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136, most current version.
- "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846.
- "Methods for Chemical Analysis of Water and Wastes," EPA 600-4-79-020, 1979 Revised 1983, U.S. EPA.
- U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis, current version.
- U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis, current version.
- "Standard Methods for the Examination of Water and Wastewater." Current Edition APHA-AWWA-WPCF.

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- "Annual Book of ASTM Standards," Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society of Testing and Materials.
- "Annual Book of ASTM Standards," Section 11: Water and Environmental Technology, American Society of Testing and Materials.
- "NIOSH Manual of Analytical Methods," U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health, most current version.
- "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water," U.S. EPA, Environmental Monitoring and Support Laboratory – Cincinnati (Sep 1986).
- Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.
- Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079C.
- Environmental Measurements Laboratory (EML) Procedures Manual, HASL-300, US DOE, February 1992.
- Requirements for Quality Control of Analytical Data, HAZWRAP, DOE/HWP-65/R1, July 1990.
- Quality Assurance Manual for Industrial Hygiene Chemistry, AIHA, most current version.
- National Environmental Laboratory Accreditation Conference (NELAC) Standard, most current version.
- ISO/IEC 17025, General requirements for the competence of testing and calibration laboratories, 2nd Edition 2005-05-15; 3rd Edition 2017-11

  The following are implemented by normative reference to ISO/IEC 17025:

  – ISO/IEC Guide 99, *International vocabulary of metrology –Basic and general concepts and associated terms*

  – ISO/IEC 17000, *Conformity assessment – Vocabulary and general principles*
- Department of Defense Quality Systems Manual (QSM), most current version.
- TNI (The NELAC Institute) Standard, 2009 and 2016 versions.
- UCMR Laboratory Approval Requirements and Information Document, most current version.
- US EPA Drinking Water Manual, most current version.

3.0 TERMS AND DEFINITIONS

Refer to Appendix C for terms, acronyms, and definitions used in this manual and in other documents used by PAS to support the QMS.

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4.0 MANAGEMENT REQUIREMENTS

4.1 Organization

4.1.1 Legal Identity

Pace® Analytical Services, LLC (PAS) is the responsible entity authorized by the State of Minnesota to do business as a limited liability company, under the parent company, PAS Parent, Inc.

4.1.1.1 Change of Ownership

If there is a change of ownership, if a PAS location goes out of business, or if the entire organization ceases to exist, PAS management must notify regulatory authorities of the change within the time required by each state agency for which the location is certified or accredited.

Specifications for retention of records and other business information are addressed in the ownership transfer agreement and in accordance with appropriate regulatory requirements. These details are not included in this manual because each situation is specific to the change event.

4.1.2 Compliance Responsibility

PAS management has the responsibility and authority to establish and implement procedures and to maintain resources necessary to assure its testing activities are carried out in such a way to meet applicable federal and statutory requirements in addition to the requirements specified in this manual and QMS supporting documents.

4.1.3 Scope of the Quality Management System

The QMS applies to work carried out at each location covered by this manual including permanent facilities, at sites away from its permanent facilities, or in associated temporary or mobile facilities.

The permanent and mobile facilities to which this manual applies are listed on the Title Page of this manual.

4.1.4 Organization History and Information

Founded in 1978, Pace® Analytical Services, LLC (PAS) is a privately held scientific services firm operating one of the largest full-service contract laboratory and service center networks in the United States.

The business purpose of PAS is to deliver the highest standard of testing and scientific services in the market. We offer the most advanced solutions in the industry, backed by transparent data, a highly trained team, and the service and support that comes from over four decades of experience.

4.1.4.1 Organization Structure

Each PAS location is led by a management team referred to as local management[1]. Local management is responsible for making day-to-day decisions regarding the operations of the facility and implementing, and sustaining the requirements, policies, and procedures of the PAS quality program.

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[1] The term "local management" does not mean "on-site" management. Some of the roles included in the local management team, work off site or from a different PAS location.

For purpose of the QMS, the job titles associated with the local management team include Vice President of Operations (VPO), General Manager (GM), Director of Laboratory Operations (DLO), Quality Program Manager (QPM), and Quality Manager (QM).

The local management team is supported by department supervisors and team leaders, and business groups that support the organization such as HR, IT, Sales & Marketing, Finance, and EHS (Environmental Health & Safety).

Technical oversight for each location is provided by local personnel with support and guidance from the PAS Chief Technical Officer (CTO). Locations that hold TNI accreditation, also have personnel appointed to serve as the "acting technical manager for TNI, however named" to perform the duties and responsibilities of this designation per the TNI Standard. See Section 4.1.5.2.1 for more information on this TNI requirement.

The reporting relationships and responsibilities of quality personnel are independent of operations in order to safeguard impartiality. See Section 4.1.5.2 for more information.

Refer to the organization charts provided in Appendix D to view the organization structure, reporting relationships, and the interrelationships between positions.

4.1.5 Management Requirements

4.1.5.1 Personnel

Each PAS location is staffed with administrative and/or technical personnel who perform and verify work under the supervision of their direct line supervisor.

All personnel are required to perform their duties in accordance with the policies and processes outlined in this manual and in accordance with standard operating procedures (SOPs) and other quality system documents. PAS policies and procedures are designed for impartiality and integrity. When these procedures are fully implemented, personnel remain free from undue pressure and other influences that adversely impact the quality of their work or data.

4.1.5.1.1 Key Personnel

Key personnel are management positions that have the authority and responsibility to plan, direct, and control activities related to the QMS for the entire division (PAS Corporate), or for one or more PAS locations (Local).

PAS job titles classified as key personnel are as follows:

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PAS Key Personnel Positions & Deputy Assignments by Role

Job Title	Acronym	Primary / Alternate Deputy
Chief Executive Officer	CEO	President
Chief Compliance Officer	CCO	CQD / As Assigned
President	NA	CEO / Sr. VPO
Corporate Quality Director	CQD	CCO or CTO
Quality Program Manager	QPM	CQD / Peer QPM / As Assigned
Chief Technical Officer	CTO	CQD / As Assigned
Sr. VP of Operations	Sr. VPO	President / VPO
Vice President of Operations	VPO	Sr. VPO / Peer VPO
Director of Lab Operations[1]	DLO	VPO / Peer DLO or Sr. VPO
Health and Safety Director	NA	As Assigned
IT Director	NA	CIO
Quality Manager	QM	QPM /Peer QM / QAA
General Manager[1]	GM	VPO / Sr. VPO or Peer GM
Operations Manager[1]	OM	GM / DL or VPO
Technical Manager[1]	TM	CTO / Peer TM
TNI Approved TM[2]	TNI TM	Another Qualified Employee

[1:] Position is not in place at all locations.

[2:] The TNI TM is not a PAS position. See Section 4.1.5.2.1 for more information.

Some certification and accreditation programs require notification any time there is a change in key personnel. Notification requirements are tracked and upheld by the local QM, when these requirements apply.

4.1.5.2 Roles and Responsibilities

The qualifications, duties, and responsibilities for each position at Pace® are detailed in job descriptions maintained by the Pace® Human Resource personnel (HR).

The following sections provide a general overview of various management and supervisory roles and are presented in no particular order.

Chief Executive Officer (CEO): Provides leadership for overall operations; development of growth strategies; and long-range capital and strategic planning for Pace®.

Chief Compliance Officer (CCO): Provides leadership for compliance related activities.

President: Provides leadership for overall operations; oversight of regulatory and compliance standards; development of growth strategies; and long-range capital and strategic planning for PAS.

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Chief Technical Officer (CTO): Provides technical oversight and leadership to all PAS locations. Responsible for innovation and standardization of technical activities.

Corporate Director of Quality (CQD): Responsible for developing the PAS quality program and the policies and procedures that support the QMS. The CQD leads the quality team, establishing functions, responsibilities, duties, and organization structure for PAS.

Corporate Quality Program Manager (QPM): Responsible for helping local management implement, monitor, maintain and improve the PAS quality program for one or more locations in the network and for direct supervision of Quality Manager(s).

Director of Information Technology: Oversees and delivers the systems and processes of information technology used by PAS. These systems include Laboratory Information Management Systems (LIMS); data acquisition, reduction, and reporting software; virus-protection, communication tools, and ensuring the integrity, security of electronic data, and associated policies and procedures.

Sr. Vice President of Operations (Sr VPO): Provides leadership, direction, and insight necessary to achieve strategic initiatives. Develops and improves processes, structure, and allocation of resources for operations for all PAS.

Vice-President of Operations (VPO): Provides leadership, guidance, and resources, including allocation of personnel, necessary to achieve the strategic goals of the organization and the PAS quality program to one or more PAS locations.

Director of Laboratory Operations (DLO): See descriptions for VPO and General Manager. The DLO is an intermediary management position that provides leadership and resources to one or more locations.

General Manager (GM): The GM is responsible for overall administration and operation of one or more PAS locations. Although task duties associated with this responsibility may be delegated, the GM is responsible for ensuring all duties and activities of the locations they oversee comply with the PAS QMS, the PAS EHS program, and with any applicable statutory, regulatory requirements or program requirements. If a GM is not assigned to the location, these responsibilities are transferred to the Operations Manager (OM) or DLO. If no DLO, to the VPO.

Any GM of a TNI Accredited laboratory is also responsible for the designation of technical personnel to serve as acting technical managers for TNI for the fields of accreditation held by the laboratory (See Section 4.1.5.2.1) and for notifying the accreditation body (AB) of any extended absence or reassignment of these designations.

Quality Manager (QM): The QM oversees and monitors the implementation, compliance, and improvement of the QMS and communicates gaps, deviations, and opportunities for improvement to local and corporate laboratory management. The QM is independent of the operation and analytical activities for which they provide oversight and has the authority to carry out the roles and responsibilities of their position without outside influence.

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The QM:

- serves as the focal point for QA/QC protocol decisions and oversees review of QC data for trend analysis;
- evaluates data objectively and performs assessments without outside influence;
- has documented training and experience in QA/QC procedures and the PAS quality system;
- has a general knowledge of the analytical methods offered by the laboratory;
- coordinates and conducts internal systems and technical audits;
- notifies laboratory management of deficiencies in the quality system;
- monitors corrective actions;
- provides support to technical personnel and may serve as the primary deputy for the acting TNI Technical Manager(s).

Manager-Client Services (CSM): This position is responsible for the training and supervision of project manager(s) and/or shipping, receiving and courier personnel. The primary responsibility of the CSM is to ensure projects are successfully managed to meet the expectations and needs of PAS customers.

Department Managers/Supervisors/Team Lead): These positions are responsible for administrative and operations management and implementation of the QMS in the work area he/she oversees. These responsibilities include but are not limited to: training and supervision of personnel, monitoring work activity to maintain compliance with this manual, SOPs, policies and other instructional documents that support the QMS; method development, validation and the establishment and implementation of SOPs to assure regulatory compliance and suitability for the intended purpose; monitoring QA/QC performance, proper handling and reporting of nonconforming work; purchasing of supplies and equipment adequate for use; maintaining instrumentation and equipment in proper working order and calibration; and general maintenance of administrative and technical processes and procedures established by the laboratory.

Operations Manager (OM): The OM is responsible for management of production and/or other duties assigned by the GM.

4.1.5.2.1 Approved Technical Manager (TNI Accreditation Only)

The requirements in this subsection apply only to PAS locations that are NELAC/TNI accredited.

The TNI Standard specifies requirements for the qualification and duties of technical personnel. The TNI Standard lists these duties under the reference "technical manager(s), however named."

At PAS, these duties closely correlate with the responsibilities and duties outlined in the PAS job descriptions for managers, supervisors, team leads, and/or scientists. However, these duties do not need to be associated with

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any specific job title and can be assigned to any one or more PAS employees that meets the qualifications specified in the TNI Standard.

Refer to the applicable version of the TNI Standard to view the required qualifications for each discipline.

PAS locations that are TNI accredited must designate one or more employees to perform these duties and submit these qualifications to the TNI accreditation body (AB) for approval.

Employees approved by the TNI AB, to perform these duties retain their Pace® assigned job title.

When TNI Accreditation Bodies (AB) refer to these employees as 'technical manager' or 'technical director' on the official certificate or the scope of accreditation, this reference is referring to their approval to perform duties of the 'technical manager, however named' as specified in the TNI Standard and not to a PAS job title.

The duties of any approved technical manager for TNI, however named, can be completed in person or remotely. If an employee that is an approved technical manager for TNI is completely absent from work or on a leave of absence for more than 15 calendar days, the duties and responsibilities specified in the TNI Standard are temporarily reassigned to another employee that meets the qualifications for the technology or field of accreditation. If the employee's absence exceeds 35 calendar days, the local QM must formally notify the TNI primary AB of the absence and the details of reassignment of duties in writing.

4.1.5.3 Conflict of Interest

A conflict of interest is a situation where a person has competing interests that may affect impartiality. It is the policy of Pace® to ensure business relationships, decisions and transactions do not place personal interest ahead of the organization, customers, colleagues, job responsibilities or the public we serve. Conflict of interest is avoided by making personnel aware of circumstances that conflict or appear to conflict with impartiality and/or designing process and procedures to include checks and balances to prevent conflict and ensure impartiality.

See the current version of policy COR-POL-0004 *Code of Ethics and Professional Conduct* for more information.

4.1.5.4 Confidentiality

PAS management is committed to preserving the confidentiality of Pace® customers and confidentiality of Pace® business information.

Client information obtained or created during work activities is considered confidential and is protected from intentional release to any person or entity other than the client or the client's authorized representative, except when Pace® is required by law to release confidential information to another party, such as a regulatory agency or for litigation purposes. In which case, Pace® will notify the client

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of the release of information and the information provided, unless notification is prohibited by law.

When Pace® obtains information about the customer from a source other than the customer, Pace® will keep the source of the information confidential unless disclosure is agreed upon by the source.

The terms of client confidentiality are included in PAS Standard Terms and Conditions (T&C). With the acceptance of the T&C and/or the implicit contract for analytical services that occurs when the client sends samples to PAS for testing, the client authorizes Pace® to release confidential information when required. Other procedures used by PAS to maintain confidentiality include:

- A Code of Ethics and Professional Conduct policy that covers this topic (COR-POL-0004);
- A Confidentiality Agreement which supervisory and sales personnel and other positions are required to sign at the time of employment and abide by the conditions of throughout employment;
- Record retention and disposal procedures that assure confidentiality is maintained; and
- Physical access controls and encryption of electronic data.

See policy COR-POL-0004 *Code of Ethics and Professional Conduct* for more information.

4.1.5.5 Communication

Communication is defined as the imparting or exchanging of news and information. Effective (good) communication occurs when the people included in the communication gets the point and understands it.

4.1.5.5.1 Workplace Communication

Effective communication in the workplace is necessary to assure work is performed correctly, efficiently, and in accordance with client specifications.

Instructions for how to conduct testing and other work activities are communicated to personnel via written policies, standard operating procedures, and other work instructions.

Information about PAS performance (positive and negative) and ideas for improvement are communicated to personnel using various communication channels such as face to face meetings, video conferencing, conference calls, email, memoranda, written reports, and posters.

4.1.5.5.2 External Communication

Communication with external parties such as customers, vendors, business partners, and regulatory agencies takes place every day.

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PAS management is responsible for training personnel to communicate in professional and respectful ways to build strong relationships and to avoid misunderstanding.

4.2 Quality Management System

4.2.1 Quality Management System Objectives

The objectives of the PAS QMS are to provide clients with consistent, exemplary professional service, and objective work product that is of known and documented quality that meets their requirements for data usability and regulatory compliance.

Objective work products are analytical services, data, test results, and information that is not influenced by personal feeling or opinions. The quality of being objective is also known as 'impartiality.'

4.2.1.1 Impartiality

PAS achieves and maintains impartiality by establishing an organizational structure that safeguards impartiality (See 4.1.4.1) and implementing and adhering to the policies and processes of the QMS outlined in this manual, which are based on industry accepted standards and methodologies.

PAS procedures for handling nonconforming work (See 4.9), corrective and preventive actions (See 4.11, 4.12) and management review (See 4.15) are the primary mechanisms used to identify risk to impartiality and to prompt actions necessary to eliminate or reduce the threat when risk to impartiality is suspected or confirmed.

4.2.1.2 Risk and Opportunity Assessment

Risks are variables that make achieving the goals and objectives of the QMS uncertain.

An opportunity is something that has potential positive consequences for the organization.

PAS personnel manage risks and opportunities on a daily basis by following policies, procedures and processes that support the QMS. Some ways in which the QMS is designed to identify, minimize, or eliminate risk on a daily basis include but are not limited to:

- Capability and capacity reviews of each analytical service request to assure the laboratory can meet the customer's requirements;
- Maintenance of accreditation and certification for test methods in multiple states and programs to cover a broad range of authority for regulatory compliance;
- SOPs and other controlled instructional documents are provided to personnel to eliminate variability in the process. These documents include actions to counter risk factors inherent in the process and are reviewed on a regular basis for on-going suitability and relevancy;
- Participation in proficiency testing programs and auditing activities to verify on-going competency and comparability in performance;

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- Provision of on-the-job training and established protocol for quality control (QC) corrective action for nonconforming events;
- An established program for ethics, and data integrity;
- Tiered data review process;
- Culture of continuous improvement;
- Monitoring activities to assess daily and long-term performance; and
- Annual critical review of the effectiveness of the QMS.

PAS also promotes a continuous improvement culture based on the principles of lean manufacturing. These principles include 3P (Process, Productivity, Performance) and Kaizen. 3P is a platform used by PAS to share best practices and standardization across the network to achieve operational excellence. Kaizen is a team-based process used to implement tools and philosophies of lean to reduce waste and achieve flow with the purpose of improving both external and internal customer satisfaction. The PAS lean program and activities help to mitigate risk because they generate a collective understanding of vulnerabilities and utilize group-effort to develop and implement solutions at all levels.

Risk and opportunities may also be formally identified using specific risk and opportunity assessment methods such as SWOT Analysis (Strength, Weakness, Opportunity, Threats) and 3-Stage Impact/Probability Grids.

4.2.1.3 Communication of the Quality Management System

This manual is the primary mechanism used by PAS management to communicate the QMS to personnel.

To assure personnel understand and implement the quality program outlined in the manual:

- PAS personnel are required to sign a Read and Acknowledgement Statement to confirm the employee has:

  1) been informed of the manual by management;

  2) has access to the manual;

  3) has read the manual;

  4) understands the content of the manual; and

  5) agrees to abide by the requirements, policies, and procedures therein.

- Personnel are informed that the manual provides the "what" of the QMS. The "how to" implementation of the QMS is provided in policy, SOPs, standard work instructions, and other instructional documents.

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- This manual and supporting policies and procedures are made readily accessible to personnel in the area where the work activity is performed.

4.2.2 Quality Policy Statement

The quality policy of PAS is to provide customers with data of known and documented quality fit for their intended purpose. PAS achieves this policy by implementing the QMS defined in this manual, by following industry accepted protocol for analytical testing and quality assurance and quality control (QA/QC) activities, by conformance with published and industry accepted testing methodologies, and by compliance with international and national standards for the competency and/or accreditation of testing laboratories.

Intrinsic to this policy statement is each of the following principles:

- PAS will provide customers with reliable, consistent, and professional service. This is accomplished by making sure each PAS location has the resources necessary to maintain capability and capacity; that staff are trained and competent to perform the tasks they are assigned; that client-facing staff are trained and prepared to find solutions to problems and to assist customers with their needs for analytical services. Customer feedback, both positive and negative, is shared with personnel and used to identify opportunities for improvement.
- PAS maintains a quality program that complies with applicable state, federal, and industry standards for analytical testing and competency.
- PAS management provides training to personnel so that all personnel are familiar with the QMS outlined in this manual and that they understand that implementation of the QMS is achieved by adherence to the Pace® and PAS policies and procedures.
- PAS management continuously evaluates and improves the effectiveness of the QMS by responding to customer feedback, and other measures of performance, such as but not limited to the results of internal/external audits, proficiency testing, metrics, trend reports, and annual and periodic management reviews.

4.2.2.1 Ethics Policy / Data Integrity Program

Pace® has established a comprehensive ethics and data integrity program that is communicated to all Pace® employees so that they understand what is expected of them. The program is designed to promote a mindset of ethical behavior and professional conduct that is applied to all work activities.

The key elements of the Pace® Ethics / Data Integrity Program include:

- Ethics Policy (COR-POL-0004);
- Standardized data integrity training course taken by all new employees on hire and a yearly refresher data integrity training course for all existing employees;
- Policy Acknowledgement Statements that all Pace® personnel, including contract and temporary, are required to sign at the time of employment and again during annual refresher training to document the employee's commitment and obligation to abide by the company's standards for ethics, data integrity and confidentiality;

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- SOPs that provide instructions for how to carry out a test method or process to assure tasks are done correctly and consistently by each employee;
- On the Job Training;
- Data integrity monitoring activities which include, but are not limited to: primary, secondary and completeness data reviews, internal technical and system audits, data audits, data surveillance, and proficiency testing; and
- Confidential reporting process for alleged ethics and data integrity issues.

All PAS managers and supervisors are expected to provide a work environment where personnel feel safe and can report unethical or improper behavior in complete confidence without fear of retaliation. Retaliation against any employee that reports a concern is not tolerated.

Pace® has engaged Lighthouse Services, Inc. to provide personnel with an anonymous reporting process available to them 24 hours a day/7 days per week. The alert line may be used by any employee to report potential violations of the company's ethics and data integrity program. Reports are forwarded to Pace® leadership to investigate and resolve the matter. Investigations concerning data integrity are kept confidential.

See COR-POL-0001 *Compliance Alertline* for more information.

Posters and flyers with the compliance alert line information must be prominently posted in each PAS location for personnel reference.

Compliance Alert Line Information:

English Speaking US & Canada	(844) 940-0003
Spanish Speaking North America	(800) 216-1288
Internet	www/lighthouse-services.com/pacelabs
Email	reports@lighthouse-services.com

4.2.3 Management Commitment: Quality Management System

Evidence of management's commitment for the development, maintenance, and on-going improvement of the QMS is provided by the application of their signature of approval to the template and/or manual. Their signature confirms they understand their responsibility to implement the QMS outlined in this manual, to communicate the quality program to personnel, and to uphold requirements of the program during work activities.

4.2.4 Management Commitment: Customer Service

Management communicates the importance of meeting customer and regulatory requirements to personnel by training personnel on the QMS outlined in this manual, implementing the QMS outlined in this manual, and upholding these requirements for all work activities.

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4.2.5 Supporting Procedures

References to processes and procedures that support the QMS are included throughout this manual. The structure of the document management system is outlined in SOP ENV-SOP-CORQ-0015 *Document Management* and summarized in the following subsections.

4.2.5.1 Quality Management System Document Structure

Documents associated with the QMS are classified into document types that identify the purpose of the document and establish how the document is managed and /or controlled.

Examples: Types of PAS Internally Created Documents

Document Type	Purpose
Quality Manual	Outlines the PAS QMS and structure and how it works for a system including policy, goals, objectives and detailed explanation of the system and the requirements for implementation of system. Includes roles and responsibilities, relationships, procedures, systems, and other information necessary to meet the objectives of the system described.
Policy	Provide requirements and rules for a process and is used to set course of actions and to guide and influence decisions. Policy describes the "what," not the "how."
Standard Operating Procedure	Provide written and consistent set of instructions or steps for execution of a routine process, method, or set of tasks performed. Assures that activities are performed properly in accordance with applicable requirements.
Standard Work Instruction	Provide step by step visual and/or written instruction to perform a specific task to improve competency, minimize variability, reduce work injury and strain, or to boost efficiency and quality of work (performance). SWI are associated with an SOP unless the task described is unrelated to generation of or contribution to environmental data or analytical results.
Template	Pre-formatted document that serves as a starting point for a new document.
Guide	Assists users in using a particular product; or a technical interpretation of a method or process by which PAS locations must abide.
Form / Worksheet	Used for a variety of purposes such as to provide a standardized format to record observations, to provide information to supplement an SOP.

Examples: Types of External Documents used by PAS

Certificate	Lists parameters, methods, and matrices for which the location is certified/accredited to perform within the authority of the issuing regulatory agency or accreditation body.
Reference Document	Provide information, protocol, instructions, and/or requirements. Issued by the specifier. Examples include ISO/IEC, TNI, DoD/DOE and published referenced methods such as Standard Methods, ASTM, SW846, EPA, and federal and state regulatory bodies.
Project Document	Provides requirements necessary to meet individual client expectations for intended use of data. Examples include project quality assurance plans (QAPP), client-program technical specifications, contracts, and other agreements.

Project documents are managed as external documents and any requirements for work specified is followed when work for the project is performed. If the project document is less stringent than the PAS QMS, policies, or SOPs, and/or is less stringent than applicable federal or state requirements, PAS locations are still required to meet the minimum requirements of the PAS QMS and any applicable statutory or federal requirements in addition to the requirements specified in the project

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document. Information and requirements from project documents are not incorporated into PAS policy or SOPs in order to maintain client confidentiality.

Document types are ranked to identify which documents takes precedence when there is an actual or perceived conflict between documents and to establish the hierarchal relationships between documents. The ranking system also provides information to document writers and reviewers to assure downline documents agree with documents of higher rank.

PAS Document Hierarchy

Rank	Document Type
1	External Reference Documents (Standards, Test Methods)
2	Quality Manual Template
3	Corporate & Division Policy
4	Corporate & Division SOP
5	Division SWI, Guides, Forms/Worksheets
6	Local Quality Manual
7	Local SOP
8	Local SWI, Guides, Forms/Worksheets

4.2.6 Roles and Responsibilities

The roles and responsibilities for technical management and the quality manager is provided in section 4.1.5.2.

4.2.7 Change Management

When significant changes to the PAS QMS are planned, these changes are managed by corporate quality personnel to assure that the integrity of the QMS is maintained.

4.3 Document Control

4.3.1 General

PAS uses electronic document management software (eDMS) to control documents that support the PAS QMS. eDMS provides centralized distribution and access to all documents used by PAS. All PAS locations must use the PAS eDMS system unless an exemption to this requirement has been granted by the PAS Corporate Quality Director.

eDMS automates the process for unique document identification, version control, approval, access, and archival and restricts access to archived documents except to authorized users to prevent the use of obsolete documents.

The local QM maintains a master list of controlled documents used at each location. The master list minimally includes the document control number, document title, and current revision status and is made available to personnel for their reference.

See SOP ENV-SOP-CORQ-0015 *Document Management* for more information.

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4.3.2 Document Approval and Issue

Documents that support the QMS are reviewed by qualified personnel and approved by management prior to release for use.

Only the approved versions of documents are available to personnel for use unless a draft document is authorized by management.

The managers responsible for authorization of each document is specific to the document type.

See SOP ENV-SOP-CORQ-0015 *Document Management* for more information.

4.3.3 Document Review and Change

Unless a more frequent review is required by regulatory, certification or accreditation program documents are reviewed at least every two years to ensure the documents remain current, appropriate, and relevant.

Documents are also informally reviewed every time the document is used. Personnel are expected to refer to and follow instructions in controlled documents when they conduct their work activities. Consequently, any concerns or problems with the document should be caught and brought to the attention of management on an on-going basis.

Documents are revised whenever necessary to ensure the document remains usable and correct. Older document versions and documents no longer needed are made obsolete and archived for historical purposes.

PAS does not allow hand-edits of documents. If an interim change is needed pending re-issue of the document, the interim change is communicated to those that use the document using a formal communication channel, such as change in progress form, email, or memorandum.

The document review, revision, and archival process is managed by quality personnel at the location from which the document was released using the procedures established in SOP ENV-SOP-CORQ-0015 *Document Management*.

4.4 Analytical Service Request, Tender, and Contract Review

PAS management and/or client service personnel perform thorough reviews of requests and contracts for analytical services to verify the location(s) performing the work has the capability, capacity, and resources necessary to successfully meet the customer's needs. These review procedures are described in SOP ENV-SOP-WES2-0125 Review of Analytical Requests.

The procedures in this SOP(s) are established to ensure:

- The PAS location(s) performing the work understand the purpose of data collection in order to ensure the test methods requested are appropriate for the intended use of the data and capable of meeting the client's data quality objectives;
- PAS locations and any external subcontractor(s) have the capability, capacity, and resources to meet the project requirements and expectations within the requested time for delivery of work product;
- Any concerns that arise from review are discussed and resolved with the client;

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- Any discrepancies between the PAS QMS, statutory or regulatory requirements and the client request are resolved; and
- The results of review and any correspondence with the client related to this process and/or any changes made to the contract are recorded and retained for historical purposes.

Capability review confirms that the PAS locations contracted to perform the work and any internal or external subcontractors hold required certification/accreditation for the test method, matrix, and analyte and verifies the location can achieve the client's target compound list and data quality objectives (DQOs) for analytical sensitivity and reporting limits, QA/QC protocol, and hardcopy test report and electronic data deliverable (EDD) formats.

Capacity review verifies that the in-network locations and any potential subcontractors are able to manage the sample load and deliver work production within the delivery time requested.

Resource review verifies that the location and any potential subcontractors have adequate qualified personnel with the skills and competency to perform the test methods and services requested and sufficient and proper equipment and instrumentation needed to perform the services requested.

Customers must be notified when there is a deviation from the contract after acceptance of the contract by both parties. Instructions for this notification process must be included in the laboratory's SOP for this process.

4.5 Subcontracting (Internal and External)

The terms 'subcontract' and "subcontracting" refers to analytical work done by an organization external to Pace® (External Subcontracting) or by a Pace® location with an address different than the address listed on the cover page of the test report (Internal Subcontracting).

The PAS network offers comprehensive analytical capability and capacity to ensure Pace® can meet a diverse range of client needs for any type of project. If a PAS laboratory receives a request for analytical services and it cannot fulfill the project specifications, the location's client services team will collaborate with the client to place the work within the PAS network.

When it is not possible to place the work within network, the location will, with documented client approval, subcontract the work to a subcontractor that has the capabilities to meet the project specifications and can meet the same commitment agreed on between the location and the client.

Whenever work is subcontracted, the PAS location responsible for management of the project verifies each of these qualifications:

- The internal or external subcontractor has the proper accreditation/certifications required for the project and these are current; and
- The use of the internal or external subcontractor is approved by the client and/or regulatory agency when such approval is required by the customer. Record of customer approval is retained in the project record.

External subcontractors selected by Pace® must be pre-qualified by quality personnel to verify their QMS is similar to Pace® and complies with all relevant Standards such, as ISO/IEC 17025 and the TNI Standard(s) and/or federal and state regulatory requirements. The list of approved subcontractors for each location is maintained by local quality personnel. Pre-qualification of a subcontractor does not eliminate the requirement for the PAS location placing work to verify the

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subcontractor has the certifications, capability, capacity, and resources to perform work on behalf of Pace® on a project-specific basis.

For all subcontracted work, the PAS location placing the work internally or externally is responsible to ensure project specifications are always communicated to and understood by the subcontractor.

4.6 Purchasing Services and Supplies

Vendors that provide services and supplies to PAS are qualified to meet the needs of Pace®. These needs include but are not limited to competitive pricing, capacity to fill purchase orders, quality of product, customer service, and business reputation and stability. Evidence of this qualification is the availability to purchase services and supplies from the vendor in the corporate purchasing system.

PAS locations may purchase goods and services from any supplier in the purchasing system.

The specifications (type, class, grade, tolerance, purity, etc.) of supplies, equipment, reagents, standard reference materials and other consumables used in the testing process are specified in SOPs. The SOP specifications are based on the governing requirements of the approved reference methods and any additional program driven regulatory specification, such as drinking water compliance.

All requisitions for materials and consumables are approved by local management who is responsible for ensuring the services and supplies procured and received are fit for intended use.

4.7 Customer Service

Project details and management is managed by PAS client services personnel.

4.7.1 Commitment to Meet Customer Expectations

PAS personnel collaborate closely with our customers to ensure their needs are met and to establish their confidence in the capability of PAS to meet their needs for analytical services and expectations for service.

The project manager (PM) is the customer's primary point of contact for each analytical service request (work order). The PM gathers information from the customer to ensure the details of their request are understood. After samples are received, the PM monitors the progress of the project and alerts the customer of any delays or excursions that may adversely impact data usability. Supervisors are expected to keep the PM informed of project status and any delays or key issues, so that the PM can keep the client informed.

PAS encourages customers to visit our locations to learn more about the capabilities, observe performance and to meet personnel.

PAS customers expect confidentiality. Personnel will not divulge or release information to a third party without proper authorization unless the information is required for litigation purposes. See Section 4.1.5.4 of this manual and policy COR-POL-0004 *Code of Ethics and Professional Conduct* for more information on the policy for client confidentiality.

4.7.2 Customer Feedback

PAS actively seeks positive and negative feedback from customers through surveys and direct communication. Information from the client about their experience working with PAS and their satisfaction with work product is used to enhance processes and practices and to improve decision making. Customer feedback is reviewed to identify risk and opportunity. Corrective,

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preventive, or continuous improvement actions are taken based on nature of and/or feedback trends.

Also see sections 4.9, 4.10, 4.11, 4.12, 4.14, and 4.15 for more information about how customer feedback is managed by PAS and used to enhance the QMS.

4.8 Complaints

Per ISO/IEC 17025:2017, a complaint is a formal expression of dissatisfaction with the performance of a service or product originating from a party external to the organization. PAS believes complaints provide opportunities to improve processes and/or build stronger working relationships with our clients.

The PAS complaint resolution process is specified in corporate SOP ENV-SOP-CORQ-0020 and all PAS locations are required to use this SOP as their process.

Complaints are thoroughly reviewed to determine if it is valid. If the complaint is valid, action is taken to resolve the situation with the customer. In accordance with ISO/IEC 17025 requirements, the response to the customer is independently reviewed before it is given to ensure the response from PAS is thorough and proper for the situation.

Complaint information is captured in a centralized database and the data is used by PAS management personnel to identify trends and opportunities for improvement during Annual Management Review - See Section 4.15 for more information about annual management review.

4.9 Nonconforming Work

4.9.1 Definition of Nonconforming Work

Nonconforming work is work that does not conform to customer requirements, standard specifications, policies, and procedures, or that does not meet acceptance criteria.

The discovery of non-conforming work comes from various sources which include, but are not limited to:

- results of quality control samples and instrument calibrations;
- quality checks on consumables and materials;
- general observations of personnel;
- data review;
- proficiency testing;
- internal and external audits;
- complaints and feedback;
- management review and reports; and
- regulatory and certification and accreditation actions.

The way in which the laboratory or service center manages nonconforming work depends on the significance and impact (risk) of the issue. Some issues may simply require correction, others may require investigation, corrective action (See 4.11) and/or data recall (See 4.16).

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When the location releases data and test results associated with nonconforming QC and acceptance criteria, test results are qualified, or non-conformances are noted in the final analytical report to apprise the data user of the situation. (See 5.10)

Nonconforming work also includes unauthorized departure from policies, procedures, and test methods. Authorized departures are explained in the following subsections. Situations that do not conform to these conditions are considered unauthorized departure(s).

4.9.1.1 Authorized Departure from SOPs

Departures from an SOP may sometimes be necessary to correct an error in an SOP or to resolve a complex problem. For example, to mitigate complex matrix interference.

An authorized departure from a test method SOP is one that has been reviewed and approved by the department leader, however named, of the work area in which the test method is performed. The leader, when authorizing a departure from an SOP, accepts full responsibility to ensure the departure does not conflict with Pace® or PAS policy or procedure, does not affect statutory, regulatory or program compliance and does not adversely affect data integrity or usability.

Departure from administrative or process-oriented SOPs must be approved by the local QM.

Documentation of the reason for the SOP departures must be retained with management approval. Approved departures from test method SOPs should be noted in the final test report to advise the data user.

See SOP ENV-SOP-CORQ-0016 *SOP for SOPs and SWI*, for more information.

4.9.1.2 Authorized Departure from Test Methods (Method Modifications)

When test results are associated to a published reference test method, the location's test method SOP must be consistent with the test method. If the test method is mandated for use by a specific regulatory program such as drinking water, wastewater or a certification or accreditation program, such as TNI/NELAC, the SOP must comply with or include these requirements, or the resulting data and test results cannot be used for regulatory compliance purposes.

If the procedures in the SOP are modified from the test method, these modifications must be clearly identified in the SOP. The conditions under which the location may establish an SOP that is modified from these reference methods or regulatory program and what is considered a modification are specified in ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification.*

Client requests to deviate from the test method are managed as client requests to depart from the test method SOP since it is the SOP that the location follows when performing work.

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4.9.1.3 Stop Work Authority

Stop Work Authority provides PAS personnel with capability to temporarily stop work when there is a perceived unsafe condition or situation that may affect data integrity or the safety of personnel.

All personnel have the authority to initiate and request a stop work order when necessary to preserve data integrity or safety of workers.

The need for the stop work order and resolution of the problem must be confirmed by subject matter experts and resumption of work must be approved as follows:

- For stop work orders related to environmental health and safety (EHS) and/or waste management, the decision to stop work may be made in real time to protect the safety of the worker. Actions taken to correct the problem and lift the stop work order are made by the EHS Director, or the deputies assigned to these positions.
- Any employee may request a temporary stop work order for concerns related to data integrity. However, the request must be reviewed and the need to stop work affirmed by the Quality Manager to confirm the concern is valid. The decision to uphold the stop work order must be made jointly by the local QM, the QPM, the GM or DLO, and the VPO. The Corporate Quality Director and Chief Technical Officer, and other members of PAS management are consulted as needed. The actions taken to correct the problem and authorization to lift the stop work order are made by the same management team.

4.10 Continuous Improvement

The PAS QMS is designed to achieve continuous improvement through the implementation of the quality policy and objectives outlined in this manual. Information about laboratory and service center activities and performance is gained from sources such as customer feedback, audits, QC, trend analysis, business analytics, management reports, proficiency testing, and management systems review. This information is subsequently used during the corrective action (see section 4.11) and preventive action (see section 4.12) processes and during annual review of the management system (see section 4.15) to establish goals and objectives for improvement.

PAS also promotes a continuous improvement culture based on the principles of lean manufacturing. These principles include 3P (Process, Productivity, Performance) and Kaizen. 3P is a platform used by Pace to share best practices and standardization across the network to achieve operational excellence. Kaizen is a team-based process used to implement tools and philosophies of lean to reduce waste and achieve flow with the purpose of improving both external and internal customer satisfaction. All activities of 3P and Lean must conform with the requirements of this quality manual and supporting policies and procedures.

4.11 Corrective Action

Corrective action is a process used to eliminate the cause of a detected nonconformity. It is different from a correction. A correction is an action taken to fix an immediate problem but that does not resolve the underlying cause of why the problem occurred. The objective of corrective action is to find the underlying cause(s) of the problem and to put in place fixes to prevent the problem from

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happening again. The corrective action process, referred to as CAPA, is one of the most effective tools used by PAS to prevent nonconforming work, identify risk and opportunity, and improve service to our customers.

PAS has two general processes for corrective action, the application of which process is used depends on the type of nonconformity.

Quality control (QC) exceptions (nonconformance) that occur during routine testing is investigated through troubleshooting and required actions for correction is specified in policies and SOPs. When action is not taken, cannot be taken, or is not successful, test results associated with the nonconforming work are qualified in the final test report. Documentation of the nonconformance and corrective action taken is documented in the analytical record.

A 7-stage corrective action process is used when there is a recurring problem. These problems are identified through various activities such as but not limited to quality control trends, internal and external audits, management review, customer feedback, and general observation.

The 7 Stage CAPA Process for PAS includes:

1) Identification and Containment
2) Evaluation
3) Investigation
4) Cause Analysis
5) Action Plan
6) Implementation
7) Follow Up and Effectiveness Review

PAS procedures for corrective action are specified in corporate SOP ENV-SOP-CORQ-0018, *Procedure for Corrective and Preventive Action*. Some key concepts and activities related to the PAS corrective action process is provided in the next three subsections.

4.11.1 Cause Analysis (AKA Root Cause Analysis)

Cause analysis is the process of investigation used to identify the underlying cause(s) of the problem. After causal factors are identified, ways to mitigate the causal factors are identified and action(s) most likely to eliminate these factors are taken.

PAS uses different methods to conduct cause analysis. The most common approach is 5-Why, 4M, Fishbone Diagrams, or brainstorming may be appropriate depending on the situation. The method used is case specific and is documented in the CAPA record.

4.11.2 Effectiveness Review

Monitoring corrective actions for effectiveness is an essential part of the corrective action process. Effectiveness means the actions taken were appropriate and sustainable. Appropriate means the action(s) taken prevented recurrence of the problem since the time corrective action was taken and sustainable means the actions taken are still in place.

The data from CAPA records are used by PAS to identify opportunities for preventive action or to gain lessons learned when actions taken were not adequate to solve the problem. See Section 4.12 (Preventive Action) and 4.15 (Management Review) for more information.

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4.11.3 Additional Audits

When cause analysis and investigation of a problem casts doubt on compliance with PAS policies, procedures, or to regulatory requirements; a special audit of the area of activity may be performed as part of the corrective action process. These special audits are used to determine the scope of the problem and to provide information for the CAPA process. Additional full-scale audits are done when a grave issue or risk to the business is identified.

4.12 Preventive Action

Preventive action(s) are actions taken to eliminate the cause of a potential nonconformity before it happens.

Some examples of preventive action include, but are not limited to:

- Routine instrument maintenance (Preventive maintenance)
- Addition of Staff and Equipment
- Professional Development Activities
- Implementation of New Technology

PAS looks for opportunities for preventive action from a variety of sources including employee idea's, customer feedback, business partners input, trend analysis, business analytics, management reviews, proficiency testing results, and risk-benefit analysis.

PAS management evaluates the success of preventive actions taken in any given year during annual management review. See Section 4.15 for more information.

4.12.1 Change Management

Preventive actions may sometimes result in significant changes to processes and procedures used by PAS locations. PAS management evaluates the risks and benefits of change and includes in its implementation of change process, actions to minimize or eliminate any risk. The types of changes for which risk are considered and managed include infrastructure change, change in analytical service offerings, certification or accreditation status, instrumentation, LIMS changes, and changes in key personnel.

4.13 Control of Records

PAS records document activities and provide evidence of conformity to the requirements established in the QMS. These records may be hardcopy or electronic on any form of media.

4.13.1 General Requirements

4.13.1.1 Procedure

PAS requirements for control of records are specified in corporate policy ENV-POL-CORQ-0013 *Record Managemen*t.

The policy was established to assure quality and technical records are identified, retained, indexed, and filed to allow for retrieval during the entire retention time. During storage, records are kept secure and protected from deterioration. At the end

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of the retention time, the records are disposed of properly in order to maintain client confidentiality and to protect the interests of the company.

In general, PAS records fall into three categories: quality, technical, and administrative.

Examples of each are provided in the following table:

Record Type	Includes Records of:
Quality	Audits: Internal and External Certificates and Scopes of Accreditation Corrective & Preventive Action Management Review Data Investigations Method Validation Instrument Verification Training Records
Technical	Raw Data Logbooks Certificates of Traceability Analytical Record Test Reports & Project Information Technical Training Records & Demonstration of Capability
Administrative	Personnel Records Finance/Business

4.13.1.2 Record Legibility and Storage

Records are designed to clearly identify the information recorded. Manual entries are made in indelible ink; automated entries are in a typeface and of sufficient resolution to be read. The records identify personnel that performed the activity or entered the information. Records are archived and stored in a way that they are retrievable. Access to archived records is controlled and managed.

For records stored electronically, the capability to restore or retrieve the electronic record is maintained for the entire retention period. Hardcopy records are filed and stored in a suitable environment to protect from damage, deterioration, or loss. Hardcopy records may be scanned to PDF for retention. Scanned records must be checked against the hardcopy to verify the scan is complete and legible.

Administrative records are kept for a minimum of 5 years and technical and quality records are kept for 10 years unless otherwise specified by the client or regulatory program.

The date from which retention time is calculated depends on the record. In general, the retention time of technical records of original observation and measurement is calculated from the date the record is created. If the technical record is kept in a chronological logbook, the date of retention may be calculated from the date the logbook is archived. The retention time of test reports and project records, which are considered technical records, is calculated from the date the test report was issued.

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The retention time of quality records is usually calculated from the date the record is archived.

Refer to the record management policy and the location specific SOP for more information.

4.13.1.3 Security

PAS locations are secure facilities and access to records is restricted to authorized personnel.

4.13.1.4 Electronic Records

The data systems used to store electronic records is backed up in accordance with SOP ENV-SOP-WES2-0115, *Computer System Backup Control*. Access to archived records stored electronically is maintained by personnel responsible for management of the electronic system.

4.13.1.5 Electronic Signature Policy

Work done by PAS locations include activities that require the application of a signature. Some work products are in electronic format and signatures are applied electronically.

The Electronic Signatures in Global and National Commerce Act (E-Sign Act) clarifies that electronic signatures are legally valid and enforceable under United States law.

See guide ENV-GUI-CORQ-0007 *Electronic Signatures* for more information.

4.13.2 Technical Records

In addition to the requirements specified in subsections 4.13.1.1 through 4.13.1.5, the requirements in the following subsections also apply to technical records.

4.13.2.1 Description

Technical records are the accumulation of data and information generated from the analytical process. These records may include forms, worksheets, workbooks, checklists, notes, raw data, calibration records, final test reports, and project records. The accumulated record needs to provide adequate detail to historically reconstruct the process and identify the personnel that performed the tasks associated with a test result.

4.13.2.2 Real Time Recordkeeping

Personnel are instructed and expected to always record observations, data, and calculations at the time they are made. PAS managers are responsible for ensuring that data entries, whether made electronically or on hardcopy, are identifiable to the task.

4.13.2.3 Error Correction

Errors in records must never be erased, deleted, or made illegible. Use of correction fluid, such as white-out is prohibited. In hardcopy records, the error is corrected by a

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single strike through the original entry and the new entry recorded alongside or footnoted to allow for readability. Corrections are initialed and dated by the person making the correction. If the correction is not self-explanatory, a reason for the correction is recorded. Maintenance of proper practices for error correction is monitored through the tiered data review process described in Section 5.9.3.

For electronic records, equivalent measures of error correction or traceability of changes made is kept. For example, audit trails provide records of change.

4.14 Audits

Quality personnel, or their designees, perform internal systems and technical audits to assess implementation of the QMS and compliance to this manual, policy, and procedures that make up the QMS.

PAS locations are also audited by external parties such as regulatory agencies, customers, consultants, and non-government assessment bodies (NGAB).

Information from internal and external audits is used by local and corporate PAS management to address deficiencies and to identify opportunities to improve customer service and quality of work, including reliability and usability of data and test results.

Deficiencies, observations, and recommendations from audits are managed by the local QM using the CAPA process. See Section 4.11 for more information.

4.14.1 Internal Audit

Internal audits are conducted to ensure laboratory and business practices match what we say we do and what we say we do is compliant with the PAS QMS and relevant standards and requirements.

The internal audit program is managed by the local QM who prepares an audit plan at the beginning of each calendar year. The schedule is prepared to assure that all work areas are reviewed over the course of the year and test methods are audited every two years, unless a more frequent test method audit is required by program. Conformance to the schedule is monitored on a monthly basis.

PAS management is responsible for ensuring the audit schedule is maintained. PAS supervisors are expected to cooperate with the quality personnel to provide them with complete access to the work area, personnel, and records needed to conduct the audit.

Internal audits may be performed by non-quality personnel when the auditor is approved by the local QM. Non-quality personnel may not audit their own work activities unless it can be demonstrated that an effective and objective audit will be conducted. The person conducting the audit must be trained, qualified, and familiar enough with the objectives and policies of the PAS QMS and knowledgeable with process and test method SOPs related to the activities audited. The auditor should be trained in auditing practices in order to perform a thorough and effective evaluation.

Test method audits include reviews of test reports to verify the product is consistent with customer/project requirements, the work was conducted in accordance with policy and SOPs, the SOP complies with the cited reference method, test results are accurate, and of known and documented quality and properly qualified, when necessary.

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Special audits are performed as needed to follow up on a specific issue such as a client complaint, negative feedback, concerns of data integrity or ethics, or a problem identified through other audits. Special audits may be scheduled or unscheduled. Unscheduled internal audits are conducted whenever doubts are cast on compliance with regulatory requirements or its own policies and procedures. These unscheduled internal audits may be conducted at any time and may be performed without an announcement to the location or work area audited.

When observations and findings from any audit (internal or external) cast doubt on the validity of testing results, the location takes immediate action to investigate the problem and take corrective action. (Also see 4.11 and 4.16)

4.14.1.1 Corporate Compliance Audit

PAS locations may also be audited by Pace® corporate personnel at discretion. The purpose of the corporate compliance audit is to assess whether the location's practices, processes and procedures conform with the PAS QMS and to identify risk and opportunity.

4.15 Management Review

Local management conducts an annual business review of each location under their purview to assess performance and to establish goals, objectives, and action plans for the upcoming year.

The procedure used to conduct this review is specified in corporate SOP ENV-SOP-CORQ-0005 *Management Review*.

At a minimum, the following topics are reviewed and discussed during annual management review:

- Changes in internal and external issues relevant to the location;
- Fulfillment of objectives and initiatives;
- suitability of policies and procedures, including EHS and waste management;
- status of actions from previous performance reviews;
- The outcome of recent internal audits;
- Corrective and preventive actions;
- Assessments by external bodies;
- The results of interlaboratory comparisons or proficiency tests;
- Changes in the volume and type of the work;
- Customer and personnel feedback, including complaints;
- Effectiveness of improvements / preventive actions made since last review;
- Adequacy of resources;
- results of risk identification;

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- Proficiency testing performance and other measures related to the assurance of validity of test results; other relevant factors, such as QC trends and training status.

The discussion and results of this review are documented in a report prepared by local management. This report includes a determination of the effectiveness of the management system and its processes, goals, and objectives for improvements in the coming year with timelines and responsibilities, and any other need for change.

Goals and action items from annual management systems review are shared with local employees and with corporate management to highlight focus areas for improvement in addition to areas in which the location has excelled.

4.16 Data Integrity

PAS procedures for the investigation and response to events that may affect data integrity are described in the PAS corporate SOP ENV-SOP-CORQ-0017 *Data Impact Assessment*s.

Customers whose data are affected by the problem are notified in a timely manner, usually within 30 days after the impact of the problem is understood. Some accreditation programs also require notification to the accreditation body (AB) within a certain timeframe from date of discovery. PAS locations must follow any program or project specific requirements for notification, when applicable.

5.0 TECHNICAL REQUIREMENTS

5.1 General

Multiple factors contribute to the correctness and reliability of the technical work performed by PAS.

These factors fall under these broad categories:

- Human Performance
- Facility and Environmental Conditions
- Test Method Performance and Validation
- Measurement Traceability
- Handling of Samples

The impact of each of these factors varies based on the type of work performed. To minimize negative effects from each of these factors, PAS accounts for the contribution from each of these categories when developing test method and process (administrative) SOPs, evaluating personnel qualifications and competence, and in the selection of equipment and supplies used.

5.2 Personnel

5.2.1 Personnel Qualifications

PAS personnel are qualified and competent to perform the roles and responsibilities of their position based on education, experience, and training.

Qualifications, duties, responsibilities, and authorities of each position are specified in job descriptions maintained by corporate HR (See Section 5.2.4). These job descriptions provide the general basis for the selection of personnel for hire and are used by the location to

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communicate to personnel the duties, responsibilities, and authorities of their position. Qualification records may include but are not limited to diploma, transcripts, and curriculum vitae (CV).

The term "personnel" refers to individuals employed by PAS or a PAS subsidiary directly as full-time, part-time, or temporary, and individuals employed by PAS by contract, such as through an employment agency. The term "personnel" is used interchangeably with the term "employee" throughout this manual. For the purposes of this manual, these terms are equivalent.

5.2.1.1 Competence

Competence is the ability to apply a skill or series of skills to complete a task or series of tasks correctly within defined expectations.

PAS requirements for competence of personnel (education, qualification, work experience, technical skills, and responsibilities) are specified in job descriptions created by management and kept by human resources (HR). The job description provides the basis for the selection of personnel for each position.

An employee is considered competent when they have completed the required training specified in Section 5.2.2.

5.2.2 Required Training

Training requirements are outlined in Pace® policies COR-POL-0023 *Mandatory Training Policy,* COR-POL-0004 *Code of Ethics and Professional Conduct* and this Quality Manual.

5.2.2.1 Training Program

The PAS training program includes these elements:

- Scheduling
- Execution
- Documentation
- Evaluation of Effectiveness

Training is scheduled by corporate personnel, local quality personnel, and the employee's direct supervisor.

Training is delivered using various methods that incorporate techniques that appeal to the four main learning styles: visual, aural, linguistic, and kinesthetic. Delivery methods include, on-the-job (OJT), instructor-led, self-study, eLearning, and blended.

Training must be documented with a training record to prove training occurred. Required training (Section 5.2.2.1.1-5.2.2.1.5) must be complete before the employee is authorized to work independently on client samples. Complete means the employee has successfully completed training and evidence of training is filed in the employees training record maintained by the corporate training group, the corporate group that assigned training, such as IT, HR, or EHS, and/or and the local quality department.

The employee's direct supervisor is responsible for monitoring completion of the employee's required training program and for providing adequate time to the

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employee to complete training assignments. The supervisor and employee are jointly responsible to ensure the employee's training status and training records for all required training is current, complete, documented and the training records are accessible.

Until the required training is complete, the employee's direct supervisor is responsible and accountable for all work produced by the employee under their supervision.

Training status is tracked by local quality personnel and made readily available to all employees so they can access and monitor their status in real time.

The following subsections specify the required PAS training for new hires and requirements for on-going training.

5.2.2.1.1 New Hire Required Training

New hire training requirements apply to personnel new to Pace® and to existing employees starting in a new position or different work area.

Required new hire training includes each of the following topics:

- Ethics and Data Integrity (5.2.2.1.3)
- Quality Management System Policy and Procedure (5.2.2.1.4)
- Environmental Health and Safety (EHS)
- Demonstration of Capability (DOC) (5.2.2.1.5): Employees that prepare and test samples may also be required to successfully complete a DOC for the test method they are assigned before working independently on customer samples and to verify capability on a routine basis thereafter. Independent means without direct supervision of the work activity by the supervisor or a qualified trainer.

5.2.2.1.2 On-Going Required Training

All personnel receive on-going training on each of the following topics:

- Ethics and Data Integrity
- Quality Management System Policy and Procedure
- EHS

5.2.2.1.3 Ethics and Data Integrity

Ethics and data integrity training is provided to all new personnel upon hire and refresher training is provided to all employees minimally on an annual basis and more frequently as needed.

The training materials emphasize expectations, what to do, what not to do and the potential consequences that result from scientific misconduct and improper practices. Employees are informed that Pace® has a Zero Tolerance Policy for scientific misconduct, improper practices, and fraud. The employment of any employee found to have conducted, sanctioned,

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or instructed an employee to engage in these infractions will be terminated and the employee may be subject to debarment, and civil/criminal prosecution.

The following topics are covered:

- Pace® Policy for honest, transparent, and objective reporting of test results and data;
- Practices that are considered scientific misconduct, fraudulent and improper;
- How and when to report data integrity concerns or ethical violations;
- Record Keeping. The training emphasizes the importance of accurate, complete, and legible documentation of work activities;
- Procedures used by Pace® to prevent and monitor for infractions;
- Specific examples of breaches of ethical behavior such as improper data manipulations, adjustments of instrument time clocks, and inappropriate changes in concentrations of standards.

See COR-POL-0004 for more information.

5.2.2.1.4 Management System Documents Training

The primary documents that support the PAS QMS include the Quality Manual, policies, and SOPs.

These documents specify what we do, how, and why and are the documents all employees must abide. The employee's commitment to abide by these documents is acknowledged with a Read and Acknowledgement Attestation (R&A). PAS employees must have a R&A on record for the local Quality Manual, and the policies and SOPs relevant to their job responsibilities. The R&A acknowledges the employee has received, read, and understands the content of the management system document, the employee agrees to follow any instructions and requirements specified in the document when carrying out their work tasks; and the employee understands that unauthorized changes or departure from procedures and requirements specified in the documents not allowed unless authorized, documented and disclosed.

Other documents that support the management system include forms and worksheets that record original observations and/or calculate test results, guides, project-specific documents, or other reference materials. Employees are not required to have a R&A for these types of documents – how to use the document is learned on the job.

Employee understanding, application, and adherence to the content specified in management system documents is continuously reinforced by

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on-the-job (OJT) training and routine checks and balances, such as secondary data review and internal audits.

5.2.2.1.5 Demonstration of Capability (DOC) Requirements

A DOC is a procedure that establishes the ability of the analyst to perform a test procedure and obtain test results with acceptable accuracy and/or precision.

Newly hired employees that prepare or test samples are required to successfully complete an initial demonstration of capability (IDOC) for the test method before they are permitted to work independently on real world samples.

On-going demonstration of capability (ODOC) must be demonstrated on an annual basis thereafter.

The procedure for IDOC and ODOC can differ based on technology, analyte group and/or the regulatory or accreditation/certification program requirements that apply. In general, the options for ODOC are more flexible than for an IDOC on the premise that the analyst has routinely analyzed samples by the test method during the calendar year.

When more than 365 days (1 Year) has passed since the employee last analyzed real world samples by the test method; the employee must successfully redemonstrate capability with an IDOC to remain authorized to independently test real world samples.

A successfully complete DOC (IDOC or ODOC) is one where:

1) The DOC study data has been compiled, reviewed, and verified to meet acceptance criteria;

2) The DOC record has been approved by the department supervisor;

3) The DOC record has been verified complete by local quality personnel; and

4) The DOC record has been archived in the employee's training file for accessibility and reference.

If any of the above conditions is not met, the DOC is not complete.

Until the required training is complete, the employee's direct supervisor is responsible and accountable for all work produced by the employee under their supervision.

DOC procedures and acceptance criteria vary by program, technology, analyte group - therefore all options are not specified in this manual.

PAS locations must put in place a local DOC program appropriate for the testing services offered and that complies with relevant regulatory and/or accreditation/certification program requirements.

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For example – an acceptable DOC program for laboratories accredited to the TNI Standard might look like this:

- A DOC for chemistry test methods where spiking is an option is based on the employee's capability to achieve acceptable precision and accuracy for each analyte accredited from a series (usually 4) of replicates that were spiked at a specific concentration, and prepared and analyzed using the laboratory's test method SOPs.
- A DOC for a test method where spiking is not an option is based on an employee's capability to achieve comparable results to a peer.
- Specific instructions and acceptance criteria for DOCs for each test method may be specified in the test method SOP or a stand-alone SOP for DOC.

5.2.2.2 Effectiveness of Training

Training effectiveness is measured by the employee's demonstrated ability to comprehend the training material and apply knowledge and skills gained to their job task. These evaluations include but are not limited to:

- Testing of the employee's knowledge of the QMS, policies, and technical and administrative procedures through various mechanisms, such as quizzes, and interviews.
- Demonstrated ability to convey information correctly and factually in written and verbal communication.
- Demonstrated ability to follow instructions and adhere to policy and procedure.
- Demonstrated ability to make sound decisions based on guidance and information available and to defend decisions made.
- Demonstrated initiative to seek help or guidance when the employee is unsure of how to proceed.

5.2.2.3 Supplemental Learning

Supplemental learning objectives may be established for newly hired personnel to aid in their development of administrative and technical skills. These learning objectives and materials, referred to as Learning Plans (LP), are managed by the employee's direct supervisor.

Pace® also offers a wide variety of supplemental learning courses that are made available to all employees for professional development. The learning may be self-initiated based on an employee's interest or may be assigned to the employee at the discretion of management as professional development as part of an employee's annual goals.

Supplemental learning assignments are not prerequisites for competency (Section 5.2.1.1) and are not included in the required training program.

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5.2.3 Personnel Supervision

Every employee is assigned a direct supervisor, however named, who is responsible for their supervision.

General supervisory responsibilities may include but are not limited to:

- Hiring Employees
- Training Employees
- Performance Management
- Development, oversight, and execution of personnel training plans
- Monitoring personnel work product to assure the work is conducted in accordance with this quality manual, policies, SOPs, and other documents that support the QMS.

5.2.4 Job Descriptions

Job Descriptions that define the required education, qualifications, experience, skills, roles and responsibilities, and reporting relationships for each Pace® position are established by top management and kept by corporate HR. The job descriptions apply to employees who are directly employed by Pace®, part-time, temporary, technical, and administrative and by those that are under contract with Pace® through other means.

The job descriptions include the education, expertise, and experience required for the position and the responsibilities and duties, including any supervisory or managerial duties assigned to the position.

5.2.5 Authorization of Technical Personnel

Technical personnel are authorized to perform the technical aspects of their position after laboratory management has verified that the employee meets the qualifications for the position, and has successfully completed required training (Section 5.2.2.1). After initial authorization, technical personnel are expected to maintain a current and complete training record, demonstrate on-going capability at least annually for each test method performed, and produce reliable results through accurate analysis of certified reference materials, proficiency testing samples, and/or routine quality control samples in order to remain authorized to continue to perform their duties.

Records used to support authorization which may include but are not limited to, transcripts, resumes, training records, experience, training, and certificates are kept by the PAS location or business function to which the employee is assigned, if the employee is in a role that support more than one location or business unit.

5.3 Accommodations and Facilities

5.3.1 Facilities

PAS facilities are designed to support the activity performed. Access to PAS facilities is controlled by various measures, such as card access, locked doors, and staffed main entry.

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5.3.2 Environmental Conditions

Each location is equipped with energy sources, lighting, heating, and ventilation necessary to facilitate proper and safe performance of calibrations and tests. Local management ensures that housekeeping, electromagnetic interference, humidity, line voltage, temperature, sound, and vibration levels are appropriately controlled to ensure the integrity of specific measurement results and to prevent adverse effects on accuracy or increases in the uncertainty of each measurement.

Environmental conditions are monitored, controlled, and recorded as required by the relevant specifications, methods, and procedures. Operations are stopped if it is discovered that the environmental conditions would jeopardize the integrity of analytical results or other work products.

5.3.3 Separation of Incompatible Activities

The layout and infrastructure of each work area including air handling systems, power supplies, and gas supplies of each work area are designed for the type of analytical activity performed. Effective separation between incompatible work activities is maintained. For example, sample storage, preparation, and chemical handling for volatile organic analysis (VOA) is kept separate from semi-volatile organic analysis (SVOA).

Samples known or suspected to contain high concentration of analytes are separated from other samples to avoid the possibility for cross-contamination. If contamination is found, the source of contamination is investigated and resolved.

5.3.4 Security

Security is maintained by controlled access to the building and by surveillance of work areas by authorized personnel. Access to each area may be controlled depending on the required personnel, the sensitivity of the operations performed, and potential safety concerns.

5.3.5 Good Housekeeping

Local management must maintain good housekeeping practices in their facilities and maintain a standard of cleanliness necessary for analytical integrity and personnel health and safety.

5.4 Test Methods

5.4.1 General Requirements

PAS locations must use test methods and procedures that are appropriate for the scope of analytical test services the laboratory offers.

Instructions on the use and operation of equipment and sample handling, preparation, and analysis of samples must be specified in SOPs. The instructions in SOPs may be supplemented with other documents including, but not limited to, standard work instructions (SWI), manuals, guides, project documents, and other reference materials.

5.4.2 Method Selection

The test methods offered by PAS are primarily industry accepted published reference test methods. Each PAS laboratory bases its test method SOP on the latest version of the reference method unless regulatory requirements specify an earlier version must be used.

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The laboratory confirms that it can perform the test method and achieve desired outcome before analyzing samples following method validation protocols specified in Section 5.4.5.

The test methods used by the laboratory to test real world samples must meet the needs of the customer, be appropriate for the intended use of the data, and must conform with applicable federal, statutory, or program requirements.

When a customer does not specify the test method(s) to be used, the laboratory may suggest test methods that are appropriate for the intended use of the data and the type of samples to be tested. The laboratory will also inform customers when test methods requested are considered inappropriate for their purpose and/or out of date. This discourse takes place during review of analytical service requests (See Section 4.4).

5.4.3 Laboratory Developed Methods

A laboratory developed method is a test method developed wholly by PAS or a modification to a published method that is substantially modified from the specifications and procedures of a published test method.

Laboratory developed methods must be validated prior to use (see section 5.4.5) and the procedure documented in a test method SOP.

The requirements for non-standard methods (Section 5.4.4) also apply to PAS developed methods.

5.4.4 Non-standard Methods

A non-standard method is a testing protocol that is not published or approved for use by conventional industry standards.

Use of a non-standard method for testing must be agreed upon with the customer. The agreement, which is retained by the laboratory in the project record, must include the specifications of the client's requirements, the purpose of testing, and their authorization for use of the non-standard method.

5.4.5 Method Validation

The laboratory confirms that it can successfully perform the test method before analyzing samples through a process called method validation. Method validation is partially or fully repeated when there is a change in the published reference method or a change in the way the laboratory performs the test method that may affect selectivity, sensitivity, accuracy, precision, or general performance of the method.

5.4.5.1 Validation Description

Validation is the process of confirmation and the provision of objective evidence that the stated requirements for a specific method/procedure are fulfilled.

The laboratory's requirements and procedures for method validation are outlined in SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification.*

5.4.5.2 Validation Summary

All test methods offered by the laboratory are validated before use to confirm the procedure works as expected and the data and test results are accurate and precise.

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The record of validation is retained and kept in accordance with method validation SOP and PAS corporate policy ENV-CORQ-POL-0013 *Record Management*.

5.4.5.3 Validation of Customer Need

The laboratory's validation process includes measures to assess accuracy, precision, sensitivity, selectivity, linearity, repeatability, reproducibility, robustness, and cross-sensitivity of the laboratory's procedure against general customer expectations for data usability.

The following subsections describe some of these concepts in relation to organic and inorganic chemistry (chemical testing). These descriptions and the requirements specified are pertinent to chemical testing and may not apply in the same context to other testing services offered by PAS such as radiochemistry, whole effluent toxicity (WET), asbestos, and microbiology. For more information about validation measures for these testing services, refer to the laboratory's test method SOPs.

5.4.5.3.1 Accuracy

Accuracy is the degree to which the result of a measurement, calculation, or specification conforms to the correct value or a standard. When the result is within a range from the known value (control limit); the result is considered accurate.

5.4.5.3.2 Precision

Precision refers to the closeness of two or more measurements to each other. It is measured by calculating the relative percent difference (RPD) or relative standard deviation (RSD) of results from separate analysis of the same sample. Precision provides information about repeatability, reproducibility, and robustness of the laboratory's procedure.

5.4.5.3.3 Sensitivity

Sensitivity refers to the smallest amount of substance in a sample that can accurately be measured.

In chemical testing, this threshold is commonly known as the detection limit (DL) or method detection limit (MDL).

The DL is defined as the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from method blank results.

The DL is either a statistically derived value obtained by multiplying the standard deviation from a series of spiked replicates by the student-T value that correlates to the number of replicates analyzed or it is set to the concentration greater than found in a population of method blanks.

Detections of target analytes below the DL are not reported – unless specified by regulatory program protocol, because presence of target analytes at this level cannot be differentiated from noise.

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Limits of Quantitation represent the threshold of the laboratory's procedure for quantitative sensitivity.

- The LOQ is the minimum level, concentration, or quantity of a target analyte that can be reported with a specified degree of confidence.
- The LLOQ is simply the concentration of the lowest calibration standard included in the calibration curve.

Although the LOQ and LLOQ may be and often are the same value; the definitions are not the same.

The LOQ is static – it is set when the DL is determined. The LOQ value must be routinely verified to confirm the laboratory can detect the concentration within a range of accuracy.

The LLOQ fluctuates based on the series of standards included in each calibration curve – the LLOQ is always the concentration of the lowest calibration standard included in the curve fit and for which samples are quantitated against. The LLOQ is not the concentration of the lowest standard analyzed, if the lowest concentration standard is not included in the curve, it is not relevant to sample quantitation. Unless otherwise specified by test method, regulatory program or project-specific protocol, accuracy at the LLOQ does not need to be verified independent of the evaluation of relative error or relative standard error.

Target analytes detected at concentrations between the DL and LOQ are known as estimated values – they are within the range of qualitative identification and outside the range of quantitative accuracy. If reported, the results must be qualified to indicate the test result is estimated.

The meaning of Limit of Detection (LOD) varies based on the context by which the term is used. Outside of the DoD program, LOD is another term used to refer to the DL or MDL.

PAS laboratories that are accredited for DoD, must also establish an LOD. The DoD defines the LOD as the smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence. This definition is different from the definition of DL - the DL represents the theoretical value at which a detection is distinguishable from a blank.

For DoD, the LOD is a value between the DL and the LOQ. Specifically, it must be 2-4X greater than the DL but less than the LLOQ or LOQ. The value of the LOD set by the laboratory must meet all criteria for qualitative identification and quantitative accuracy based on the signal to noise ratio.

For DoD/DOE project work, target analytes that are not detected at the DL are reported as undetected at the LOD and results between the DL and the LOQ are reported as estimated values.

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The reporting limit (RL) is the value to which test results are reported as detected or not detected. The RL may be project-defined based on project data quality objectives (DQO) and it may be above or below the LOQ or LLOQ. In the absence of project specific requirements, the RL is usually the same value as the LLOQ. The RL is also called the practical quantitation limit (PQL) – but this can be misleading if the RL is less than the LLOQ or LOQ.

In general, the RL should always be greater than or equal to the value that establishes the threshold for quantitative sensitivity, but it is common for clients to request laboratories to report undetected target analytes at the DL value. This instruction has evolved as an acceptable reporting practice; however, data users should understand the DL is a statistically derived value that does not represent the limit of quantitation.

Unless otherwise specified by regulatory program or test method, detection, quantitation, and reporting limits are adjusted for the amount of sample used (preparation factor) and the dilution factor and are adjusted for percent moisture when samples are reported on a dry weight basis.

To ensure detection and quantitation limits are sound and defensible, the procedures that PAS laboratories use to establish detection, quantitation and reporting limits for chemical testing must comply with the current version of each of the following documents:

- EPA document EPA-821-R-16-006 Definition and Procedure for the Determination of the Method Detection Limit;
- 2016 TNI Standard V1M4; and
- TNI GUID-3-109-Rev. 0, V1M4 2016 Standard Update Guidance on Detection and Quantitation.

5.4.5.3.4 Selectivity

Selectivity refers to the ability of a method to correctly determine an analyte without interference from other compounds.

5.4.5.3.5 Linearity

Linearity is a mathematical concept applied to calibration models that employ multiple points to establish a calibration range used for quantitative analysis. Linearity is measured differently based on the calibration model. In general, if linearity is demonstrated then the slope of the response of standards are sufficiently close to one another. The accuracy of the linear regression and non-linear curves is verified by checking percent error or relative standard error (RSE), which is the process of refitting calibration data back to the model to determine if the results are accurate. For linear curves that use average calibration or response factor, error is measured by relative standard difference (RSD).

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Linearity also establishes the range of quantitation for the test method used which directly impacts the sensitivity of the test method and uncertainty in measurement results. As previously noted, the LLOQ establishes the lower limit of quantitation. The upper range of linearity establishes the upper limit of quantitation. For most technologies, results outside of this range are considered qualitative values. However, some inorganic test methods allow the linear range to be extended above the upper limit of quantitation when accuracy at this level is verified.

Linearity can also be used to establish repeatability, reproducibility, and robustness of the laboratory's test method. When linearity is demonstrated using a specific calibration model during method validation, then use of this same calibration model to achieve linearity on a day-to-day basis confirms the laboratory's method is repeatable, reproducible, and robust.

5.4.6 Measurement Uncertainty

PAS provides an estimate of uncertainty with analytical results on request, or when required. For example, for radiochemistry testing, uncertainty is always reported with the test result.

For chemistry the uncertainty of the test method is embedded in the control limits used to assess performance of quality control (QC) samples. Language in ISO/IEC 17025 clarifies that measurement uncertainty is satisfied when a well-recognized test method specifies limits that account for source of bias, the laboratory has satisfied the requirements on analytical uncertainty by following the test method and reporting instructions. See Section 5.9.1.1.9 for more information.

5.4.7 Control of Data

PAS has policies and processes in place to assure that reported data is free from calculation and transcription errors, manual changes to data are thoroughly reviewed for need and correctness, and software systems used to generate or store data is secure.

5.4.7.1 Calculations, Data Transfer, Reduction and Review

Data reduction is performed using commercially available data processing/reduction software, the LIMS (Laboratory Information Management System) and/or programs and worksheets created by PAS.

PAS does not permit manual calculation of results – that is the calculation of a test result by pen and paper or calculator. When there is no commercial software or LIMS option, PAS laboratories must create a worksheet from a spreadsheet program, such as Excel, to calculate results based on raw inputs and original observations. Spreadsheets for these purposes must be validated and controlled in accordance with policy ENV-CORQ-POL-0015 *Spreadsheet Validation.*

5.4.7.1.1 Manual Integration

The PAS policy and procedures for manual integration are specified in PAS SOP ENV-SOP-CORQ-0006 *Manual Integration.*

This SOP includes the conditions under which manual integration is allowed and the requirements for documentation.

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Required documentation of manual integration minimally includes:

- complete audit trail to permit reconstruction of before and after results;
- identification of the analyst that performed the integration and the reason the integration was performed; and
- identification of the individual(s) that reviewed the integration and verified the integration was done and documented in compliance with the SOP.

5.4.7.2 Use of Computers and Automated Acquisition

Whenever possible, PAS uses software and automation for the acquisition, processing, recording, reporting, storage, and/or retrieval of data.

In-house developed software is validated by Pace® corporate IT for adequacy before release for routine use. Commercial off the shelf software is considered sufficiently validated when the location follows the manufacturer or vendor's manual for set-up and use. Records of validation are kept by the corporate information technology (IT) group or by the group that performed the validation.

The PAS process for the protection of data stored in electronic systems includes:

- Individual usernames and passwords for Laboratory Information Management Systems (LIMS) and auxiliary systems used to store or process data.
- Employee Training in Computer Security Awareness
- Validation of spreadsheets used for calculations to verify formulas and logic yield correct results and protection of these cells to prevent unauthorized change.
- Operating system and file access safeguards
- Protection from Computer Viruses
- Regular system backup; and testing of retrieved data
- Verification the software application works as expected and is adequate for use and fulfills compliance requirements, such as the need to record date/time of data generation.
- Change control to assure requests for changes are reviewed and approved by management before the change is made.
- Communication channels to assure all staff are aware of changes made.
- Version Control and maintenance of historical records.

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5.5 Calibration Requirements

5.5.1 Availability of Equipment

Local management ensures the laboratory is furnished with all equipment and instrumentation necessary to correctly perform the tests offered in compliance with the specifications of the test method and to achieve the accuracy and sensitivity required.

5.5.2 Calibration

Equipment and instrumentation are checked prior to use to verify it performs within tolerance for its intended application.

5.5.2.1 Support Equipment

Support equipment is verified to be in proper working order, and to meet the tolerance specifications for the purpose(s) for which it will be used prior to placement in service. Thereafter, periodic checks are performed to verify on-going performance per a schedule maintained by local quality personnel. Support equipment that does not meet specifications is removed from service and either repaired or replaced. Records of repair and maintenance activities are maintained.

Procedures used to conduct and record these checks are outlined in SOP ENV-SOP-WES2-0036, *Balance Calibration Verification*, ENV-SOP-WES2-0039 *Thermometer Calibration*, ENV-SOP-WES2-0123 *Autopipet and Wet Chem Titrator Calibration Verification*.

5.5.2.2 Analytical Instruments

Analytical instruments are checked prior to placement in service in accordance with SOP ENV-SOP-CORQ-0011 *Method Validation and Instrument Verification*. After the initial service date, the calibration of instruments and verification of calibration is performed in accordance with local test method SOPs.

The calibration procedures in the test method SOPs must comply with the requirements for acceptable calibration practices outlined in corporate policy ENV-POL-CORQ-0005 *Acceptable Calibration Practices*, the reference methods, and any applicable regulatory or program requirements.

5.5.3 Equipment Use and Operation

Up-to-date instructions and procedures for the use and maintenance of analytical equipment are included in SOPs and/or supplemental documents such as standard work instructions (SWI) or instrument manuals which are made readily accessible in the work area to all laboratory personnel.

5.5.4 Equipment Identification

Each piece of equipment is uniquely identified by serial number or any other unique ID system. The identifier is included in the equipment list maintained by the quality department and may not be reused or used interchangeably. New equipment and replacement equipment are assigned a new unique ID.

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5.5.5 Equipment Lists and Records

5.5.5.1 Equipment List

Each PAS location maintains a list of equipment that includes information about the equipment including a description, manufacturer, serial number, date placed in service, condition when received, identity, and the work area where the equipment is used. The equipment list(s) for each location covered by this manual is provided in Appendix E.

5.5.5.2 Equipment Records

In addition to the equipment list, other equipment records maintained by PAS include:

- Verification that equipment conforms with specifications.
- Calibration records including dates, results, acceptance criteria, and next calibration dates.
- Maintenance plan and records.
- Records of damage, malfunction, or repair.

The laboratory follows an equipment maintenance program designed to optimize performance and to prevent instrument failure which is described in the analytical method SOPs.

The maintenance program includes routine maintenance activities which are performed as recommended by the manufacturer at the frequency recommended and non-routine maintenance, which is performed to resolve a specific problem such as degradation of peak resolution, shift in calibration relationship, loss of sensitivity, or repeat failure of instrument performance checks and quality control samples.

Maintenance is performed by PAS personnel or by outside service providers.

All maintenance activities performed by PAS personnel are recorded by the individual(s) that performed the activity at the time the maintenance was performed in an instrument maintenance log.

The maintenance record minimally includes the date of maintenance, the initials of the person(s) performing maintenance, a description of the activity performed, why (when the maintenance is non-routine), and the return to analytical control. When maintenance is performed by an external vendor, the service record must be maintained and accessible for easy retrieval. The location must provide personnel with unrestricted access to instrument maintenance logs in order to promote good instrument maintenance and recordkeeping practices.

If an instrument must be moved, the location will use safe practices for handling and transport to minimize damage and contamination.

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5.5.6 Out of Service Protocol

Equipment that has been subjected to overloading, mishandling, gives suspect results, has been shown to be defective, or is performing outside of specified limits is taken out of service and either removed from the work area or labeled to prevent accidental use until it has been repaired and verified to perform correctly.

When analytical equipment is taken out of service because it no longer meets tolerance specifications, the potential effect the nonconformity may have had on previously reported analytical results is evaluated.

5.5.7 Calibration Status

Support equipment is labeled to identify its calibration status.

The calibration status of analytical instruments is identified in the instrument run log, in the data processing software or other systems the laboratory may use for processing of data. Analysts verify on-going acceptability of calibration status prior to use and with instrument performance check standards. These procedures are described in test method SOPs.

5.5.8 Returned Equipment Checks

When equipment or an instrument is sent out for service, the function and calibration status of the equipment must be rechecked and shown to be satisfactory before the equipment is returned to service. This requirement must be included in the laboratory's protocols for servicing equipment.

5.5.9 Intermediate Equipment Checks

PAS performs intermediate checks on equipment to verify the on-going calibration status. For example, most test methods require some form of continuing calibration verification check, and these procedures are included in the test method SOP. Periodic checks of support equipment are also performed; see SOP ENV-SOP-WES2-0036 *Balance Calibration Verification*, ENV-SOP-WES2-0039 *Thermometer Calibration*, ENV-SOP-WES2-0039 Autopipet and *Wet Chem Titrator Calibration Verification* for more information.

5.5.10 Safeguarding Equipment Integrity

Equipment integrity in maintained by:

- Following manufacturer's specification for instrument use so that settings do not exceed manufacturer's recommendation or stress the performance of the equipment;
- Established maintenance programs;
- Transparent maintenance records and unrestricted access to maintenance logs;
- Validation and approval of software before use;
- Audits to confirm instrument settings are consistent with SOPs; and
- On-the-job training for safe and proper use of laboratory equipment.

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5.6 Measurement Traceability

5.6.1 General

Measurement traceability is best described as the way to ensure measurements take into consideration all uncertainties and are representative of the most accurate measurement captured. Traceability is accomplished with the recording of the lot numbers of materials and equipment used in the testing process and by purchasing materials traceable to the international standards of units (SI), International Bureau of Weights and Measures (BIPM) and/or National Measures Institute NMI).

When traceability to SI units, BIPM, or NMI cannot be achieved, traceability is accomplished with the use of reference standards and equipment obtained from competent suppliers that provide calibration certificates and/or certificates of analysis (COA).

5.6.2 Equipment Correction Factors

Measurements are adjusted by the correction factor when the equipment used for the measurement has a correction factor.

5.6.2.1 Requirements for Calibration Laboratories

PAS does not offer calibration services, therefore, the ISO/IEC 17025 for calibration laboratories do not apply.

5.6.2.2 Requirements for Testing Laboratories

Requirements to verify equipment is calibrated prior to being put into service and to maintain measurement traceability are described in sections 5.5.2 and 5.6, respectively.

5.6.3 Reference Standards and Reference Materials

5.6.3.1 Reference Standards

The laboratory uses measurement reference standards to verify adequacy of working weights and thermometers. The working weights are the weight(s) used for daily balance calibration checks and the working thermometers are used for daily temperature measurements.

Working weights and thermometers are periodically checked to verify on-going adequacy for use between the calibrations performed by an external calibration laboratory using reference standards traceable to SI or a national standard and that are used solely for verification purposes.

For example:

- An acceptable reference standard to verify working thermometers is a NIST Certified thermometer or a NIST Traceable thermometer that is not used for any other purpose other than to check the adequacy of the working thermometer.
- An acceptable reference standard for the working weights is a set of Class S weights that is not used for any other purpose than to verify the weights used daily.

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The working weights are checked against the reference standard annually and recertified by an ISO accredited calibration body every 5 years. "Annually" means within thirteen (13) months from the date of the last check.

Working thermometers are checked against the reference thermometer prior to placement in service to establish a correction factor (CF) and then re-checked annually (± 13 months from date of last check) or if electronic, every three (3) months (± 100 days from date of last check).

Exceptions to the 3-month recheck for battery operated sensors are allowed when the sensor is embedded in a unit and the manufacturer/vendor has evidence to show that the accuracy of the sensor is not affected by battery life.

Liquid in Glass NIST Certified reference thermometers must be recertified by an ISO/IEC accredited calibration laboratory every 5 years. If the reference thermometer is NIST Traceable or is a digital NIST Certified thermometer, the reference thermometer must be recertified annually by an ISO/IEC 17025 accredited calibration laboratory or service provider that provides traceability to a national standard.

5.6.3.2 Reference Materials

PAS purchases chemical reference materials (also known as stock standards) from vendors that are accredited to ISO 17034 or Guide 34. Purchased reference materials must be received with a Certificate of Analysis (COA) where available. If a reference material cannot be purchased with a COA, it must be verified by analysis and comparison to a certified reference material and/or there must be a demonstration of capability for characterization. COA are reviewed for adequacy and retained by the laboratory for future reference.

All prepared standards, reference materials, and reagents are verified to meet the requirements of the test method through routine analyses of quality control samples.

The laboratory procedure for traceability and use of these materials is provided in SOP ENV-SOP-WES2-0038 *Reagent Solvent and Standard Control.*

This SOP must minimally include each of the following requirements:

- Procedures for receipt and tracking of reference and working standards to ensure the record includes material name, name of the material, lot number, receipt date, and manufacturer's expiration date.

- Storage conditions and the requirement that reference standards are stored separately from samples, extracts, and digestates.

- Requirements to assure that preparation of intermediate or working solutions are recorded and assigned a unique identification number for tracking. Records of preparation include the lot number of the stock standard(s) used, the type and lot number of the solvent, the formulation, date, new expiration date, and the preparer's initials. The lot number of working standards is recorded in the analytical record to provide traceability to the standard preparation record. The

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preparation record provides traceability to the COA, which is traceable to SI or the national measurement standard.

- A requirement that the expiration dates of prepared standards may not exceed the expiration date of the parent standard. Standards, reference materials, and reagents are not used after their expiration dates unless it is not possible to procure a new standard and the reliability of the expired material is verified and documented by the location using a procedure approved by corporate quality personnel. Otherwise, the expired material is promptly removed from the work area or clearly labeled as acceptable for qualitative/troubleshooting purposes only.

- The second source materials used for verification of instrument calibration are obtained from a different manufacturer or may be a different lot from the same manufacturer, when only one vendor or manufacturer provides the standard.

- Procedures to check reference materials for degradation and replacement of material if degradation or evaporation is suspected.

- Procedures for labeling. At a minimum, the container must identify the material, the ID of the material and the expiration date. Original containers should also be labeled with date opened.

5.6.3.3 Intermediate Checks

Checks to confirm the acceptability of reference standards and materials is specified in test method SOPs and are appropriate for the purpose for which the standard is used. For example, a second source standard analyzed after an initial calibration may help the analyst identify if degradation of the standards used for calibration has occurred.

5.6.3.4 Transport and Storage

PAS handles, transports and stores reference standards in a manner that protects the integrity of the materials. The integrity of the reference standard is protected by segregation from incompatible materials and/or minimizing exposure to degrading environments or materials. As previously stated, standards and reference materials are stored separately from samples, extracts, and digestates to prevent contamination. All standards are stored according to the manufacturer's recommended conditions. Temperatures colder than the manufacturer's recommendation are acceptable if it does not compromise the integrity of the material (e.g., remains in liquid state and does not freeze solid).

Specific requirements are provided in test method SOPs.

5.7 Sampling

Sampling refers to both the field collection of samples and to subsamples taken by the laboratory for analysis.

Subsampling procedures are included in each test method SOP or a stand-alone SOP to assure the aliquot used for testing is representative of the field collected sample.

The requirements in the following subsections apply when sampling is performed by PAS.

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5.7.1 Sampling Plans and SOPs

When PAS performs field collection of samples, the sampling is conducted in accordance with a project sampling plan and sampling SOPs to ensure representative samples are collected.

5.7.2 Customer Requested Deviations

When the customer requests deviations, additions, or exclusions from the project sampling plan and/or the SOP, the laboratory records the client's change request in detail with the sampling record, communicates the change to sampling personnel, and includes this information in the final test report.

5.7.3 Recordkeeping

PAS assures the sampling record includes the sampling procedure used, any deviations from the procedure, the date and time of sampling, the identification of the sampler, environmental conditions (if relevant), and the sampling location.

5.8 Sample Management & Handling

5.8.1 Procedures

The procedures for sample management and handling are outlined in SOP ENV-SOP-WES2-0003 *Sample Receipt and Login* and SOP ENV-SOP-WES2-0002 *Sample Custody and Tracking*. The procedures in the local SOP must meet the requirements specified in this quality manual to guarantee the integrity of the sample from receipt to disposal.

5.8.1.1 Chain of Custody

All samples received by PAS must be accompanied with a Chain of Custody (COC). The COC is the legal record that documents the collection of samples and the transfer of samples to the laboratory. The COC provides PAS with information about the samples collected and tests requested.

The COC is completed by the sample collector and must minimally include the following information:

- Client name, address, phone number;
- Project Reference;
- Client Sample Identification (Client ID);
- Date, Time, and Location of Sampling;
- Sampler's Name or Initials;
- Matrix;
- Type of container, and total number collected;
- Preservative;
- Analyses Requested;
- Mode of collection;

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- Any special instructions; and
- The date and time and signature of sample transfer from collection to PAS. When samples are shipped to the laboratory by a 3rd party such as UPS or Fed EX, the COC is placed inside the sealed shipping container at time of collection and the container is not opened until it is received by PAS. Thus, the intermediate courier does not sign the COC. The shipping manifest and/or air bill is the record of possession of samples during transport to the laboratory. The shipping manifest and/or air bill is part of the COC record and must be retained and provided in the test report with the COC record, when required. (See Section 5.10.3).

A complete and legible COC is required. When the COC is incomplete, illegible, or cannot be understood, the client is contacted for resolution.

Hand entries on the COC must be made in indelible ink and errors corrected by drawing a single line through the original entry, so the original entry is not obscured and entering the correct information with the date the change was made and the initials of the person making the change.

5.8.1.2 Legal Chain of Custody

Legal chain of custody (Legal COC) is an industry defined protocol that is sometimes used for evidentiary or legal purposes that establishes an intact, continuous record of the physical possession, storage, and disposal of samples, including sample aliquots and prepared samples.

The legal COC record accounts for all time periods associated with the samples and identifies all individuals who physically handled them from the point established by legal authority, which is usually at the time the sample containers are provided for sample collection or at the time of sample collection.

A sample is in someone's legal custody if:

- It is in one's physical possession;
- It is in one's view after being in one's physical possession;
- It has been in one's physical possession and then locked or sealed so that no one can tamper with it; and/or
- It is kept in a secure area, restricted to authorized personnel only.

Legal COC is not a common request, but if it is requested and the laboratory agrees to the project specification; the laboratory must maintain a procedure or process to guarantee all aspects of the protocol are met.

5.8.2 Unique Identification

Each sample received by PAS is assigned a unique identification number (Lab ID) after the sample has been accepted by PAS in accordance with the PAS sample acceptance policy (See 5.8.3). The Lab ID is affixed to the sample container using a durable label.

The lab ID is linked to the field ID (client ID) in the sample receipt and log-in record so that both IDs are traceable to testing activities performed.

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5.8.3 Sample Receipt Checks and Sample Acceptance Policy

The condition and integrity of samples is checked on receipt and the samples received are verified to match the COC. Any problem with the samples or discrepancy between the COC is recorded. If the problem impacts the suitability of the sample for analysis or if the CoC is incomplete, the client is notified for resolution and decisions and instructions from the client regarding the samples are maintained in the project record.

5.8.3.1 Sample Receipt Checks

The following checks are performed:

- The COC is complete and legible;
- Sample container labels include the client sample ID, the date and time of collection, and the preservative used;
- The container type and preservative are appropriate for the test requested;
- The required volume is received for each test requested;
- There is no evidence of damage or tampering;
- There is no headspace in VOA vials greater than 5-6 mm (the size of a pea);
- Thermal Preservation, when required. When thermal preservation is required, temperature on receipt is typically considered acceptable if the temperature is within 0-6°C and the samples are not frozen solid unless otherwise specified by federal, statutory, program or test method. If samples are received on the same day of collection, there must be evidence that the chilling process has begun, such as arrival of the samples on ice.
- Chemical Preservation, as required.
- Holding Time. When the holding time for the test method is longer than 15 minutes from time of collection and samples are received out of hold, the client is notified of the situation so they can make a decision to proceed or cancel analysis. Tests with a holding time of 15 minutes or less from time of collection are those where the method specifies the holding time as "immediate" are excluded from the notification requirement because the customer is aware that the holding time cannot be met unless the test is performed in the field (in situ).

5.8.3.2 Sample Acceptance Policy

PAS maintains a sample acceptance policy to clearly establish the circumstances in which samples are accepted.

When these conditions are not met, the exception must be documented, and the client notified. Decisions to accept or reject the samples are documented and when required, test results are qualified in the test report.

Criteria for Sample Acceptance:

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- All samples received must be listed on the COC and information provided must include the client ID of the sample, date and time of collection, collector's name, preservation type and type of sample;
- The sample containers must be properly labeled to uniquely identify the sample; and the label must be both durable and legible;
- The sample container must be appropriate for the tests requested;
- Samples must be received within holding time. Exceptions are made for samples where the test method specifies a holding time of "immediate or immediately" – which is translated to within 15 minutes from sample collection. For these test methods, samples are always received out of hold and/or there is no expectation that the laboratory perform the test within the 15-minute holding time.
- Sufficient sample volume must be provided to perform the test method per the laboratory's SOP. If the test is performed for regulatory compliance and the method requested is mandated; the volume provided must be adequate for the laboratory to perform the test per the test method without modification.
- Be received within appropriate temperature ranges unless program requirements or customer contractual obligations mandate otherwise.
- Be free of evidence of tampering or signs of contamination; and
- Meet applicable requirements for thermal and chemical preservation.

Additional conditions may also apply depending on the services offered. For example, some PAS locations will not accept samples known to be radioactive.

5.8.4 Sample Control and Tracking

Samples received by PAS are tracked using the laboratory's Laboratory Information Management System (LIMS).

After the sample receipt process is complete, samples are logged into the LIMS and a Lab ID is assigned and affixed to each container with a durable label. The Lab ID is used to track the samples through the testing process through test report generation and sample disposal.

Minimally, the following information is included in the log-in for each sample:

- Client Name and Contact Information;
- The laboratory ID linked to the client ID;
- Date and time of sample collection;
- Date and time of sample receipt;
- Matrix; and
- Tests Requested.

After log-in is complete, the log-in record must be independently reviewed by the PAS Project Manager (PM) responsible for the work order to verify the log-in is complete and correct per

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the testing agreement with the customer and the chain-of-custody so that any log-in errors or questions about the samples received are corrected and resolved before work begins.

Documentation of the log-in record, called a Sample Acknowledgement Form (SRF) is provided to customers per their request. The SRF provides information necessary for the customer to verify that there are no errors with the information provided to PAS with the sample shipment.

As previously stated in this manual, the customer must be notified of any problems or discrepancies between the information expected and information received and/or nonconformances observed during the sample receipt and log-in process. Authorization to proceed with testing and/or instructions to correct the problem must be retained for historical recordkeeping purposes. How the notification requirement is achieved must be included in a laboratory SOP.

5.8.5 Sample Storage, Handling, and Disposal

5.8.5.1 Sample Storage

The samples are stored according to the specification in the test method and any appliable program and regulatory requirements. Samples are stored separately from standards, reagents, or other potential sources of contamination and in a manner that prevents cross contamination. All sample fractions, extracts, leachates, and other sample preparation products are stored in the same manner as actual samples or as specified by the analytical method.

Refrigerated storage areas are maintained at 4°C ±2 (but not frozen) and freezer storage areas are maintained at <-10°C, unless otherwise required per method or regulatory program. The temperature of each storage area is checked and documented at least once for each day of use. If the temperature falls outside the acceptable limits, corrective actions are taken and documented.

Samples are placed in the storage location immediately after sample receipt and log-in procedures are completed. All sample storage areas have limited access. Samples are removed from storage areas by designated personnel and returned to the storage areas as soon as possible after the required sample quantity has been taken.

5.8.5.2 Sample Retention and Disposal

The procedures used by the location for sample retention and disposal are detailed in SOP ENV-SOP-WES2-0002 *Sample Custody and Tracking* and ENV-SOP-MANS-0077 *Hazardous Waste Management and Sample Disposal.*

In general, PAS retains unused sample volume and prepared samples such as extracts, digestates, distillates and leachates (samples) per Pace® Terms & Conditions.

After analysis, samples may be stored at ambient temperature until time of sample disposal, unless otherwise specified by regulation or agreed upon contract specifications.

After the retention time has expired non-hazardous samples are properly disposed. Hazardous samples are returned to the customer, unless other arrangements for disposal have been made with the laboratory.

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5.9 Assuring the Quality of Test Results

5.9.1 Quality Control (QC) Procedures

PAS monitors the validity and reliability of test results using quality control (QC) samples that are prepared and analyzed concurrently with and in the same manner as field samples. QC results are always associated to and reported with the field samples they were prepared and analyzed with.

When the results are not within acceptance criteria correction and corrective action(s) are taken. These actions may include retesting or qualification of test results and/or disclosure of the situation in the test report to alert the data user of the situation.

Other control measures to maintain quality of test results include the use of certified reference materials (see 5.6.3), participation in interlaboratory proficiency testing (see 5.9.1.2), verification that formulae used for reduction of data and calculation of results is accurate (see 5.9.3), on-going monitoring of environmental conditions that could impact test results (see 5.3.2), and evaluation and verification of method selectivity and sensitivity (see 5.4.5).

QC results are also used for trend analysis and to establish acceptance criteria when no method or regulatory criteria exist. (See 5.9.1.1.9).

5.9.1.1 Essential QC

QC protocol is technology and test method specific and is specified in test method SOPs. The SOP includes the type of QC required, frequency for analysis, acceptance criteria, recommended corrective actions, and procedures for reporting test results associated with QC exceptions.

PAS ensures the QC requirements in the SOP conform to the reference method and applicable regulations for which results of the test are used and to any program requirements for the process required by the certification/accreditation program required for testing.

- When a project requires more stringent QC protocol than specified in PAS policy or the test method SOP, the project specification is followed.
- When the project requires less stringent QC protocol than PAS policy or the SOP, the project specification may be followed as an authorized departure if the project specification still meet the requirements in the test method and any applicable regulatory and certification/accreditation requirements.

The following sections describe essential QC for chemistry. These QC types may not apply to other technologies and disciplines such as microbiology, radiochemistry, whole effluent toxicity, and/or asbestos. For essential QC for these test offerings, refer to test method SOPs.

5.9.1.1.1 Calibration Verification (Second Source)

A second source standard is a standard from a different vendor or from a different lot than the standards used for initial calibration. A different lot from the same vendor is permissible, when the standard is only available from a single vendor.

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It is a positive control used to verify the accuracy of instrument calibration relative to the purity of the standards used for calibration. This check may be referred to in published test methods and quality system standards as the initial calibration verification (ICV) or a quality control sample (QCS). For most test methods, it is analyzed immediately after the calibration and before analysis of any samples. When the ICV is not within acceptance criteria, a problem with the purity or preparation of the standards may be indicated. The source of the problem should be investigated and corrected prior to use of the calibration/instrument for sample analysis.

5.9.1.1.2 Calibration Verification (CCV)

The CCV is used to determine if the analytical response has significantly changed since calibration. The CCV is usually made from the same standards used for initial calibration, but a second source standard may be used.

When the response of the CCV is within criteria, the calibration is considered valid. If not, there may be a problem that requires further investigation and correction. The actions taken are technology and method specific.

It is sometimes acceptable to report test results associated with a CCV that does not meet criteria.

For example, when a CCV exceeds criteria above the limit; high bias is suspected in the measurement system. If the analyte is not detected in the sample; then the high bias has no impact on the validity of the test result. If the analyte is detected in sample, then corrective action to bring the CCV within criteria is expected to eliminate the possibility that the detection is biased.

5.9.1.1.3 Blanks

Blanks are negative controls used to assess for contamination.

The method blank (MB) is a clean matrix similar to the associated samples that is known to be free of analytes of interest. The MB, unless otherwise specified by the test method, is processed with, and carried through all preparation and analytical steps as the associated samples.

The criteria used to assess for contamination depends on the intended use of data. In general, detections in the MB above the RL or ½ the RL indicate contamination. When contamination is indicated, the source is investigated, and corrections are taken to reduce or eliminate it. Analytical results associated with MB that do not meet criteria are qualified in the final test report.

Other types of blanks that serve as negative controls in the process may include:

- Trip Blanks (VOA)

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- Storage Blanks
- Equipment Blanks
- Field Blanks
- Calibration Blanks
- Cleanup Blanks
- Instrument Blanks

5.9.1.1.4 Laboratory Control Sample (LCS)

The LCS is a positive control used to measure the accuracy. The LCS is a blank matrix spiked with standard solution that includes target analytes or it may be a pre-made certified reference standard. Like the MB, unless otherwise specified in the test method, the LCS is processed with and carried through all preparation and analytical steps as the associated samples.

When the percent recovery (%R) of the LCS is within the established control limit, the test is within control. If not, the cause of the problem is investigated and corrected, and the procedure may be repeated. Analytical results associated with LCS that does not meet criteria are qualified in the final test report.

5.9.1.1.5 Matrix Spike (MS) and Matrix Spike Duplicate (MSD)

The MS and MSD are replicates of a client sample that is spiked with known amount of target analyte. When there is not a client-designated matrix spike on any sample in the batch, the MS/MSD may be performed on a randomly selected sample and these MS/MSD are called "batch" MS/MSD.

MS/MSD results provide information about the effects the sample matrix has on recovery and reproducibility of target analytes on site samples. MS/MSD results are not used to control performance of the preparation or analytical batch because the way in which the matrix impacts efficiency and robustness of the test cannot be mitigated or controlled by the laboratory.

The heterogeneity of the sample submatrix influences accuracy and precision. For example, MS/MSD results for a sample that is mostly sand will not be the same for a sediment sample even though both matrices are "soil" and both samples subjected to the same test procedure in the same batch. For this reason, batch MS/MSD results may not be representative of the effect the matrix has on accuracy and precision on test results for all samples in the batch – except when all samples in the batch are from the same collection site and are identical in composition.

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5.9.1.1.6 Sample Duplicate (SD)

A sample duplicate is a second replicate of sample taken through the testing process used to measure precision and/or homogeneity of samples collected.

The relative percent difference (RPD) between replicates are evaluated against the established acceptance criteria and if the RPD is not met, associated test results are reported with qualification.

5.9.1.1.7 Surrogates

Surrogates are compounds not normally found in the environment but that have similar physical and chemical properties of target analytes that are added to environmental samples, QC, and calibration standards prior to preparation and analysis. They are used to evaluate extraction efficiency and matrix interference on a sample specific basis.

The percent recovery of surrogates is evaluated against method-specified limits or statistically derived in-house limits. Project-specific limits and/or program-specific limits are used when required. Results with surrogate recovery out of limits in samples are reported with qualification. Samples with surrogate failures can also be re-extracted and/or re-analyzed to confirm that the out-of-control value was caused by the matrix of the sample and not by some other systematic error.

5.9.1.1.8 Internal Standards

Internal Standards are compounds not expected to occur naturally in field samples. They are added to every standard and sample at a known concentration prior to analysis for the purpose of adjusting the response factor used in quantifying target analytes. The location follows specific guidelines for the treatment of internal standard recoveries and further information can be found in the applicable test method SOP.

5.9.1.1.9 QC Acceptance Criteria and Control Limits

QC acceptance criteria are specified in test method SOPs. The criteria in the SOP are based on the requirements in the published test method or regulatory program. When there are no established acceptance criteria, the location develops acceptance criteria in accordance with recognized industry standards.

Some methods and programs require the laboratory to establish control limits for LCS, MS/MSD, and surrogate evaluation using historical data. PAS developed limits are referred to as "in-house" control limits. In-house control limits represent ± 3 Standard Deviations (99% confidence level) from the average recovery of at least 20 data points generated using the same preparation and analytical procedure in a similar matrix.

See SOP ENV-SOP-WES2-0119 *Control Limit Generation* for more information about the procedures used to establish in-house control limits.

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5.9.1.2 Proficiency Testing (PT)

PAS participates in interlaboratory proficiency testing (PT) studies to measure our performance of the test method.

PT participation is based on the certification and accreditation requirements held by the laboratory. The PT samples are obtained from accredited proficiency testing providers (PTP) and treated as field samples which means they are incorporated into our normal analytical processes and do not receive extraordinary attention due to their nature.

PAS locations do not share PT samples with other PAS locations, does not communicate with other PAS locations regarding current PT sample results during the duration of the study, and does not attempt to obtain the assigned value of any PT sample from the PT provider.

PT results scored unacceptable are investigated and correction action taken, when necessary.

Refer to corporate policy ENV-POL-CORQ-0002, *PT Policy* for more information.

5.9.2 QC Corrective Action

When the results of QC are not within acceptance criteria or expectations for method performance, correction and corrective action(s) are taken per the specifications in the test method SOP. These actions may include retesting or reporting of data with qualification to alert the end user of the situation.

5.9.3 Data Review

PAS uses a tiered system for data review. The tiered process includes as series of sequential checks to verify data transfer is complete; manual calculations, if performed, are correct, manual integrations are appropriate and documented, calibration and QC requirements are met, appropriate corrective action was taken when required, test results are properly qualified, process and test method SOPs were followed, project specific requirements were met, when applicable, and the test report is complete.

The sequential process includes three tiers referred to as primary review, secondary review, and administrative/completeness review.

Detailed procedures for the data review process are described in SOP ENV-SOP-WES2-0124 *Data Review*. General expectations for the tiered data review process are specified in the following sections:

5.9.3.1 Primary Review

Primary review is performed by the individual that performed the task. All PAS personnel are responsible for the review of their own work to assure it is complete, accurate, documented, and consistent with PAS policy and SOPs.

Checks performed during primary review include but are not limited to:

- Verification that data transfer and acquisition is complete;
- Manual calculations, if performed, are documented and accurate;

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- Manual integrations, if performed, are documented, and comply with SOP ENV-SOP-CORQ-006 *Manual Integration;*
- Calibration and QC criteria were met, instrument calibration conforms with policy ENV-POL-CORQ-005 *Acceptable Calibration Practices*, and/or proper correction and corrective actions were taken, and data and test results associated with QC and criteria exceptions are properly qualified;
- Work is consistent with SOPs and any other relevant instructional document such as SWI, program requirements, or project quality assurance plans (QAPP).

5.9.3.2 Secondary Review

Secondary review is an independent review of the data set performed by a qualified peer or supervisor. Secondary review includes a repeat of the checks performed during primary review in addition to chromatography review to verify the accuracy of quantitative analyte identification.

5.9.3.3 Completeness Review

Completeness review is an administrative review that is performed prior to release of the test report to the customer to verify the final test report is complete and meets project specifications. This review also assures that information necessary for the client's interpretation of results are explained in the case narrative or footnoted in the test report.

5.9.3.4 Data Audits

Test reports are randomly audited by local quality personnel to verify compliance with SOPs and to check for data integrity, technical accuracy, and compliance with the PAS QMS and any applicable federal, statutory, and program requirements. These audits are not part of the tertiary data review process that is performed prior to release of the test report. These audits are performed on test reports that have been previously released to the customer to avoid potential risks to impartiality and other factors that may influence thoroughness of review.

If any problems with the data or test results are found during the data audit, the impact of the nonconforming work is evaluated using the process described in Section 4.9.

Also see Section 4.14 for internal audits.

5.9.4 Calibration Certificates

PAS does not perform calibration activities for its customers and calibration certificates are not offered or issued.

5.9.5 Opinions and Interpretations

PAS provides objective data and information to its customers of sufficient detail for their interpretation and decision making. Objective data and information are based solely on fact and PAS does not attempt to explain the meaning (interpret) or offer a view or judgement (opinion) on usability of data.

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PAS personnel are not permitted to provide opinion on how a nonconformance impacts the usability of results or how test results and information should be used, or suggestions or guidance to the customer for improvement..

5.9.6 Subcontractor Reports

When analytical work is subcontracted to an organization external to PAS or its subsidiaries, PAS includes the test report from the subcontractor in its entirety as an amendment to the final test report.

Test results performed by multiple locations within the PAS network (internal subcontracting) may be merged into a single test report so long as the test report issued clearly identifies the location and address of each network location that performed testing, and which tests each PAS location performed. (See 5.10.2)

5.9.7 Electronic Transmission of Results

When test results and/or reports are submitted to the customer through electronic transmission, the procedures established in this manual for confidentiality and protection of data apply.

5.9.8 Format of Test Reports

The test formats offered by PAS are designed to accommodate each type of analytical test method performed and to minimize the possibility of misunderstanding or misuse of analytical results. The format of electronic data deliverables (EDD) follows the specifications for the EDD.

5.9.9 Amendments to Test Reports

Test reports that are revised or amended by the location after date of release of the original final test report to the customer are issued as a new test report that is clearly identified as an amendment or revision and that includes a reference to the originally issued final test report.

The customer is the organization doing business with PAS external to PAS.

Changes made to test results and data before the final test report is issued to the customer are not amendments or revisions and do not need to be tracked or marked as such, these changes are simply corrections to errors found during data review.

The procedure for report amendments and revision are outlined in SOP ENV-SOP-WES2-0120 *Report Generation and Approval.*

5.10 Reporting

5.10.1 General Requirements

PAS offers a wide variety of test report formats to meet project needs of Pace® customers and federal and state regulatory program requirements

The type and level of deliverable, including the electronic data deliverable (EDD) format are established between PAS and the customer during the contracting process. The report specifications include the test report format, protocol for the reporting limit (RL), conventions for the reporting of results less than the limit of quantitation (LOQ), and specification for the

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use of project or program specific data qualifiers. Information about review of analytical service requests is provided in Section 4.4.

5.10.2 Test Reports: Required Items

Regardless of deliverable or report requested, every test report issued by PAS and its subsidiaries includes each of the following items:

a) A Title;

b) The name and address of the location issuing the test report and for each location where testing was performed if different than address of the location issuing the report. When testing is done at multiple PAS locations, the report must also clearly identify which PAS location performed each test method;

c) Unique identification of the test report and on each page an identification number to link each page to the test report, and clear identification of the end of the report;

d) The name and address of the customer;

e) Identification of test methods used. When testing is done at multiple PAS locations, the report must also clearly identify which PAS location performed each test method;

f) Cross reference between client sample identification number (Sample ID) and the identification number for the sample (Lab ID) to provide unambiguous identification of samples;

g) The date of receipt of samples, condition of samples on receipt, and identification of any instance where receipt of the samples did not meet sample acceptance criteria;

h) Date and times of sample collection, receipt, preparation, and analysis;

i) Test results and units of measurement, and qualification of results associated with QC criteria exceptions, and identification of reported results outside of the calibration range;

j) All chains of custody (COC) including records of internal transfer between locations within PAS;

k) Name, title, signature of the person(s) authorizing release of the test report and date of release;

l) A statement that the results in the test report relate only to the items tested; and

m) Statement that the test report may not be reproduced except in full without written approval from PAS.

5.10.3 Test Reports: Supplemental Items

5.10.3.1 Supplemental Requirements

The following items are included in the test report when required or relevant:

a) Shipping manifests/bill of ladings as applicable when common couriers are utilized for shipment of samples,

b) Explanation of departure from test method SOPs including, what the departure was and why it was necessary.

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c) Statistical methods used. (Required for Whole Effluent Toxicity)

d) For solid samples, specification that results are reported on a dry weight or wet weight basis.

e) Signed Affidavit, when required by client or regulatory agency.

f) A statement of compliance/non-compliance with requirements or specifications (client, program, or standard) that includes identification of test results that did not meet acceptance criteria.

g) When requested by the client, statement of estimated measurement uncertainty. In general, for environmental testing, estimated uncertainty of measurement is extrapolated from LCS control limits. Control limits incorporate the expected variation of the data derived from the laboratory's procedure. When the control limits are specified by the test method or regulatory program, the control limits represent the expected variation of the test method and/or matrices for which the test method was designed.

h) If a claim of accreditation/certification is included in the test report, identification of any test methods or analytes for which accreditation/certification is not held by the location if the accrediting body offers accreditation/certification for the test method/analyte. The fields of accreditation/certification vary between agencies, and it cannot be presumed that because accreditation/certification is not held that it is offered or required.

i) Certification Information, including certificate number and issuing body.

For PAS locations accredited to ISO/IEC 17025:2017:

- Data included in the test report provided by a customer should be clearly identified.
- A statement that the test results apply only to the samples as received.

PAS does not provide opinion or interpretations about usability of data or test results in order to maintain impartiality with the testing services provided.

5.10.3.2 Test Reports: Sampling Information

The following items are included in the test report when samples are collected by PAS or when this information is necessary for the interpretation of test results:

a) Date of Sampling.

b) Unambiguous identification of material samples.

c) Location of sampling including diagrams, sketches, or photographs.

d) Reference to the sampling plan and procedures used.

e) Details of environmental conditions at time of sample that may impact test results.

f) Any standard or other specification for the sampling method or procedure, and deviations, additions to or exclusions from the specification concerned.

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6.0 REVISION HISTORY

This Version (Version 3):

SECTION	DESCRIPTION OF CHANGE
Management Approval Page	Created a new cover page for management approval date / time stamps to allow room for signatories. This information was previously on the Title Page.
Header	Updated Copyright
Title Page	Simplified options for laboratory name based on legal entity.
Approval Page	Revised language for clarity, added spot for TNI accredited laboratories to list the AB approved "TNI Technical Manager, however named" – this description is not a job title, it refers to the individual designated by the laboratory and approved by the AB to provide technical oversight for a specific discipline.
All Sections	Updated format where needed.
4.1.5.1.1	Updated deputies for key personnel.
4.4	Added TNI requirement to notify customers when PAS is proposing or has made a deviation to a contract previously approved by both parties.
4.8	Added reference to the PAS Corporate SOP for Client Complaints
4.9.1.3	Added "Temporary" to "Stop Work Order" to clarify that a stop work is temporary and distinct from permanent discontinuation of a testing service, which is called a reduction in capabilities. Updated content to clearly identify the roles authorized to lift the stop work order.
4.13.15	Deleted the reference to an obsolete policy and inserted a reference to the technical guide that replaced the policy.
4.13.2.3	Replaced last paragraph with a reference to another section to eliminate redundancy.
4.14	Rewrote entire section to refine language; no change in requirements made.
4.16	Added reference to PAS Corporate SOP for Data Impact Assessments
5.2.1	Updated definition for competency. Added roles authorized to provide opinion or consult on usability of test results.
5.2.2	Rewrote entire section to refine language; no change in requirements made.
5.4	Rewrote entire section to refine language; no change in requirements made.
5.5	Rewrote entire section to refine language; no change in requirements made.
5.6	Rewrote entire section to refine language; no change in requirements made.
5.8	Rewrote entire section to refine language; no change in requirements made.
5.8.1.1 -1.2	Clarified that a CoC is a legal record for sample collection and "legal COC" is an industry-defined protocol for tracking possession of samples.
5.9	Rewrote section and re-organized content to be more concise.
Glossary	Updated Definition List and Definitions
Section 8.0	Added Suggested Addendum for AIHA and NVLAP

This document supersedes the following documents:

Document Number	Title	Version
ENV-TMP-CORQ-0007	Quality Manual Template	02
ENV-TMP-CORQ-0007	Quality Manual Template	01
ENV-MAN-CORQ-0001	Quality Manual	00

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7.0 APPENDICES

7.1 Appendix A: Certification / Accreditation Listing

Disclaimer: The certifications / accreditation lists provided in this Appendix are those that were held by the PAS location on the effective date of this manual. This information is subject to change without notice and must not be considered valid proof of certification or accreditation status. This manual is not updated with each change made. Current certificates are accessible via the eDMS Portal for PAS employees. External parties should contact the location for the most current information.

7.1.1 PAS-WES2 8 Walkup Drive, Westborough, MA

Authority	ID	Authority	ID
Connecticut	PH-0826	North Carolina (DW)	25700
Illinois	200077	North Carolina (NPW/SCM)	666
Indiana	C-MA-03	Ohio - VAP	Based on NH
Kentucky	KY98045	Oregon	MA-1316
Maine	MA00086	Pennsylvania	68-03671
Maryland	348	Rhode Island	LAO00065
Massachusetts	M-MA086	Texas	T104704476-17-14
New Hampshire Primary	2064	Vermont	VT-0935
New Jersey	MA935	Virginia	460195
New York	11148		

7.1.2 PAS-MANS 320 Forbes Blvd. Mansfield, MA

Authority	ID	Authority	ID
Connecticut	PH-0825	New Jersey	MA015
ANAB/DoD	L2474	New York	11627
Illinois	200081	North Carolina	685
Indiana	C-MA-04	Ohio-VAP	Based on NH
Kentucky	KY98046	Oregon	MA-0262
Louisiana	3090	Pennsylvania	68-02089
Maine	MA00030	Rhode Island	LAO00299
Maryland	350	Texas	T104704419
Massachusetts	M-MA030	Vermont	VT-0015
Michigan	9110	Virginia	460194
Minnesota	025-999-495	Washington	C954
New Hampshire Primary	2062		

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7.1.3 PAS-MANS 120 Forbes Blvd. Mansfield, MA

Authority	ID	Authority	ID

7.2 Appendix B: Capability Listing

The capabilities listed in this Appendix were held by the location referenced on the effective date of this manual. This information is subject to change without notice. External parties should contact the location for the most current information.

Table Legend:

- Air = Air
- DW = Drinking Water
- NPW = Non-Potable Water
- SCM = Solid and Chemical Materials
- Waste = Non-Aqueous Phase Liquid (NAPL), Oil
- Tissue = Biota and Tissue

7.2.1 PAS-WES2 8 Walkup Drive, Westborough, MA

Parameter	Method	Matrices			
		DW	NPW	SCM	Waste
1,1,1,2-Tetrachloroethane	EPA 524.2	X			
1,1,1,2-Tetrachloroethane	EPA 8260D		X	X	
1,1,1-Trichloroethane	EPA 524.2	X			
1,1,1-Trichloroethane	EPA 624.1		X		
1,1,1-Trichloroethane	EPA 8260D		X	X	
1,1,2,2-Tetrachloroethane	EPA 524.2	X			
1,1,2,2-Tetrachloroethane	EPA 624.1		X		
1,1,2,2-Tetrachloroethane	EPA 8260D		X	X	
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	EPA 624.1		X		
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	EPA 8260D		X	X	
1,1,2-Trichloroethane	EPA 524.2	X			
1,1,2-Trichloroethane	EPA 624.1		X		
1,1,2-Trichloroethane	EPA 8260D		X	X	
1,1'-Biphenyl	EPA 8270E		X	X	
1,1-Dichloroethane	EPA 524.2	X			
1,1-Dichloroethane	EPA 624.1		X		

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1,1-Dichloroethane	EPA 8260D		X	X	
1,1-Dichloroethene	EPA 524.2	X			
1,1-Dichloroethene	EPA 624.1		X		
1,1-Dichloroethene	EPA 8260D		X	X	
1,1-Dichloropropene	EPA 524.2	X			
1,1-Dichloropropene	EPA 8260D		X	X	
1,2,3-Trichlorobenzene	EPA 524.2	X			
1,2,3-Trichlorobenzene	EPA 624.1		X		
1,2,3-Trichlorobenzene	EPA 8260D		X	X	
1,2,3-Trichloropropane	EPA 504.1	X			
1,2,3-Trichloropropane	EPA 524.2	X			
1,2,3-Trichloropropane	EPA 8011		X		
1,2,3-Trichloropropane	EPA 8260D		X	X	
1,2,4,5-Tetrachlorobenzene	EPA 8270E		X	X	
1,2,4-Trichlorobenzene	EPA 524.2	X			
1,2,4-Trichlorobenzene	EPA 624.1		X		
1,2,4-Trichlorobenzene	EPA 625.1		X		
1,2,4-Trichlorobenzene	EPA 8260D		X	X	
1,2,4-Trichlorobenzene	EPA 8270E		X	X	
1,2,4-Trimethylbenzene	EPA 524.2	X			
1,2,4-Trimethylbenzene	EPA 8260D		X	X	
1,2-Dibromo-3-Chloropropane (DBCP)	EPA 504.1	X			
1,2-Dibromo-3-Chloropropane (DBCP)	EPA 524.2	X			
1,2-Dibromo-3-Chloropropane (DBCP)	EPA 624.1		X		
1,2-Dibromo-3-Chloropropane (DBCP)	EPA 8011		X		
1,2-Dibromo-3-Chloropropane (DBCP)	EPA 8260D		X	X	
1,2-Dibromoethane (EDB)	EPA 504.1	X			
1,2-Dibromoethane (EDB)	EPA 524.2	X			
1,2-Dibromoethane (EDB)	EPA 8011		X		
1,2-Dibromoethane (EDB)	EPA 8260D		X	X	
1,2-Dichlorobenzene	EPA 524.2	X			
1,2-Dichlorobenzene	EPA 624.1		X		
1,2-Dichlorobenzene	EPA 8260D		X	X	

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1,2-Dichlorobenzene	EPA 8270E		X	X	
1,2-Dichloroethane	EPA 524.2	X			
1,2-Dichloroethane	EPA 624.1		X		
1,2-Dichloroethane	EPA 8260D		X	X	
1,2-Dichloropropane	EPA 524.2	X			
1,2-Dichloropropane	EPA 624.1		X		
1,2-Dichloropropane	EPA 8260D		X	X	
1,2-Diphenylhydrazine	EPA 625.1		X		
1,2-Diphenylhydrazine	EPA 8270E		X	X	
1,3,5-Trichlorobenzene	EPA 524.2	X			
1,3,5-Trichlorobenzene	EPA 8260D		X	X	
1,3,5-Trimethylbenzene	EPA 524.2	X			
1,3,5-Trimethylbenzene	EPA 8260D		X	X	
1,3-Dichlorobenzene	EPA 524.2	X			
1,3-Dichlorobenzene	EPA 624.1		X		
1,3-Dichlorobenzene	EPA 8260D		X	X	
1,3-Dichlorobenzene	EPA 8270E		X	X	
1,3-Dichloropropane	EPA 524.2	X			
1,3-Dichloropropane	EPA 624.1		X		
1,3-Dichloropropane	EPA 8260D		X	X	
1,3-Dinitrobenzene (1,3-DNB)	EPA 8270E		X		
1,4-Dichlorobenzene	EPA 524.2	X			
1,4-Dichlorobenzene	EPA 624.1		X		
1,4-Dichlorobenzene	EPA 8260D		X	X	
1,4-Dichlorobenzene	EPA 8270E		X	X	
1,4-Dioxane	EPA 624.1		X		
1,4-Dioxane	EPA 8260D		X	X	
1,4-Dioxane	EPA 8260D-SIM		X		
1,4-Dioxane	EPA 8270E			X	
1-Methylnaphthalene	EPA 8270E		X	X	
1-Methylnaphthalene	EPA 8270E-SIM			X	
2,2-Dichloropropane	EPA 524.2	X			
2,2-Dichloropropane	EPA 8260D		X	X	

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2,3,4,6-Tetrachlorophenol	EPA 625.1		X		
2,3,4,6-Tetrachlorophenol	EPA 8270E		X	X	
2,3-Dimethylaniline	EPA 8270E		X		
2,4,5-T	EPA 8151A		X	X	
2,4,5-TP (Silvex)	EPA 8151A		X	X	
2,4,5-Trichlorophenol	EPA 625.1		X		
2,4,5-Trichlorophenol	EPA 8270E		X	X	
2,4,5-Trimethylaniline	EPA 8270E		X		
2,4,6-Trichlorophenol	EPA 625.1		X		
2,4,6-Trichlorophenol	EPA 8270E		X	X	
2,4-D	EPA 8151A		X	X	
2,4-DB	EPA 8151A		X	X	
2,4-Dichlorophenol	EPA 625.1		X		
2,4-Dichlorophenol	EPA 8270E		X	X	
2,4-Dimethylaniline	EPA 8270E		X		
2,4-Dimethylphenol	EPA 625.1		X		
2,4-Dimethylphenol	EPA 8270E		X	X	
2,4-Dinitrophenol	EPA 625.1		X		
2,4-Dinitrophenol	EPA 8270E		X	X	
2,4-Dinitrotoluene (2,4-DNT)	EPA 625.1		X		
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270E		X	X	
2,6-Dinitrotoluene (2,6-DNT)	EPA 625.1		X		
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270E		X	X	
2-Butanone (Methyl ethyl ketone, MEK)	EPA 524.2	X			
2-Butanone (Methyl ethyl ketone, MEK)	EPA 624.1		X		
2-Butanone (Methyl ethyl ketone, MEK)	EPA 8260D		X	X	
2-Chloroethyl Vinyl ether	EPA 624.1		X		
2-Chloroethyl Vinyl ether	EPA 8260D		X	X	
2-Chloronaphthalene	EPA 625.1		X		
2-Chloronaphthalene	EPA 8270E		X	X	
2-Chlorophenol	EPA 625.1		X		
2-Chlorophenol	EPA 8270E		X	X	
2-Chlorotoluene	EPA 524.2	X			

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2-Chlorotoluene	EPA 8260D		X	X	
2-Ethylaniline	EPA 8270E		X		
2-Hexanone	EPA 524.2	X			
2-Hexanone	EPA 624.1		X		
2-Hexanone	EPA 8260D		X	X	
2-Methyl-4,6-dinitrophenol	EPA 625.1		X		
2-Methyl-4,6-dinitrophenol	EPA 8270E		X	X	
2-Methyl-4,6-dinitrophenol	EPA 8270E-SIM		X	X	
2-Methylnaphthalene	EPA 625.1		X		
2-Methylnaphthalene	EPA 8270E		X	X	
2-Methylnaphthalene	EPA 8270E-SIM		X	X	
2-Methylphenol	EPA 625.1		X		
2-Methylphenol	EPA 8270E		X	X	
2-Naphthylamine	EPA 8270E		X		
2-Nitroaniline	EPA 625.1		X		
2-Nitroaniline	EPA 8270E		X	X	
2-Nitrophenol	EPA 625.1		X		
2-Nitrophenol	EPA 8270E		X	X	
2-Nitropropane	EPA 8260D			X	
3,3'-Dichlorobenzidine	EPA 625.1		X		
3,3'-Dichlorobenzidine	EPA 8270E		X	X	
3,4-Dimethylaniline	EPA 8270E		X		
3-Methylphenol	EPA 625.1		X		
3-Methylphenol	EPA 8270E		X	X	
3-Nitroaniline	EPA 625.1		X		
3-Nitroaniline	EPA 8270E		X	X	
4,4'-DDD	EPA 608.3		X		
4,4'-DDD	EPA 8081B		X	X	
4,4'-DDE	EPA 608.3		X		
4,4'-DDE	EPA 8081B		X	X	
4,4'-DDT	EPA 608.3		X		
4,4'-DDT	EPA 8081B		X	X	
4-Bromophenyl phenyl ether	EPA 625.1		X		

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4-Bromophenyl phenyl ether	EPA 8270E		X	X	
4-Chloro-3-methylphenol	EPA 625.1		X		
4-Chloro-3-methylphenol	EPA 8270E		X	X	
4-Chloroaniline	EPA 625.1		X		
4-Chloroaniline	EPA 8270E		X	X	
4-Chloro-o-Toluidine	EPA 8270E		X		
4-Chlorophenyl phenyl ether	EPA 625.1		X		
4-Chlorophenyl phenyl ether	EPA 8270E		X	X	
4-Chlorotoluene	EPA 524.2	X			
4-Chlorotoluene	EPA 8260D		X	X	
4-Methyl-2-pentanone (MIBK)	EPA 524.2	X			
4-Methyl-2-pentanone (MIBK)	EPA 624.1		X		
4-Methyl-2-pentanone (MIBK)	EPA 8260D		X	X	
4-Methylphenol	EPA 625.1		X		
4-Methylphenol	EPA 8270E		X	X	
4-Nitroaniline	EPA 625.1		X		
4-Nitroaniline	EPA 8270E		X	X	
4-Nitrophenol	EPA 625.1		X		
4-Nitrophenol	EPA 8270E		X	X	
Acenaphthene	EPA 625.1		X		
Acenaphthene	EPA 8270E		X	X	
Acenaphthene	EPA 8270E-SIM		X	X	
Acenaphthylene	EPA 625.1		X		
Acenaphthylene	EPA 8270E		X	X	
Acenaphthylene	EPA 8270E-SIM		X		
Acenaphthylene	EPA 8270E-SIM		X		
Acetone	EPA 524.2	X			
Acetone	EPA 624.1		X		
Acetone	EPA 8260D		X	X	
Acetophenone	EPA 625.1		X		
Acetophenone	EPA 8270E		X	X	
Acid Digestion of Waters	EPA 3005A			X	
Acidity	SM 2310B		X		

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Acrolein	EPA 624.1		X		
Acrolein	EPA 8260D		X	X	
Acrylonitrile	EPA 624.1		X		
Acrylonitrile	EPA 8260D		X	X	
Alachlor	EPA 8081B		X	X	
Aldrin	EPA 608.3		X		
Aldrin	EPA 8081B		X	X	
Alkalinity	SM 2320B	X	X		
Allyl Chloride	EPA 8260D			X	
Alpha-BHC	EPA 608.3		X		
alpha-BHC	EPA 8081B		X	X	
alpha-Chlordane	EPA 608.3		X		
alpha-Chlordane	EPA 8081B		X	X	
Ammonia	EPA 350.1		X		
Ammonia	SM 4500NH3-B		X		
Ammonia	SM 4500NH3-H		X		
Aniline	EPA 625.1		X		
Aniline	EPA 8270E		X	X	
Anthracene	EPA 625.1		X		
Anthracene	EPA 8270E		X	X	
Anthracene	EPA 8270E-SIM		X	X	
Atrazine	EPA 625.1		X		
Atrazine	EPA 8270E		X	X	
Azobenzene	EPA 625.1		X		
Benzaldehyde	EPA 8270E		X	X	
Benzene	EPA 524.2	X			
Benzene	EPA 624.1		X		
Benzene	EPA 8260D		X	X	
Benzidine	EPA 625.1		X		
Benzidine	EPA 8270E		X	X	
Benzo(a)anthracene	EPA 625.1		X		
Benzo(a)anthracene	EPA 8270E		X	X	
Benzo(a)anthracene	EPA 8270E-SIM		X	X	

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Benzo(a)pyrene	EPA 625.1		X		
Benzo(a)pyrene	EPA 8270E		X	X	
Benzo(a)pyrene	EPA 8270E-SIM		X	X	
Benzo(b)fluoranthene	EPA 625.1		X		
Benzo(b)fluoranthene	EPA 8270E		X	X	
Benzo(b)fluoranthene	EPA 8270E-SIM		X	X	
Benzo(g,h,i)perylene	EPA 625.1		X		
Benzo(g,h,i)perylene	EPA 8270E		X	X	
Benzo(g,h,i)perylene	EPA 8270E-SIM		X	X	
Benzo(k)fluoranthene	EPA 625.1		X		
Benzo(k)fluoranthene	EPA 8270E		X	X	
Benzo(k)fluoranthene	EPA 8270E-SIM		X	X	
Benzoic acid	EPA 625.1		X		
Benzoic acid	EPA 8270E		X	X	
Benzyl alcohol	EPA 625.1		X		
Benzyl alcohol	EPA 8270E		X	X	
Beta-BHC	EPA 608.3		X		
beta-BHC	EPA 8081B		X	X	
Biochemical Oxygen Demand	SM 5210B		X		
Biochemical Oxygen Demand - Carbonaceous	SM 5210B		X		
bis(2-Chloroethoxy)methane	EPA 625.1		X		
bis(2-Chloroethoxy)methane	EPA 8270E		X	X	
bis(2-Chloroethyl)ether	EPA 625.1		X		
bis(2-Chloroethyl)ether	EPA 8270E		X	X	
bis(2-Chloroethyl)ether	EPA 8270E-SIM		X		
bis(2-Chloroisopropyl)ether	EPA 625.1		X		
bis(2-Chloroisopropyl)ether	EPA 8270E		X	X	
bis(2-Ethylhexyl)phthalate	EPA 625.1		X		
bis(2-Ethylhexyl)phthalate	EPA 8270E		X	X	
Bromide	EPA 300.0		X		
Bromobenzene	EPA 524.2	X			
Bromobenzene	EPA 8260D		X	X	
Bromochloromethane	EPA 524.2	X			

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Bromochloromethane	EPA 8260D		X	X	
Bromodichloromethane	EPA 524.2	X			
Bromodichloromethane	EPA 624.1		X		
Bromodichloromethane	EPA 8260D		X	X	
Bromoform	EPA 524.2	X			
Bromoform	EPA 624.1		X		
Bromoform	EPA 8260D		X	X	
Bromomethane	EPA 524.2	X			
Bromomethane	EPA 624.1		X		
Bromomethane	EPA 8260D		X	X	
Butylbenzyl phthalate	EPA 625.1		X		
Butylbenzyl phthalate	EPA 8270E		X	X	
Caprolactam	EPA 8270E		X	X	
Carbazole	EPA 625.1		X		
Carbazole	EPA 8270E		X	X	
Carbon Dioxide	SM 4500 CO2-D [1]		X		
Carbon disulfide	EPA 524.2	X			
Carbon disulfide	EPA 624.1		X		
Carbon disulfide	EPA 8260D		X	X	
Carbon Tetrachloride	EPA 524.2	X			
Carbon Tetrachloride	EPA 624.1		X		
Carbon Tetrachloride	EPA 8260D		X	X	
Chemical Oxygen Demand	EPA 410.4		X		
Chemical Oxygen Demand	SM 5220D		X		
Chemical Oxygen Demand, Flocculated	SM 5220D(m) [1]		X		
Chlordane	EPA 608.3		X		
Chlordane	EPA 8081B		X	X	
Chloride	EPA 300.0	X			
Chloride	EPA 300.0		X		
Chloride	EPA 9056A		X		
Chloride	EPA 9251		X	X	
Chloride	SM 4500CL-E		X		
Chlorine Demand	SM 2350B [1]	X	X		

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Chlorobenzene	EPA 524.2	X			
Chlorobenzene	EPA 624.1		X		
Chlorobenzene	EPA 8260D		X	X	
Chlorodifluoromethane	EPA 8260D			X	
Chloroethane (Ethyl chloride)	EPA 524.2	X			
Chloroethane (Ethyl chloride)	EPA 624.1		X		
Chloroethane (Ethyl chloride)	EPA 8260D		X	X	
Chloroform	EPA 524.2	X			
Chloroform	EPA 624.1		X		
Chloroform	EPA 8260D		X	X	
Chloromethane	EPA 524.2	X			
Chloromethane	EPA 624.1		X		
Chloromethane	EPA 8260D		X	X	
Chlorophyll A	SM 10200H [1]	X	X		
Chlorophyll A	EPA 446.0(m) [1]	X	X		
Chromium VI	EPA 7196A		X	X	
Chromium VI	SM 3500Cr-B		X		
Chromium VI Digestion	EPA 3060A		X	X	
Chrysene	EPA 625.1		X		
Chrysene	EPA 8270E		X	X	
Chrysene	EPA 8270E-SIM		X	X	
cis-1,2-Dichloroethene	EPA 524.2	X			
cis-1,2-Dichloroethene	EPA 624.1		X		
cis-1,2-Dichloroethene	EPA 8260D		X	X	
cis-1,3-Dichloropropene	EPA 524.2	X			
cis-1,3-Dichloropropene	EPA 624.1		X		
cis-1,3-Dichloropropene	EPA 8260D		X	X	
Coliform, Fecal (Enumeration QuantiTray)	SM 9223B		X		
Coliform, Total (Colilert P/A)	SM 9223B	X			
Coliform, Total (Enumeration QuantiTray)	SM 9223B	X			
Color	SM 2120B	X	X		
Cyanide, Amenable	EPA 9012B		X	X	
Cyanide, Free	EPA 9016 [1]		X	X	

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Cyanide, Free	SM 4500 CN-E (m) [1]		X	X	
Cyanide, Reactive	SW-846 Ch 7.3 [1]		X	X	
Cyanide, Total	EPA 9010C		X	X	
Cyanide, Total	EPA 9012B		X	X	
Cyanide, Total	EPA 9014		X	X	
Cyanide, Total	Lachat 10-204-00-1-A		X		
Cyanide, Total	SM 4500CN-CE	X	X		
Cyclohexane	EPA 8260D		X	X	
Cyclohexanone	EPA 8260D		X	X	
Dalapon	EPA 8151A		X	X	
Delta-BHC	EPA 608.3		X		
delta-BHC	EPA 8081B		X	X	
Density	ASTM D1475 [1]		X	X	X
Dibenzo(a,h)anthracene	EPA 625.1		X		
Dibenzo(a,h)anthracene	EPA 8270E		X	X	
Dibenzo(a,h)anthracene	EPA 8270E-SIM		X	X	
Dibenzofuran	EPA 625.1		X		
Dibenzofuran	EPA 8270E		X	X	
Dibromochloromethane	EPA 524.2	X			
Dibromochloromethane	EPA 624.1		X		
Dibromochloromethane	EPA 8260D		X	X	
Dibromomethane	EPA 524.2	X			
Dibromomethane	EPA 624.1		X		
Dibromomethane	EPA 8260D		X	X	
Dicamba	EPA 8151A		X	X	
Dichlorodifluoromethane (Freon-12)	EPA 524.2	X			
Dichlorodifluoromethane (Freon-12)	EPA 624.1		X		
Dichlorodifluoromethane (Freon-12)	EPA 8260D		X	X	
Dichloroprop	EPA 8151A		X	X	
Dieldrin	EPA 608.3		X		
Dieldrin	EPA 8081B		X	X	
Diesel Range Organics	EPA 8015D		X	X	
Diethyl ether	EPA 524.2	X			

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Diethyl ether	EPA 8260D		X	X	
Diethyl phthalate	EPA 625.1		X		
Diethyl phthalate	EPA 8270E		X	X	
Di-isopropylether (DIPE)	EPA 524.2	X			
Di-isopropylether (DIPE)	EPA 8260D		X	X	
Dimethyl phthalate	EPA 625.1		X		
Dimethyl phthalate	EPA 8270E		X	X	
Di-n-butyl phthalate	EPA 625.1		X		
Di-n-butyl phthalate	EPA 8270E		X	X	
Di-n-octyl phthalate	EPA 625.1		X		
Di-n-octyl phthalate	EPA 8270E		X	X	
Dinoseb	EPA 8151A		X		
Diphenylamine	EPA 8270E		X	X	
Dissolved Organic Carbon (DOC)	SM 5310C	X			
Dissolved Oxygen	SM 4500O-C		X		
E. Coli (Colilert P/A)	SM 9223B	X			
E. Coli (Enumeration QuantiTray)	SM 9223B	X			
E. Coli (Enumeration QuantiTray)	SM 9223B		X		
Endosulfan I	EPA 608.3		X		
Endosulfan I	EPA 8081B		X	X	
Endosulfan II	EPA 608.3		X		
Endosulfan II	EPA 8081B		X	X	
Endosulfan Sulfate	EPA 608.3		X		
Endosulfan Sulfate	EPA 8081B		X	X	
Endrin	EPA 608.3		X		
Endrin	EPA 8081B		X	X	
Endrin Aldehyde	EPA 608.3		X		
Endrin Aldehyde	EPA 8081B		X	X	
Endrin Ketone	EPA 608.3		X		
Endrin Ketone	EPA 8081B		X	X	
EPH	MA EPH v. 2.1		X	X	
Ethanol	EPA 624.1		X		
Ethanol	EPA 8260D		X	X	

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Ethyl Acetate	EPA 8260D		X	X	
Ethyl Methacrylate	EPA 8260D		X	X	
Ethylbenzene	EPA 524.2	X			
Ethylbenzene	EPA 624.1		X		
Ethylbenzene	EPA 8260D		X	X	
Ethyl-Tert-Butyl-Ether	EPA 524.2	X			
Ethyl-Tert-Butyl-Ether	EPA 8260D		X	X	
ETPH	CT ETPH		X	X	X
Ferrous Iron	SM 3500Fe-B		X		
Flashpoint	EPA 1010A		X	X	
Florisil Cleanup	EPA 3620C			X	
Fluoranthene	EPA 625.1		X		
Fluoranthene	EPA 8270E		X	X	
Fluoranthene	EPA 8270E-SIM		X	X	
Fluorene	EPA 625.1		X		
Fluorene	EPA 8270E		X	X	
Fluorene	EPA 8270E-SIM		X	X	
Fluoride	EPA 300.0	X	X		
Fluoride	EPA 9056A		X		
Fluoride	SM 4500F-C	X	X		
Fluoride Preliminary Distillation	SM 4500F-B		X		
Formaldehyde	EPA 8315A		X		
gamma-Chlordane	EPA 608.3		X		
gamma-Chlordane	EPA 8081B		X	X	
Gasoline Range Organics	EPA 8015D		X	X	
Heptachlor	EPA 608.3		X		
Heptachlor	EPA 8081B		X	X	
Heptachlor Epoxide	EPA 608.3		X		
Heptachlor Epoxide	EPA 8081B		X	X	
Heterotrophic Plate Count	SM 9215B	X	X		
Hexachlorobenzene	EPA 625.1		X		
Hexachlorobenzene	EPA 8270E		X	X	
Hexachlorobenzene	EPA 8270E-SIM		X	X	

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Hexachlorobutadiene	EPA 524.2	X			
Hexachlorobutadiene	EPA 625.1		X		
Hexachlorobutadiene	EPA 8260D		X	X	
Hexachlorobutadiene	EPA 8270E		X	X	
Hexachlorobutadiene	EPA 8270E-SIM		X	X	
Hexachlorocyclopentadiene	EPA 625.1		X		
Hexachlorocyclopentadiene	EPA 8270E		X	X	
Hexachloroethane	EPA 625.1		X		
Hexachloroethane	EPA 8260D			X	
Hexachloroethane	EPA 8270E		X	X	
Ignitability	EPA 1030			X	
Indeno(1,2,3-cd)pyrene	EPA 625.1		X		
Indeno(1,2,3-cd)pyrene	EPA 8270E		X	X	
Indeno(1,2,3-cd)pyrene	EPA 8270E-SIM		X	X	
Iodomethane (Methyl Iodide)	EPA 8260D		X	X	
Isophorone	EPA 625.1		X		
Isophorone	EPA 8270E		X	X	
Isopropyl Alcohol	EPA 8260D		X		
Isopropylbenzene	EPA 524.2	X			
Isopropylbenzene	EPA 624.1		X		
Isopropylbenzene	EPA 8260D		X	X	
Langelier Index Calculation	SM 2330B [1]	X	X		
Limestone Equivalent	1.1.04 B			X	
Lindane (gamma-BHC)	EPA 608.3		X		
Lindane (gamma-BHC)	EPA 8081B		X	X	
m+p-Xylene	EPA 624.1	X			
m+p-Xylene	EPA 524.2	X			
MCPA	EPA 8151A		X	X	
MCPP	EPA 8151A		X	X	
Methoxychlor	EPA 608.3		X		
Methoxychlor	EPA 8081B		X	X	
Methyl Acetate	EPA 8260D		X	X	
Methyl Cyclohexane	EPA 624.1		X		

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Methyl Cyclohexane	EPA 8260D		X	X	
Methyl Methacrylate	EPA 8260D		X	X	
Methyl tert-butyl ether (MTBE)	EPA 524.2	X			
Methyl tert-butyl ether (MTBE)	EPA 624.1		X		
Methyl tert-butyl ether (MTBE)	EPA 8260D		X	X	
Methylene Chloride	EPA 524.2	X			
Methylene Chloride	EPA 624.1		X		
Methylene Chloride	EPA 8260D		X	X	
Microwave Acid Digestion	EPA 3015A			X	
Microwave Extraction	EPA 3546			X	
Mirex	EPA 608.3		X		
M-Xylene	EPA 8260D		X	X	
Naphthalene	EPA 524.2	X			
Naphthalene	EPA 625.1		X		
Naphthalene	EPA 8260D		X	X	
Naphthalene	EPA 8270E		X	X	
Naphthalene	EPA 8270E-SIM		X	X	
n-Butyl Alcohol	EPA 8260D		X	X	
n-Butylbenzene	EPA 524.2	X			
n-Butylbenzene	EPA 8260D		X	X	
n-Decane	EPA 625.1		X		
n-Hexane	EPA 624.1		X		
n-Hexane	EPA 8260D		X	X	
Nitrate-N	EPA 300.0	X	X		
Nitrate-N	EPA 353.2	X	X		
Nitrate-N	EPA 9056A		X		
Nitrate-N	SM 4500NO3-F	X	X		
Nitrate-Nitrite	EPA 353.2		X		
Nitrate-Nitrite	SM 4500NO3-F	X	X		
Nitrite-N	EPA 353.2	X	X		
Nitrite-N	SM 4500NO2-B		X		
Nitrite-N	SM 4500NO3-F	X	X		
Nitrobenzene	EPA 625.1		X		

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Nitrobenzene	EPA 8260D			X	
Nitrobenzene	EPA 8270E		X	X	
Nitrogen, Total Kjeldahl	EPA 351.1		X		
Nitrogen, Total Kjeldahl	Lachat 10-107-06-2-D		X		
Nitrogen, Total Kjeldahl	SM 4500NH3-H		X		
Nitrogen, Total Kjeldahl (Distillation)	SM 4500Norg-C		X		
N-Nitrosodiethylamine	EPA 625.1		X		
n-Nitrosodimethylamine	EPA 625.1		X		
n-Nitrosodimethylamine	EPA 8270E		X	X	
n-Nitroso-di-n-propylamine	EPA 625.1		X		
n-Nitroso-di-n-propylamine	EPA 8270E		X	X	
N-Nitrosodiphenylamine	EPA 625.1		X		
N-Nitrosodiphenylamine	EPA 8270E		X	X	
N-Nitrosopyrrolidine	EPA 625.1		X		
N-Octadecane	EPA 625.1		X		
n-Propylbenzene	EPA 524.2	X			
n-Propylbenzene	EPA 8260D		X	X	
Odor	SM 2150B	X			
Oil & Grease	EPA 1664B		X		
Oil & Grease	EPA 9071B			X	
Oil & Grease (TPH)	EPA 1664B		X		
Oil Range Organics	EPA 8015D		X	X	
Organic Matter, Total	ASTM D2974		X	X	
Orthophosphate	SM 4500P-E	X			
Orthophosphate	SM 4500P-E		X		
Oxygen Reduction Potential	EPA 1498 [1]			X	
o-Toluidine	EPA 8270E		X		
O-Xylene	EPA 524.2	X			
O-Xylene	EPA 8260D		X	X	
Paint Filter (Free Liquid)	EPA 9095B			X	
Parathion	EPA 8270E		X	X	
PCB-1016	EPA 608.3		X		
PCB-1016	EPA 8082A		X	X	X

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PCB-1221	EPA 608.3		X		
PCB-1221	EPA 8082A		X	X	X
PCB-1232	EPA 608.3		X		
PCB-1232	EPA 8082A		X	X	X
PCB-1242	EPA 608.3		X		
PCB-1242	EPA 8082A		X	X	X
PCB-1248	EPA 608.3		X		
PCB-1248	EPA 8082A		X	X	X
PCB-1254	EPA 608.3		X		
PCB-1254	EPA 8082A		X	X	X
PCB-1260	EPA 608.3		X		
PCB-1260	EPA 8082A		X	X	X
PCB-1262	EPA 8082A		X	X	X
PCB-1268	EPA 8082A		X	X	X
Pentachloroethane	EPA 8260D		X		
Pentachloronitrobenzene	EPA 8270E		X	X	
Pentachlorophenol	EPA 625.1		X		
Pentachlorophenol	EPA 8270E		X	X	
Pentachlorophenol	EPA 8270E-SIM		X	X	
pH	EPA 9040C		X	X	
pH	EPA 9045D			X	
pH	SM 4500H+-B	X	X		
Phenanthrene	EPA 625.1		X		
Phenanthrene	EPA 8270E		X	X	
Phenanthrene	EPA 8270E-SIM		X	X	
Phenol	EPA 625.1		X		
Phenol	EPA 8270E		X	X	
p-Isopropyltoluene	EPA 524.2	X			
p-Isopropyltoluene	EPA 8260D		X	X	
Purge & Trap Aqueous	EPA 5030C		X	X	
Purge & Trap Soil Low/High	EPA 5035A			X	
P-Xylene	EPA 8260D		X	X	
Pyrene	EPA 625.1		X		

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Pyrene	EPA 8270E		X	X	
Pyrene	EPA 8270E-SIM		X	X	
Pyridine	EPA 625.1		X		
Pyridine	EPA 8270E		X	X	
Salinity	SM 2520B		X		
sec-Butylbenzene	EPA 524.2	X			
Sec-Butylbenzene	EPA 8260D		X	X	
Separatory Funnel Extraction	EPA 3510C		X		
Settleable Solids	SM 2540F		X		
Shake Extraction of Solid Waste With Water	ASTM D3987-12			X	
Silica Gel Cleanup	EPA 3630C		X	X	
Soxhlet Extraction	EPA 3540C			X	
Specific Conductance	EPA 120.1		X		
Specific Conductance	EPA 9050A		X	X	
Specific Conductance	SM 2510B	X	X		
SPLP	EPA 1312			X	
Styrene	EPA 524.2	X			
Styrene	EPA 624.1		X		
Styrene	EPA 8260D		X	X	
Sulfate	EPA 300.0	X	X		
Sulfate	EPA 9038		X	X	
Sulfate	EPA 9056A		X		
Sulfate	SM 4500SO4-E		X		
Sulfide	SM 4500S2-D		X		
Sulfide, Reactive	SW-846 Ch 7.3 [1]		X	X	
Sulfide Distillation	EPA 9030B		X		
Sulfite	SM 4500SO3-B		X		
Sulfur Cleanup	EPA 3660B			X	
Sulfuric Acid Cleanup	EPA 3665A		X	X	
Surfactants (MBAS)	SM 5540C		X		
Tannin & Lignin	SM 5550B		X		
TCLP	EPA 1311			X	
tert-Amyl methyl ether (TAME)	EPA 524.2	X			

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tert-Amyl methyl ether (TAME)	EPA 624.1		X		
tert-Amyl methyl ether (TAME)	EPA 8260D		X	X	
Tert-Butyl Alcohol	EPA 524.2	X			
Tert-Butyl Alcohol	EPA 624.1		X		
Tert-Butyl Alcohol	EPA 8260D		X	X	
Tert-Butylbenzene	EPA 524.2	X			
Tert-Butylbenzene	EPA 8260D		X	X	
Tetrachloroethene	EPA 524.2	X			
Tetrachloroethene	EPA 624.1		X		
Tetrachloroethene	EPA 8260D		X	X	
Tetrahydrofuran	EPA 524.2	X			
Tetrahydrofuran	EPA 8260D		X	X	
Thionazin	EPA 8270E		X		
Toluene	EPA 524.2	X			
Toluene	EPA 624.1		X		
Toluene	EPA 8260D		X	X	
Total Dissolved Solids	SM 2540C	X			
Total Dissolved Solids	SM 2540C		X		
Total EPH	NJDEP EPH		X	X	
Total Organic Carbon	EPA 9060A		X		
Total Organic Carbon	SM 5310C	X	X		
Total Petroleum Hydrocarbons	EPA 8015D		X		
Total Phenolics	EPA 420.1		X		
Total Phenolics	EPA 9065		X	X	
Total Phosphorus	SM 4500P-E		X		
Total Phosphorus (Digestion)	SM 4500P-B		X		
Total Residue	SM 2540B		X		
Total Residue (% Total Solids)	SM 2540G			X	
Total Suspended Solids	SM 2540D		X		
Total Volatile Solids	SM 2540E		X		
Total Volatile Solids	SM 2540G			X	
Toxaphene	EPA 608.3		X		
Toxaphene	EPA 8081B		X	X	

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Trans-1,2-Dichloroethene	EPA 524.2	X			
Trans-1,2-Dichloroethene	EPA 624.1		X		
Trans-1,2-Dichloroethene	EPA 8260D		X	X	
Trans-1,3-Dichloropropene	EPA 524.2	X			
Trans-1,3-Dichloropropene	EPA 624.1		X		
Trans-1,3-Dichloropropene	EPA 8260D		X	X	
Trans-1,4-Dichloro-2-butene	EPA 8260D		X	X	
Trichloroethene	EPA 524.2	X			
Trichloroethene	EPA 624.1		X		
Trichloroethene	EPA 8260D		X	X	
Trichlorofluoromethane	EPA 524.2	X			
Trichlorofluoromethane	EPA 624.1		X		
Trichlorofluoromethane	EPA 8260D		X	X	
Turbidity	EPA 180.1	X			
Turbidity	EPA 180.1		X		
Turbidity	SM 2130B	X	X		
Ultrasonic Extraction	EPA 3550C			X	
Ultraviolet Absorbance	EPA 5910B [1]	X	X		
Vinyl Acetate	EPA 624.1		X		
Vinyl Acetate	EPA 8260D		X	X	
Vinyl chloride	EPA 524.2	X			
Vinyl Chloride	EPA 624.1		X		
Vinyl Chloride	EPA 8260D		X	X	
VPH	MA VPH v. 2.1		X	X	
Waste Dilution	EPA 3580A			X	X
Xylenes, Total	EPA 524.2	X			
Xylenes, Total	EPA 624.1		X		
Xylenes, Total	EPA 8260D		X	X	

[1] = Laboratory does not hold TNI Accreditation for this test method.

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7.2.2 PAS-MANS 320 Forbes Blvd. Mansfield, MA

Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
1,1,1-Trichloroethane	EPA TO-15	X					
1,1,1-Trichloroethane	EPA TO-15 SIM	X					
1,1,2,2-Tetrachloroethane	EPA TO-15	X					
1,1,2,2-Tetrachloroethane	EPA TO-15 SIM	X					
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	EPA TO-15	X					
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	EPA TO-15 SIM	X					
1,1,2-Trichloroethane	EPA TO-15	X					
1,1,2-Trichloroethane	EPA TO-15 SIM	X					
1,1'-Biphenyl	EPA 8270E			X	X		X
1,1-Dichloroethane	EPA TO-15	X					
1,1-Dichloroethane	EPA TO-15 SIM	X					
1,1-Dichloroethene	EPA TO-15	X					
1,1-Dichloroethene	EPA TO-15 SIM	X					
1,2,3,5-Tetrachlorobenzene	EPA 8270E			X			
1,2,4,5-Tetrachlorobenzene	EPA 8270E			X	X		
1,2,4-Trichlorobenzene	EPA 8270E			X	X		
1,2,4-Trichlorobenzene	EPA TO-15	X					
1,2,4-Trichlorobenzene	EPA TO-15 SIM	X					
1,2,4-Trimethylbenzene	EPA TO-15	X					
1,2,4-Trimethylbenzene	EPA TO-15 SIM	X					
1,2-Dibromo-3-Chloropropane (DBCP)	EPA TO-15	X					
1,2-Dibromo-3-Chloropropane (DBCP)	EPA TO-15 SIM	X					
1,2-Dibromoethane (EDB)	EPA TO-15	X					
1,2-Dibromoethane (EDB)	EPA TO-15 SIM	X					
1,2-Dichlorobenzene	EPA 8270E			X	X		
1,2-Dichlorobenzene	EPA TO-15	X					
1,2-Dichlorobenzene	EPA TO-15 SIM	X					
1,2-Dichloroethane	EPA TO-15	X					
1,2-Dichloroethane	EPA TO-15 SIM	X					
1,2-Dichloropropane	EPA TO-15	X					

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
1,2-Dichloropropane	EPA TO-15 SIM	X					
1,2-Dichlorotetrafluoroethane	EPA TO-15	X					
1,2-Dichlorotetrafluoroethane	EPA TO-15 SIM	X					
1,2-Diphenylhydrazine	EPA 8270E			X	X		
1,3,5-Trimethylbenzene	EPA TO-15	X					
1,3,5-Trimethylbenzene	EPA TO-15 SIM	X					
1,3-Butadiene	EPA TO-15	X					
1,3-Butadiene	EPA TO-15 SIM	X					
1,3-Dichlorobenzene	EPA 8270E			X	X		
1,3-Dichlorobenzene	EPA TO-15	X					
1,3-Dichlorobenzene	EPA TO-15 SIM	X					
1,4-Dichlorobenzene	EPA 8270E			X	X		
1,4-Dichlorobenzene	EPA TO-15	X					
1,4-Dichlorobenzene	EPA TO-15 SIM	X					
1,4-Dioxane	EPA 522		X	X			
1,4-Dioxane	EPA 8270E-SIM			X	X		
1,4-Dioxane	EPA TO-15	X					
1,4-Dioxane	EPA TO-15 SIM	X					
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS) 763051-92-9	EPA 1633 DRAFT 2			X	X		X
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS) 763051-92-9	EPA 533		X				
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS) 763051-92-9	EPA 537.1		X				
11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11-Cl-PF3OUdS) 763051-92-9	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
1H, 1H, 2H, 2H-Perfluorodecanesulfonic acid (8:2 FTS)	EPA 1633 DRAFT 2			X	X		X
1H, 1H, 2H, 2H-Perfluorohexanesulfonic acid (4:2 FTS)	EPA 1633 DRAFT 2						X
1H, 1H, 2H, 2H-Perfluorooctanesulfonic acid (6:2 FTS)	EPA 1633 DRAFT 2						X
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2FTS)	EPA 533		X				
1H,1H, 2H, 2H-Perfluorodecane sulfonic acid (8:2FTS)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2FTS)	EPA 1633 DRAFT 2			X	X		
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2FTS)	EPA 533		X				
1H,1H, 2H, 2H-Perfluorohexane sulfonic acid (4:2FTS)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2FTS)	EPA 1633 DRAFT 2			X	X		
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2FTS)	EPA 533		X				

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
1H,1H, 2H, 2H-Perfluorooctane sulfonic acid (6:2FTS)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
1H,1H,2H,2H-perfluorododecane sulfonic acid (10:2FTS)	EPA 1633 DRAFT 2			X	X		
1H,1H,2H,2H-perfluorododecane sulfonic acid (10:2FTS)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
1-Methylnaphthalene	EPA 8270E						X
1-Methylnaphthalene	EPA 8270E-SIM			X	X		
1-Methylnaphthalene	EPA TO-13A SIM	X					
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol (NEtFOSE)	EPA 1633 DRAFT 2			X	X		
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol (NEtFOSE)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol (NMeFOSE)	EPA 1633 DRAFT 2			X	X		
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol (NMeFOSE)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
2,2,4-Trimethylpentane	EPA TO-15	X					
2,2,4-Trimethylpentane	EPA TO-15 SIM	X					
2,3,4,6-Tetrachlorophenol	EPA 8270E			X	X		
2,4,5-Trichlorophenol	EPA 8270E			X	X		
2,4,6-Trichlorophenol	EPA 8270E			X	X		
2,4-Dichlorophenol	EPA 8270E			X	X		
2,4-Dimethylphenol	EPA 8270E			X	X		
2,4-Dinitrophenol	EPA 8270E			X	X		
2,4-Dinitrotoluene (2,4-DNT)	EPA 8270E			X	X		
2,6-Dinitrotoluene (2,6-DNT)	EPA 8270E			X	X		
2-Butanone (Methyl ethyl ketone, MEK)	EPA TO-15	X					
2-Butanone (Methyl ethyl ketone, MEK)	EPA TO-15 SIM	X					
2-Chloronaphthalene	EPA 8270E			X	X		X
2-Chlorophenol	EPA 8270E			X	X		
2-Chlorotoluene	EPA TO-15	X					
2-Chlorotoluene	EPA TO-15 SIM	X					
2H,2H,3H,3H-Perfluorodecanoic acid (7:3 FTCA) 812-70-4	EPA 1633 DRAFT 2						X
2H,2H,3H,3H-Perfluorooctanoic acid (5:3 FTCA) 914637-49-3	EPA 1633 DRAFT 2			X	X		X
2-Hexanone	EPA TO-15	X					
2-Hexanone	EPA TO-15 SIM	X					
2-Methyl-4,6-dinitrophenol	EPA 8270E			X	X		
2-Methylnaphthalene	EPA 8270E			X	X		X

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
2-Methylnaphthalene	EPA 8270E-SIM			X	X		X
2-Methylnaphthalene	EPA TO-13A SIM	X					
2-Methylphenol	EPA 8270E			X	X		
2-Nitroaniline	EPA 8270E			X	X		
2-Nitrophenol	EPA 8270E			X	X		
3,3'-Dichlorobenzidine	EPA 8270E			X	X		
3-Methylphenol	EPA 8270E			X	X		
3-Nitroaniline	EPA 8270E			X	X		
3-Perfluoropropyl propanoic acid 3:3FTCA	EPA 1633 DRAFT 2			X	X		
4,4,5,5,6,6,6-Heptafluorohexanoic acid (3:3 FTCA) 356-02-5	EPA 1633 DRAFT 2						X
4,4'-DDD	EPA 8081B			X	X		X
4,4'-DDE	EPA 8081B			X	X		X
4,4'-DDT	EPA 8081B			X	X		X
4,8-Dioxa-3H-perfluorononanoic acid (ADONA) 919005-14-4	EPA 1633 DRAFT 2			X	X		
4,8-Dioxa-3H-perfluorononanoic acid (ADONA) 919005-14-4	EPA 533		X				
4,8-Dioxa-3H-perfluorononanoic acid (ADONA) 919005-14-4	EPA 537.1		X				
4,8-Dioxa-3H-perfluorononanoic acid (ADONA) 919005-14-4	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
4,8-Dioxa-3H-perfluorononanoic acid (DONA) 919005-14-4	EPA 1633 DRAFT 2						X
4-Bromophenyl phenyl ether	EPA 8270E			X	X		
4-Chloro-3-methylphenol	EPA 8270E			X	X		
4-Chloroaniline	EPA 8270E			X	X		
4-Chlorophenyl phenyl ether	EPA 8270E			X	X		
4-Ethyltoluene	EPA TO-15	X					
4-Ethyltoluene	EPA TO-15 SIM	X					
4-Methyl-2-pentanone (MIBK)	EPA TO-15	X					
4-Methyl-2-pentanone (MIBK)	EPA TO-15 SIM	X					
4-Methylphenol	EPA 8270E			X	X		
4-Nitroaniline	EPA 8270E			X	X		
4-Nitrophenol	EPA 8270E			X	X		
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS) 756426-58-1	EPA 1633 DRAFT 2			X	X		X
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS) 756426-58-1	EPA 533		X				
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS) 756426-58-1	EPA 537.1		X				

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9-Cl-PF3ONS) 756426-58-1	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Acenaphthene	EPA 8270E			X	X		X
Acenaphthene	EPA 8270E-SIM			X	X		X
Acenaphthene	EPA TO-13A Full Scan	X					
Acenaphthene	EPA TO-13A SIM	X					
Acenaphthylene	EPA 8270E			X	X		X
Acenaphthylene	EPA 8270E-SIM			X	X		X
Acenaphthylene	EPA TO-13A Full Scan	X					
Acenaphthylene	EPA TO-13A SIM	X					
Acetaldehyde	EPA TO-15	X					
Acetaldehyde	EPA TO-15 SIM	X					
Acetone	EPA TO-15	X					
Acetone	EPA TO-15 SIM	X					
Acetonitrile	EPA TO-15	X					
Acetonitrile	EPA TO-15 SIM	X					
Acetophenone	EPA 8270E			X	X		
Acetylene	RSK-175 [1]			X			
Acid Digestion of Waters	EPA 3015A			X			
Acrolein	EPA TO-15	X					
Acrolein	EPA TO-15 SIM	X					
Acrylonitrile	EPA TO-15	X					
Acrylonitrile	EPA TO-15 SIM	X					
Aldrin	EPA 8081B			X	X		X
Alkalinity	SM 2320B			X			
Alkylated PAHs	EPA 8270E-SIM			X	X		
Allyl chloride	EPA TO-15	X					
Allyl chloride	EPA TO-15 SIM	X					
alpha-BHC	EPA 8081B			X	X		X
alpha-Chlordane	EPA 8081B			X	X		X
Alumina Cleanup	EPA 3610B						X
Aluminum	EPA 200.7		X	X			
Aluminum	EPA 200.8		X	X			

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Aluminum	EPA 6010D			X	X		
Aluminum	EPA 6020B			X	X		X
Aniline	EPA 8270E			X	X		
Anthracene	EPA 8270E			X	X		X
Anthracene	EPA 8270E-SIM			X	X		X
Anthracene	EPA TO-13A Full Scan	X					
Anthracene	EPA TO-13A SIM	X					
Antimony	EPA 200.7			X			
Antimony	EPA 200.8		X	X			
Antimony	EPA 6010D			X	X		
Antimony	EPA 6020B			X	X		X
Arsenic	EPA 200.7			X			
Arsenic	EPA 200.8		X	X			
Arsenic	EPA 6010D			X	X		
Arsenic	EPA 6020B			X	X		X
Arsenic	EPA 1632A(m) [1]			X	X		
Ash Solids	ASTM D2974 [1]						X
Atrazine	EPA 8270E			X			
Barium	EPA 200.7		X	X			
Barium	EPA 200.8		X	X			
Barium	EPA 6010D			X	X		
Barium	EPA 6020B			X	X		X
Benzaldehyde	EPA 8270E			X	X		X
Benzene	EPA TO-15	X					
Benzene	EPA TO-15 SIM	X					
Benzidine	EPA 8270E			X	X		
Benzo(a)anthracene	EPA 8270E			X	X		X
Benzo(a)anthracene	EPA 8270E-SIM			X	X		X
Benzo(a)anthracene	EPA TO-13A Full Scan	X					
Benzo(a)anthracene	EPA TO-13A SIM	X					
Benzo(a)pyrene	EPA 8270E			X	X		X
Benzo(a)pyrene	EPA 8270E-SIM			X	X		X

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Benzo(a)pyrene	EPA TO-13A Full Scan	X					
Benzo(a)pyrene	EPA TO-13A SIM	X					
Benzo(b)fluoranthene	EPA 8270E			X	X		X
Benzo(b)fluoranthene	EPA 8270E-SIM			X	X		X
Benzo(b)fluoranthene	EPA TO-13A Full Scan	X					
Benzo(b)fluoranthene	EPA TO-13A SIM	X					
Benzo(e)pyrene	EPA TO-13A Full Scan	X					
Benzo(e)pyrene	EPA TO-13A SIM	**X**					
Benzo(g,h,i)perylene	EPA 8270E			X	X		X
Benzo(g,h,i)perylene	EPA 8270E-SIM			X	X		X
Benzo(g,h,i)perylene	EPA TO-13A Full Scan	X					
Benzo(g,h,i)perylene	EPA TO-13A SIM	X					
Benzo(k)fluoranthene	EPA 8270E			X	X		X
Benzo(k)fluoranthene	EPA 8270E-SIM			X	X		X
Benzo(k)fluoranthene	EPA TO-13A Full Scan	X					
Benzo(k)fluoranthene	EPA TO-13A SIM	X					
Benzoic acid	EPA 8270E			X	X		
Benzyl alcohol	EPA 8270E			X	X		
Benzyl Chloride	EPA TO-15	X					
Benzyl Chloride	EPA TO-15 SIM	X					
Beryllium	EPA 200.7			X			
Beryllium	EPA 200.8		X	X			
Beryllium	EPA 6010D			X	X		
Beryllium	EPA 6020B			X	X		X
beta-BHC	EPA 8081B			X	X		X
bis(2-Chloroethoxy)methane	EPA 8270E			X	X		
bis(2-Chloroethyl)ether	EPA 8270E			X	X		
bis(2-Chloroisopropyl)ether	EPA 8270E			X	X		
bis(2-Ethylhexyl)phthalate	EPA 8270E			X	X		X
Boron	EPA 200.7		X	X			
Boron	EPA 6010D			X	X		
Boron	EPA 6020B			X			

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Bromodichloromethane	EPA TO-15	X					
Bromodichloromethane	EPA TO-15 SIM	X					
Bromoform	EPA TO-15	X					
Bromoform	EPA TO-15 SIM	X					
Bromomethane	EPA TO-15	X					
Bromomethane	EPA TO-15 SIM	X					
Butane	RSK-175 [1]			X			
Butylbenzyl phthalate	EPA 8270E			X	X		X
Cadmium	EPA 200.7		X	X			
Cadmium	EPA 200.8		X	X			
Cadmium	EPA 6010D			X	X		
Cadmium	EPA 6020B			X	X		X
Cadmium	AVS-SEM [1]				X		
Calcium	EPA 200.7		X	X			
Calcium	EPA 6010D			X	X		
Calcium	EPA 6020B			X	X		X
Calcium Hardness	EPA 200.7		X				
Caprolactam	EPA 8270E			X	X		
Carbazole	EPA 8270E			X	X		
Carbon Dioxide	RSK-175 [1]			X			
Carbon disulfide	EPA 3C	X					
Carbon disulfide	EPA TO-15	X					
Carbon disulfide	EPA TO-15 SIM	X					
Carbon Tetrachloride	EPA TO-15	X					
Carbon Tetrachloride	EPA TO-15 SIM	X					
Chlordane	EPA 8081B			X	X		X
Chlorobenzene	EPA TO-15	X					
Chlorobenzene	EPA TO-15 SIM	X					
Chloroethane (Ethyl chloride)	EPA TO-15	X					
Chloroethane (Ethyl chloride)	EPA TO-15 SIM	X					
Chloroform	EPA TO-15	X					
Chloroform	EPA TO-15 SIM	X					

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Chloromethane	EPA TO-15	X					
Chloromethane	EPA TO-15 SIM	X					
Chromium	EPA 200.7		X	X			
Chromium	EPA 200.8		X	X			
Chromium	EPA 6010D			X	X		
Chromium	EPA 6020B			X	X		X
Chromium	AVS-SEM [1]				X		
Chrysene	EPA 8270E			X	X		X
Chrysene	EPA 8270E-SIM			X	X		
Chrysene	EPA TO-13A Full Scan	X					
Chrysene	EPA TO-13A SIM	X					
cis-1,2-Dichloroethene	EPA TO-15	X					
cis-1,2-Dichloroethene	EPA TO-15 SIM	X					
cis-1,3-Dichloropropene	EPA TO-15	X					
cis-1,3-Dichloropropene	EPA TO-15 SIM	X					
Cobalt	EPA 200.7		X	X			
Cobalt	EPA 200.8		X	X			
Cobalt	EPA 6010D			X	X		
Cobalt	EPA 6020B			X	X		X
Copper	EPA 200.7		X	X			
Copper	EPA 200.8		X	X			
Copper	EPA 6010D			X	X		
Copper	EPA 6020B			X	X		X
Copper	AVS-SEM [1]				X		
Cyclohexane	EPA TO-15	X					
Cyclohexane	EPA TO-15 SIM	X					
Delta-BHC	EPA 8081B			X	X		X
Density	SM 2520C [1]					X	X
Density	ASTM D7263 [1]				X		
Dibenzo(a,h)anthracene	EPA 8270E			X	X		X
Dibenzo(a,h)anthracene	EPA 8270E-SIM			X	X		X
Dibenzo(a,h)anthracene	EPA TO-13A Full Scan	X					

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Dibenzo(a,h)anthracene	EPA TO-13A SIM	X					
Dibenzofuran	EPA 8270E			X	X		X
Dibromochloromethane	EPA TO-15	X					
Dibromochloromethane	EPA TO-15 SIM	X					
Dichlorodifluoromethane (Freon-12)	EPA TO-15	X					
Dichlorodifluoromethane (Freon-12)	EPA TO-15 SIM	X					
Dichlorofluoromethane	EPA TO-15	X					
Dichlorofluoromethane	EPA TO-15 SIM	X					
Dieldrin	EPA 8081B			X	X		X
Diesel Range Organics	EPA 8015D			X	X		
Diethyl phthalate	EPA 8270E			X	X		X
Dimethyl phthalate	EPA 8270E			X	X		X
Di-n-butyl phthalate	EPA 8270E			X	X		X
Di-n-octyl phthalate	EPA 8270E			X	X		X
Endosulfan I	EPA 8081B			X	X		X
Endosulfan II	EPA 8081B			X	X		X
Endosulfan Sulfate	EPA 8081B			X	X		X
Endrin	EPA 8081B			X	X		X
Endrin Aldehyde	EPA 8081B			X	X		X
Endrin ketone	EPA 8081B			X	X		X
Ethane	RSK-175 [1]			X			
Ethene	RSK-175 [1]			X			
Ethanol	EPA 8015D			X	X		
Ethanol	EPA 8270E-SIM(m) 1					**6**	
Ethanol	EPA TO-15	X					
Ethanol	EPA TO-15 SIM	X					
Ethyl acetate	EPA TO-15	X					
Ethyl acetate	EPA TO-15 SIM	X					
Ethylbenzene	EPA TO-15	X					
Ethylbenzene	EPA TO-15 SIM	X					
Ethylene Glycol	EPA 8015D			X	X		
Fluoranthene	EPA 8270E			X	X		X

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Fluoranthene	EPA 8270E-SIM			X	X		X
Fluoranthene	EPA TO-13A Full Scan	X					
Fluoranthene	EPA TO-13A SIM	X					
Fluorene	EPA 8270E			X	X		X
Fluorene	EPA 8270E-SIM			X	X		X
Fluorene	EPA TO-13A Full Scan	X					
Fluorene	EPA TO-13A SIM	X					
Fractional Organic Carbon	ASTM D2974 [1]				X		
gamma-Chlordane	EPA 8081B			X	X		X
Gel-Permeation Clean-up	EPA 3640A			X	X		X
Grain Size	ASTM D6913 [1]				X		
Grain Size	ASTM D7928 [1]				X		
Heptachlor	EPA 8081B			X	X		X
Heptachlor Epoxide	EPA 8081B			X	X		X
Hexachlorobenzene	EPA 8081B			X	X		
Hexachlorobenzene	EPA 8270E			X	X		
Hexachlorobutadiene	EPA 8270E			X	X		
Hexachlorobutadiene	EPA TO-15	X					
Hexachlorobutadiene	EPA TO-15 SIM	X					
Hexachlorocyclopentadiene	EPA 8270E			X	X		
Hexachloroethane	EPA 8270E			X	X		
Hexafluoropropylene oxide dimer acid (HFPO-DA) 13252-13-6	EPA 533		X				
Hexafluoropropylene oxide dimer acid (HFPO-DA) 13252-13-6	EPA 537.1		X				
Hexafluoropropyleneoxide dimer acid (HFPO-DA) (GenX) 13252-13-6	EPA 1633 DRAFT 2						X
Indeno(1,2,3-cd)pyrene	EPA 8270E			X	X		X
Indeno(1,2,3-cd)pyrene	EPA 8270E-SIM			X	X		X
Indeno(1,2,3-cd)pyrene	EPA TO-13A Full Scan	X					
Indeno(1,2,3-cd)pyrene	EPA TO-13A SIM	X					
Iron	EPA 200.7		X	X			
Iron	EPA 200.8		X	X			
Iron	EPA 6010D			X	X		
Iron	EPA 6020B			X	X		X

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Iron	AVS-SEM [1]				X		
Iso-butyl Alcohol	EPA 8015D			X	X		
Isophorone	EPA 8270E			X	X		
Isopropyl Alcohol	EPA 8015D			X	X		
Isopropyl Alcohol	EPA TO-15	X					
Isopropyl Alcohol	EPA TO-15 SIM	X					
Isopropylbenzene	EPA TO-15	X					
Isopropylbenzene	EPA TO-15 SIM	X					
Lead	EPA 200.7			X			
Lead	EPA 200.8		X	X			
Lead	EPA 6010D			X	X		
Lead	EPA 6020B			X	X		X
Lead	AVS-SEM [1]				X		
Lead, Tetraethyl	EPA 8270E-SIM(m) [1]			X	X	X	
Lead, Tetramethyl	EPA 8270E-SIM(m) [1]			X	X	X	
Lead, Trimethyl Ethyl	EPA 8270E-SIM(m) [1]			X	X	X	
Lead, Diethyl Dimethyl	EPA 8270E-SIM(m) [1]			X	X	X	
Lead, Methyl Triethyl	EPA 8270E-SIM(m) [1]			X	X	X	
Lead on Air Filter	EPA 40 CFR Part 50 App. G	X					
Lindane (gamma-BHC)	EPA 8081B			X	X		X
Lipids, Percent	NOS ORCA 130 [1]						X
Lithium	EPA 200.7		X				
Lithium	EPA 6010D			X			
Magnesium	EPA 200.7		X	X			
Magnesium	EPA 6010D			X	X		
Magnesium	EPA 6020B			X	X		X
Manganese	EPA 200.7		X	X			
Manganese	EPA 200.8		X	X			
Manganese	EPA 6010D			X	X		
Manganese	EPA 6020B			X	X		X
Manganese	AVS-SEM [1]				X		
Mercury	EPA 1631E			X			

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Mercury	EPA 245.1		X	X			
Mercury	EPA 7470A			X			
Mercury	EPA 7471B				X		
Mercury	EPA 7474				X		
Mercury	NIOSH 6009 [1]	X					
Methane	EPA 3C	X					
Methane	RSK-175 [1]			X			
Methoxychlor	EPA 8081B			X	X		X
Methyl Alcohol (methanol)	EPA 8015D			X	X		
Methyl Alcohol (methanol)	EPA TO-15	X					
Methyl Alcohol (methanol)	EPA TO-15 SIM	X					
Methyl methacrylate	EPA TO-15	X					
Methyl methacrylate	EPA TO-15 SIM	X					
Methyl tert-butyl ether (MTBE)	EPA TO-15	X					
Methyl tert-butyl ether (MTBE)	EPA TO-15 SIM	X					
Methylene Chloride	EPA TO-15	X					
Methylene Chloride	EPA TO-15 SIM	X					
Microscale Solvent Extraction (MSE)	EPA 3570				X		X
Microwave Acid Digestion	EPA 3050B				X		X
Microwave Acid Digestion	EPA 3051A				X		X
Mirex	EPA 8081B			X	X		
Molybdenum	EPA 200.7			X			
Molybdenum	EPA 200.8			X			
Molybdenum	EPA 6010D			X	X		
Molybdenum	EPA 6020B			X	X		X
m-Xylene	EPA TO-15	X					
m-Xylene	EPA TO-15 SIM	X					
Naphthalene	EPA 8270E			X	X		X
Naphthalene	EPA 8270E-SIM			X	X		X
Naphthalene	EPA TO-13A Full Scan	X					
Naphthalene	EPA TO-13A SIM	X					
Naphthalene	EPA TO-15	X					

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Naphthalene	EPA TO-15 SIM	X					
n-Butylbenzene	EPA TO-15	X					
n-Butylbenzene	EPA TO-15 SIM	X					
N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	EPA 1633 DRAFT 2			X	X		
N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	EPA 537.1		X				
N-ethyl perfluorooctanesulfonamidoacetic acid (NEtFOSAA)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
N-ethylperfluoro-1-octanesulfonamide (NEtFOSA)	EPA 1633 DRAFT 2			X	X		
N-ethylperfluoro-1-octanesulfonamide (NEtFOSA)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
N-Ethylperfluorooctane sulfonamide (EtFOSAm) 4151-50-2	EPA 1633 DRAFT 2						X
N-Ethylperfluorooctane sulfonamido acetic acid (NEtFOSAA) 2991-50-6	EPA 1633 DRAFT 2						X
N-Ethylperfluorooctane sulfonamido ethanol (EtFOSE) 1691-99-2	EPA 1633 DRAFT 2						X
n-Heptane	EPA TO-15	X					
n-Heptane	EPA TO-15 SIM	X					
n-Hexane	EPA TO-15	X					
n-Hexane	EPA TO-15 SIM	X					
Nickel	EPA 200.7		X	X			
Nickel	EPA 200.8		X	X			
Nickel	EPA 6010D			X	X		
Nickel	EPA 6020B			X	X		X
Nickel	AVS-SEM [1]				X		
Nitrobenzene	EPA 8270E			X	X		
Nitrogen	EPA 3C	X					
N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	EPA 1633 DRAFT 2			X	X		
N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	EPA 537.1		X				
N-methyl perfluorooctanesulfonamidoacetic acid (NMeFOSAA)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
N-methylperfluoro-1-octanesulfonamide (NMeFOSA)	EPA 1633 DRAFT 2			X	X		
N-methylperfluoro-1-octanesulfonamide (NMeFOSA)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
N-Methylperfluorooctane sulfonamide (MeFOSA) 31506-32-8	EPA 1633 DRAFT 2						X
N-Methylperfluorooctane sulfonamido acetic acid (NMeFOSAA) 2355-31-9	EPA 1633 DRAFT 2						X
N-Methylperfluorooctane sulfonamido ethanol (MeFOSE) 24448-09-7	EPA 1633 DRAFT 2						X
n-Nitrosodimethylamine	EPA 8270E			X	X		
n-Nitroso-di-n-propylamine	EPA 8270E			X	X		

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
N-Nitrosodiphenylamine	EPA 8270E			X	X		
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) 151772-58-6	EPA 1633 DRAFT 2			X	X		X
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) 151772-58-6	EPA 533		X				
Nonafluoro-3,6-dioxaheptanoic acid (NFDHA) 151772-58-6	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Non-Methane Organic Compounds	EPA TO-12	X					
n-Propanol	EPA 8015D			X	X		
n-Propylbenzene	EPA TO-15	X					
n-Propylbenzene	EPA TO-15 SIM	X					
Organic Matter, Total	ASTM D2974 [1]				X		
Oxygen	EPA 3C	X					
o-Xylene	EPA TO-15	X					
o-Xylene	EPA TO-15 SIM	X					
Particulate Matter (PM-10)	IO-3.1 [1]	X					
Particulate Matter (TSP)	IO-3.1 [1]	X					
PCB-1016	EPA 8082A			X	X		X
PCB-1016	EPA TO-10A	X					
PCB-1016	EPA TO-4A	X					
PCB-1221	EPA 8082A			X	X		X
PCB-1221	EPA TO-10A	X					
PCB-1221	EPA TO-4A	X					
PCB-1232	EPA 8082A			X	X		X
PCB-1232	EPA TO-10A	X					
PCB-1232	EPA TO-4A	X					
PCB-1242	EPA 8082A			X	X		X
PCB-1242	EPA TO-10A	X					
PCB-1242	EPA TO-4A	X					
PCB-1248	EPA 8082A			X	X		X
PCB-1248	EPA TO-10A	X					
PCB-1248	EPA TO-4A	X					
PCB-1254	EPA 8082A			X	X		X
PCB-1254	EPA TO-10A	X					
PCB-1254	EPA TO-4A	X					

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
PCB-1260	EPA 8082A			X	X		X
PCB-1260	EPA TO-10A	X					
PCB-1260	EPA TO-4A	X					
PCB-1262	EPA 8082A			X	X		
PCB-1262	EPA TO-10A	X					
PCB-1262	EPA TO-4A	X					
PCB-1268	EPA 8082A			X	X		
PCB-1268	EPA TO-10A	X					
PCB-1268	EPA TO-4A	X					
PCB Congeners and Homologs	EPA 680(m) [1]			X	X		X
PCB Congeners and Homologs	EPA 8270E-SIM(m) [1]			X	X		X
Pentachloronitrobenzene	EPA 8270E			X	X		
Pentachlorophenol	EPA 8270E			X	X		X
Pentachlorophenol	EPA 8270E-SIM			X	X		
Perfluoro(2-ethoxyethane) sulfonic acid (PFEESA) 113507-82-7	EPA 1633 DRAFT 2			X	X		X
Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA) 113507-82-7	EPA 533		X				
Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA) 113507-82-7	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluoro-3-methoxypropannoic acid (PFMPA) 377-73-1	EPA 1633 DRAFT 2			X	X		
Perfluoro-3-methoxypropannoic acid (PFMPA) 377-73-1	EPA 533		X				
Perfluoro-3-methoxypropannoic acid (PFMPA) 377-73-1	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluoro-3-methoxypropanoic acid (PFMPA) 377-73-1	EPA 1633 DRAFT 2						X
Perfluoro-4-methoxybutanoic acid (PFMBA) 863090-89-5	EPA 1633 DRAFT 2			X	X		X
Perfluoro-4-methoxybutanoic acid (PFMBA) 863090-89-5	EPA 533		X				
Perfluoro-4-methoxybutanoic acid (PFMBA) 863090-89-5	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorobutane sulfonic acid (PFBS) 375-73-5	EPA 1633 DRAFT 2			X	X		X
Perfluorobutane sulfonic acid (PFBS) 375-73-5	EPA 533		X				
Perfluorobutane sulfonic acid (PFBS) 375-73-5	EPA 537.1		X				
Perfluorobutane sulfonic acid (PFBS) 375-73-5	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorobutanoic acid (PFBA) 375-22-4	EPA 1633 DRAFT 2			X	X		X
Perfluorobutanoic acid (PFBA) 375-22-4	EPA 533		X				
Perfluorobutanoic acid (PFBA) 375-22-4	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorodecane sulfonic acid (PFDS) 335-77-3	EPA 1633 DRAFT 2			X	X		X

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Perfluorodecanesulfonic acid (PFDS)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorodecanoic acid (PFDA) 335-76-2	EPA 1633 DRAFT 2			X	X		X
Perfluorodecanoic acid (PFDA) 335-76-2	EPA 533		X				
Perfluorodecanoic acid (PFDA) 335-76-2	EPA 537.1		X				
Perfluorodecanoic acid (PFDA) 335-76-2	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorododecane sulfonic acid (PFDoS) 79780-39-5	EPA 1633 DRAFT 2			X	X		X
Perfluorododecanesulfonic acid (PFDoS)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorododecanoic acid (PFDOA) 307-55-1	EPA 1633 DRAFT 2			X	X		X
Perfluorododecanoic acid (PFDoA) 307-55-1	EPA 533		X				
Perfluorododecanoic acid (PFDoA) 307-55-1	EPA 537.1		X				
Perfluorododecanoic acid (PFDoA) 307-55-1	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluoroheptane sulfonic acid (PFHpS) 375-92-8	EPA 1633 DRAFT 2			X	X		X
Perfluoroheptanesulfonic Acid (PFHPS) 375-92-8	EPA 533		X				
Perfluoroheptanesulfonic Acid (PFHPS) 375-92-8	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluoroheptanoic acid (PFHpA) 375-85-9	EPA 1633 DRAFT 2			X	X		X
Perfluoroheptanoic acid (PFHpA) 375-85-9	EPA 533		X				
Perfluoroheptanoic acid (PFHpA) 375-85-9	EPA 537.1		X				
Perfluoroheptanoic acid (PFHpA) 375-85-9	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluoroheptyl propanoic acid 7:3FTCA	EPA 1633 DRAFT 2			X	X		
Perfluorohexadecanoic acid (PFHxDA)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorohexane sulfonic acid (PFHxS) 355-46-4	EPA 1633 DRAFT 2			X	X		X
Perfluorohexane sulfonic acid (PFHxS) 355-46-4	EPA 533		X				
Perfluorohexane sulfonic acid (PFHxS) 355-46-4	EPA 537.1		X				
Perfluorohexane sulfonic acid (PFHxS) 355-46-4	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorohexanoic acid (PFHxA) 307-24-4	EPA 1633 DRAFT 2			X	X		X
Perfluorohexanoic acid (PFHXA) 307-24-4	EPA 533		X				
Perfluorohexanoic acid (PFHXA) 307-24-4	EPA 537.1		X				
Perfluorohexanoic acid (PFHXA) 307-24-4	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorononane sulfonic acid (PFNS) 68259-12-1	EPA 1633 DRAFT 2			X	X		X
Perfluorononanesulfonic acid (PFNS)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorononanoic acid (PFNA) 375-95-1	EPA 1633 DRAFT 2			X	X		X
Perfluorononanoic acid (PFNA) 375-95-1	EPA 533		X				

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Perfluorononanoic acid (PFNA) 375-95-1	EPA 537.1		X				
Perfluorononanoic acid (PFNA) 375-95-1	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorooctadecanoic acid (PFODA)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorooctane Sulfonamide (PFOSA)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorooctane sulfonamide (PFOSAm) 754-91-6	EPA 1633 DRAFT 2						X
Perfluorooctane sulfonic acid (PFOS) 1763-23-1	EPA 1633 DRAFT 2						X
Perfluorooctane sulfonic acid (PFOS) 1763-23-1	EPA 533		X				
Perfluorooctanesulfonamide (PFOSA)	EPA 1633 DRAFT 2			X	X		
Perfluorooctanesulfonic acid (PFOS)	EPA 1633 DRAFT 2			X	X		
Perfluorooctanesulfonic acid (PFOS)	EPA 537.1		X				
Perfluorooctanesulfonic acid (PFOS)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X				
Perfluorooctanesulfonic acid (PFOS)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17			X	X		
Perfluorooctanoic acid (PFOA) 335-67-1	EPA 1633 DRAFT 2			X	X		X
Perfluorooctanoic acid (PFOA) 335-67-1	EPA 533		X				
Perfluorooctanoic acid (PFOA) 335-67-1	EPA 537.1		X				
Perfluorooctanoic acid (PFOA) 335-67-1	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluoropentane sulfonic acid (PFPeS) 2706-91-4	EPA 1633 DRAFT 2			X	X		X
Perfluoropentane sulfonic acid (PFPES) 2706-91-4	EPA 533		X				
Perfluoropentane sulfonic acid (PFPES) 2706-91-4	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluoropentanoic acid (PFPeA) 2706-90-3	EPA 1633 DRAFT 2			X	X		X
Perfluoropentanoic acid (PFPEA) 2706-90-3	EPA 533		X				
Perfluoropentanoic acid (PFPEA) 2706-90-3	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluoropropanesulfonic acid (PFPrS)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorotetradecanoic acid (PFTA)	EPA 1633 DRAFT 2			X	X		
Perfluorotetradecanoic acid (PFTA)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorotetradecanoic acid (PFTDA) 376-06-7	EPA 1633 DRAFT 2						X
Perfluorotridecanoic acid (PFTrDA)	EPA 1633 DRAFT 2			X	X		
Perfluorotridecanoic acid (PFTrDA)	EPA 537.1		X				
Perfluorotridecanoic acid (PFTrDA)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluorotridecanoic acid (PFTrDA) 72629-94-8	EPA 1633 DRAFT 2			X	X		X
Perfluoroundecanoic acid (PFUnA) 2058-94-8	EPA 533		X				
Perfluoroundecanoic acid (PFUnA) 2058-94-8	EPA 537.1		X				

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Perfluoroundecanoic acid (PFUnA) 2058-94-8	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Perfluoroundecanoic acid (PFUnDA) 2058-94-8	EPA 1633 DRAFT 2						X
Perylene	EPA TO-13A Full Scan	X					
Perylene	EPA TO-13A SIM	X					
Phenanthrene	EPA 8270E			X	X		X
Phenanthrene	EPA 8270E-SIM			X	X		X
Phenanthrene	EPA TO-13A Full Scan	X					
Phenanthrene	EPA TO-13A SIM	X					
Phenol	EPA 8270E			X	X		
P.I.A.N.O.	EPA 8260D(m) [1]	X		X	X	X	
Potassium	EPA 200.7		X	X			
Potassium	EPA 200.8		X	X			
Potassium	EPA 6010D			X	X		
Potassium	EPA 6020B			X	X		X
Preconcentration Under Acid	EPA 3005A			X			
Propane	RSK-175 [1]			X			
Propene	RSK-175 [1]			X			
Propylene	EPA TO-15	X					
Propylene	EPA TO-15 SIM	X					
Propylene Glycol	EPA 8015D			X	X		
p-Xylene	EPA TO-15	X					
p-Xylene	EPA TO-15 SIM	X					
Pyrene	EPA 8270E			X	X		X
Pyrene	EPA 8270E-SIM			X	X		X
Pyrene	EPA TO-13A Full Scan	X					
Pyrene	EPA TO-13A SIM	X					
Pyridine	EPA 8270E			X	X		
sec-Butylbenzene	EPA TO-15	X					
sec-Butylbenzene	EPA TO-15 SIM	X					
Selenium	EPA 200.7			X			
Selenium	EPA 200.8		X	X			
Selenium	EPA 6010D			X	X		

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Selenium	EPA 6020B			X	X		X
Selenium	EPA 1632A(m) [1]			X	X		
Separatory Funnel Extraction	EPA 3510C			X			X
Silica Gel Cleanup	EPA 3630C			X	X		X
Silica, Dissolved	EPA 200.7			X			
Silica, Dissolved	EPA 6010D			X			
Silver	EPA 200.7		X	X			
Silver	EPA 200.8		X	X			
Silver	EPA 6010D			X	X		
Silver	EPA 6020B			X	X		X
Silver	AVS-SEM [1]				X		
Sodium	EPA 200.7		X	X			
Sodium	EPA 200.8		X	X			
Sodium	EPA 6010D			X	X		
Sodium	EPA 6020B			X	X		
Solid Phase Micro Extraction	ASTM D7363-13A [1]				X		
Specific Gravity	ASTM D854 [1]				X		
Strontium	EPA 200.7		X	X			
Strontium	EPA 6010D			X	X		
Strontium	EPA 6020B			X	X		X
Styrene	EPA TO-15	X					
Styrene	EPA TO-15 SIM	X					
Sulfide, Acid Volatile	AVS-SEM [1]				X		
Sulfur	EPA 6010D			X	X		
Sulfur Cleanup	EPA 3660B			X	X		
Sulfuric Acid Cleanup	EPA 3665A			X	X		
t-Amyl Alcohol	EPA 8015D			X	X		
Tert-Butyl Alcohol	EPA 8015D			X	X		
Tert-Butyl Alcohol	EPA TO-15	X					
Tert-Butyl Alcohol	EPA TO-15 SIM	X					
Tert-Butylbenzene	EPA TO-15	X					
Tert-Butylbenzene	EPA TO-15 SIM	X					

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Tetrachloroethene	EPA TO-15	X					
Tetrachloroethene	EPA TO-15 SIM	X					
Tetrafluoro-2(heptafluoropropoxy)propanoic acid (HFPO-DA)	EPA 1633 DRAFT 2			X	X		
Tetrafluoro-2(heptafluoropropoxy)propanoic acid (HFPO-DA)	PFAS Isotope Dilution Alpha SOP 23528 Rev 17		X	X	X		
Tetrahydrofuran	EPA TO-15	X					
Tetrahydrofuran	EPA TO-15 SIM	X					
Thallium	EPA 200.7			X			
Thallium	EPA 200.8		X	X			
Thallium	EPA 6010D		X	X			
Thallium	EPA 6020B			X	X		X
Tin	EPA 200.7		X	X			
Tin	EPA 6010D			X	X		
Tin	EPA 6020B			X	X		X
Titanium	EPA 200.7		X	X			
Titanium	EPA 6010D			X	X		
Toluene	EPA TO-15	X					
Toluene	EPA TO-15 SIM	X					
Total Hardness (CaCO3)	EPA 200.7		X	X			
Total Hardness (CaCO3)	EPA 6010D			X			
Total Hardness (CaCO3)	EPA 6020B			X			
Total Hardness (CaCO3)	SM 2340B			X			
Total Organic Carbon	EPA 9060A				X		
Total Organic Carbon	Lloyd Kahn				X		
Total Residue (% Total Solids)	SM 2540G				X		
Toxaphene	EPA 8081B			X	X		X
Trans-1,2-Dichloroethene	EPA TO-15	X					
Trans-1,2-Dichloroethene	EPA TO-15 SIM	X					
Trans-1,3-Dichloropropene	EPA TO-15	X					
Trans-1,3-Dichloropropene	EPA TO-15 SIM	X					
Trichloroethene	EPA TO-15	X					
Trichloroethene	EPA TO-15 SIM	X					
Trichlorofluoromethane	EPA TO-15	X					

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Parameter	Method	Matrices					
		Air	DW	NPW	SCM	Waste	Tissue
Trichlorofluoromethane	EPA TO-15 SIM	X					
Vanadium	EPA 200.7		X	X			
Vanadium	EPA 200.8		X	X			
Vanadium	EPA 6010D			X	X		
Vanadium	EPA 6020B			X	X		X
Vinyl acetate	EPA TO-15	X					
Vinyl acetate	EPA TO-15 SIM	X					
Vinyl Bromide	EPA TO-15	X					
Vinyl Bromide	EPA TO-15 SIM	X					
Vinyl Chloride	EPA TO-15	X					
Vinyl Chloride	EPA TO-15 SIM	X					
Waste Dilution	EPA 3580A			X	X	X	
Water Content	ASTM D2216-98 [1]				X	X	
Xylenes, Total	EPA TO-15 SIM	X					
Zinc	EPA 200.7		X	X			
Zinc	EPA 200.8		X	X			
Zinc	EPA 6010D			X	X		
Zinc	EPA 6020B			X	X		X
Zinc	AVS-SEM [1]				X		

[1] = Laboratory does not hold TNI Accreditation for this test method.

7.2.3 PAS-MANS 120 Forbes Blvd. Mansfield, MA

Parameter	Method	Matrices	
		Air	NPW
1,1,1-Trichloroethane	EPA TO-15	X	
1,1,1-Trichloroethane	EPA TO-15 SIM	X	
1,1,2,2-Tetrachloroethane	EPA TO-15	X	
1,1,2,2-Tetrachloroethane	EPA TO-15 SIM	X	
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	EPA TO-15	X	
1,1,2-Trichloro-1,2,2-Trifluoroethane (Freon 113)	EPA TO-15 SIM	X	
1,1,2-Trichloroethane	EPA TO-15	X	
1,1,2-Trichloroethane	EPA TO-15 SIM	X	
1,1-Dichloroethane	EPA TO-15	X	
1,1-Dichloroethane	EPA TO-15 SIM	X	

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Parameter	Method	Matrices	
		Air	NPW
1,1-Dichloroethene	EPA TO-15	X	
1,1-Dichloroethene	EPA TO-15 SIM	X	
1,2,4-Trichlorobenzene	EPA TO-15	X	
1,2,4-Trichlorobenzene	EPA TO-15 SIM	X	
1,2,4-Trimethylbenzene	EPA TO-15	X	
1,2,4-Trimethylbenzene	EPA TO-15 SIM	X	
1,2-Dibromo-3-Chloropropane (DBCP)	EPA TO-15	X	
1,2-Dibromo-3-Chloropropane (DBCP)	EPA TO-15 SIM	X	
1,2-Dibromoethane (EDB)	EPA TO-15	X	
1,2-Dibromoethane (EDB)	EPA TO-15 SIM	X	
1,2-Dichlorobenzene	EPA TO-15	X	
1,2-Dichlorobenzene	EPA TO-15 SIM	X	
1,2-Dichloroethane	EPA TO-15	X	
1,2-Dichloroethane	EPA TO-15 SIM	X	
1,2-Dichloropropane	EPA TO-15	X	
1,2-Dichloropropane	EPA TO-15 SIM	X	
1,2-Dichlorotetrafluoroethane	EPA TO-15	X	
1,2-Dichlorotetrafluoroethane	EPA TO-15 SIM	X	
1,3,5-Trimethylbenzene	EPA TO-15	X	
1,3,5-Trimethylbenzene	EPA TO-15 SIM	X	
1,3-Butadiene	EPA TO-15	X	
1,3-Butadiene	EPA TO-15 SIM	X	
1,3-Dichlorobenzene	EPA TO-15	X	
1,3-Dichlorobenzene	EPA TO-15 SIM	X	
1,4-Dichlorobenzene	EPA TO-15	X	
1,4-Dichlorobenzene	EPA TO-15 SIM	X	
1,4-Dioxane	EPA TO-15	X	
1,4-Dioxane	EPA TO-15 SIM	X	
2,2,4-Trimethylpentane	EPA TO-15	X	
2,2,4-Trimethylpentane	EPA TO-15 SIM	X	
2-Butanone (Methyl ethyl ketone, MEK)	EPA TO-15	X	
2-Butanone (Methyl ethyl ketone, MEK)	EPA TO-15 SIM	X	

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Parameter	Method	Matrices	
		Air	NPW
2-Chlorotoluene	EPA TO-15	X	
2-Chlorotoluene	EPA TO-15 SIM	X	
2-Hexanone	EPA TO-15	X	
2-Hexanone	EPA TO-15 SIM	X	
4-Ethyltoluene	EPA TO-15	X	
4-Ethyltoluene	EPA TO-15 SIM	X	
4-Methyl-2-pentanone (MIBK)	EPA TO-15	X	
4-Methyl-2-pentanone (MIBK)	EPA TO-15 SIM	X	
Acetaldehyde	EPA TO-15	X	
Acetaldehyde	EPA TO-15 SIM	X	
Acetone	EPA TO-15	X	
Acetone	EPA TO-15 SIM	X	
Acetonitrile	EPA TO-15	X	
Acetonitrile	EPA TO-15 SIM	X	
Acrolein	EPA TO-15	X	
Acrolein	EPA TO-15 SIM	X	
Acrylonitrile	EPA TO-15	X	
Acrylonitrile	EPA TO-15 SIM	X	
Allyl chloride	EPA TO-15	X	
Allyl chloride	EPA TO-15 SIM	X	
Benzene	EPA TO-15	X	
Benzene	EPA TO-15 SIM	X	
Benzyl Chloride	EPA TO-15	X	
Benzyl Chloride	EPA TO-15 SIM	X	
Bromodichloromethane	EPA TO-15	X	
Bromodichloromethane	EPA TO-15 SIM	X	
Bromoform	EPA TO-15	X	
Bromoform	EPA TO-15 SIM	X	
Bromomethane	EPA TO-15	X	
Bromomethane	EPA TO-15 SIM	X	
Carbon disulfide	EPA 3C	X	
Carbon disulfide	EPA TO-15	X	

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Parameter	Method	Matrices	
		Air	NPW
Carbon disulfide	EPA TO-15 SIM	X	
Carbon Tetrachloride	EPA TO-15	X	
Carbon Tetrachloride	EPA TO-15 SIM	X	
Chlorobenzene	EPA TO-15	X	
Chlorobenzene	EPA TO-15 SIM	X	
Chloroethane (Ethyl chloride)	EPA TO-15	X	
Chloroethane (Ethyl chloride)	EPA TO-15 SIM	X	
Chloroform	EPA TO-15	X	
Chloroform	EPA TO-15 SIM	X	
Chloromethane	EPA TO-15	X	
Chloromethane	EPA TO-15 SIM	X	
cis-1,2-Dichloroethene	EPA TO-15	X	
cis-1,2-Dichloroethene	EPA TO-15 SIM	X	
cis-1,3-Dichloropropene	EPA TO-15	X	
cis-1,3-Dichloropropene	EPA TO-15 SIM	X	
Cyclohexane	EPA TO-15	X	
Cyclohexane	EPA TO-15 SIM	X	
Dibromochloromethane	EPA TO-15	X	
Dibromochloromethane	EPA TO-15 SIM	X	
Dichlorodifluoromethane (Freon-12)	EPA TO-15	X	
Dichlorodifluoromethane (Freon-12)	EPA TO-15 SIM	X	
Dichlorofluoromethane	EPA TO-15	X	
Dichlorofluoromethane	EPA TO-15 SIM	X	
Ethanol	EPA TO-15	X	
Ethanol	EPA TO-15 SIM	X	
Ethyl acetate	EPA TO-15	X	
Ethyl acetate	EPA TO-15 SIM	X	
Ethylbenzene	EPA TO-15	X	
Ethylbenzene	EPA TO-15 SIM	X	
Hexachlorobutadiene	EPA TO-15	X	
Hexachlorobutadiene	EPA TO-15 SIM	X	
Isopropyl Alcohol	EPA TO-15	X	

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Parameter	Method	Matrices	
		Air	NPW
Isopropyl Alcohol	EPA TO-15 SIM	X	
Isopropylbenzene	EPA TO-15	X	
Isopropylbenzene	EPA TO-15 SIM	X	
Methane	EPA 3C	X	
Methyl Alcohol (methanol)	EPA TO-15	X	
Methyl Alcohol (methanol)	EPA TO-15 SIM	X	
Methyl methacrylate	EPA TO-15	X	
Methyl methacrylate	EPA TO-15 SIM	X	
Methyl tert-butyl ether (MTBE)	EPA TO-15	X	
Methyl tert-butyl ether (MTBE)	EPA TO-15 SIM	X	
Methylene Chloride	EPA TO-15	X	
Methylene Chloride	EPA TO-15 SIM	X	
m-Xylene	EPA TO-15	X	
m-Xylene	EPA TO-15 SIM	X	
Naphthalene	EPA TO-15	X	
Naphthalene	EPA TO-15 SIM	X	
n-Butylbenzene	EPA TO-15	X	
n-Butylbenzene	EPA TO-15 SIM	X	
n-Heptane	EPA TO-15	X	
n-Heptane	EPA TO-15 SIM	X	
n-Hexane	EPA TO-15	X	
n-Hexane	EPA TO-15 SIM	X	
Nitrogen	EPA 3C	X	
Non-Methane Organic Compounds	EPA TO-12	X	
n-Propylbenzene	EPA TO-15	X	
n-Propylbenzene	EPA TO-15 SIM	X	
Oxygen	EPA 3C	X	
o-Xylene	EPA TO-15	X	
o-Xylene	EPA TO-15 SIM	X	
Propylene	EPA TO-15	X	
Propylene	EPA TO-15 SIM	X	
p-Xylene	EPA TO-15	X	

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Parameter	Method	Matrices	
		Air	NPW
p-Xylene	EPA TO-15 SIM	X	
sec-Butylbenzene	EPA TO-15	X	
sec-Butylbenzene	EPA TO-15 SIM	X	
Styrene	EPA TO-15	X	
Styrene	EPA TO-15 SIM	X	
Tert-Butyl Alcohol	EPA TO-15	X	
Tert-Butyl Alcohol	EPA TO-15 SIM	X	
Tert-Butylbenzene	EPA TO-15	X	
Tert-Butylbenzene	EPA TO-15 SIM	X	
Tetrachloroethene	EPA TO-15	X	
Tetrachloroethene	EPA TO-15 SIM	X	
Tetrahydrofuran	EPA TO-15	X	
Tetrahydrofuran	EPA TO-15 SIM	X	
Toluene	EPA TO-15	X	
Toluene	EPA TO-15 SIM	X	
Trans-1,2-Dichloroethene	EPA TO-15	X	
Trans-1,2-Dichloroethene	EPA TO-15 SIM	X	
Trans-1,3-Dichloropropene	EPA TO-15	X	
Trans-1,3-Dichloropropene	EPA TO-15 SIM	X	
Trichloroethene	EPA TO-15	X	
Trichloroethene	EPA TO-15 SIM	X	
Trichlorofluoromethane	EPA TO-15	X	
Trichlorofluoromethane	EPA TO-15 SIM	X	
Vinyl acetate	EPA TO-15	X	
Vinyl acetate	EPA TO-15 SIM	X	
Vinyl Bromide	EPA TO-15	X	
Vinyl Bromide	EPA TO-15 SIM	X	
Vinyl Chloride	EPA TO-15	X	
Vinyl Chloride	EPA TO-15 SIM	X	
Xylenes, Total	EPA TO-15 SIM	X	

[1] = Laboratory does not hold TNI Accreditation for this test method.

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7.3 Appendix C: Glossary

This glossary provides common terms and definitions used in environmental testing; it is not intended to be a complete list of all terms and definitions used; some terms are defined in the context of the document or application. The definitions have been compiled mostly from the TNI Standard and DoD QSM. Although this information has been reproduced with care, errors cannot be entirely excluded.

Term	Definition
Acceptance Criteria	Specified limits placed on characteristics of an item, process, or service defined in requirement documents.
Accreditation	The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.
Accreditation Body	The organization having responsibility and accountability for environmental laboratory accreditation, and which grants accreditation.
Accuracy	The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; a data quality indicator.
Aliquot	A discrete, measured, representative portion of a sample taken for analysis.
Analysis	A combination of sample preparation and instrument determination.
Analysis Sequence	A compilation of all samples, standards and quality control samples run during a specific amount of time on a particular instrument in the order they are analyzed.
Analyte	A substance, organism, physical parameter, property, or chemical constituent(s) for which an environmental sample is being analyzed.
Analytical Method	A formal process that identifies and quantifies the chemical components of interest in a sample.
Analytical Uncertainty	A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.
Annual (or annually)	Defined by PAS as every 12 months ± 30 days.
Assessment	The evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its system to defined criteria (to the standards and requirements of laboratory accreditation).
Atomic Absorption Spectrometer	Instrument used to measure concentration in metals samples.
Audit	A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives.
Batch	Environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same quality systems matrix, meeting the above-mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours or the time specified by the regulatory program. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed 20 samples.
Bias	The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample's true value).
Blank	A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage, or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results (See Method Blank).

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Blind Sample	A sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.
Calibration	A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards.
Calibration Curve	The mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.
Calibration Method	A defined technical procedure for performing a calibration.
Calibration Range	The range of values (concentrations) between the lowest and highest calibration standards of a multi-level calibration curve.
Calibration Standard	A substance or reference material used for calibration.
Certified Reference Material	Reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute.
Chain of Custody	An unbroken trail of accountability that verifies the physical security of samples, data, and records.
Client /Customer	Any individual or organization external to Pace for whom items or services are furnished or work performed in response to defined requirements and expectations.
Code of Federal Regulations (CFR)	A codification of the general and permanent rules published in the Federal Register by agencies of the federal government.
Comparability	An assessment of the confidence with which one data set can be compared to another.
Completeness	The percentage of valid data obtained from a measurement system compared to the amount of valid data expected under normal conditions. This attribute is always calculated by the end data user, not by the laboratory.
Confirmation	Verification of the identity of a component through the use of an approach with a different scientific principle from the original method.
Continuing Calibration Blank (CCB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Continuing Calibration Check Compounds (CCC)	Compounds listed in mass spectrometry methods are used to evaluate an instrument calibration from the standpoint of the integrity of the system.
Continuing Calibration Verification (CCV) Standard	A standard used to verify the initial calibration of compounds in an analytical method. May be referred to as a CCC in some programs, such as UCMR.
Control Limit	A range within which specified measurement results must fall to verify that the analytical system is in control.
Correction	Action taken to eliminate a detected non-conformity.
Corrective Action	The action taken to eliminate the causes of an existing non-conformity, defect, or other undesirable situation in order to prevent recurrence.
Data Integrity	The condition that exists when data are sound, correct, and complete, and accurately reflect activities and requirements.
Data Quality Objective (DQO)	Strategic planning tool that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use or end user.
Data Reduction	The process of transforming the number of data items by arithmetic or statistical calculation, standard curves, and concentration factors, and collating them into a more usable form.
Demonstration of Capability (DOC)	A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision.
Detection Limit (DL)	The minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration is distinguishable from the method blank results.
Dry Weight	The percent of sample that is not water, which may be used to adjust the reported results.

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Electronic Data Deliverable (EDD)	A summary of environmental data which clients request for ease of data review and comparison to historical results.
Environmental Protection Agency (EPA)	An agency of the federal government of the United States which was created for the purpose of protecting human health and the environment by writing and enforcing regulations based on laws passed by Congress.
Equipment Blank	A sample of analyte-free media used to rinse common sampling equipment to check effectiveness of decontamination procedures.
Extracted Internal Standard Analyte	Isotopically labeled analogs of analytes of interest added to all standards, blanks and samples analyzed. Added to samples and batch QC samples prior to the first step of sample extraction and to standards and instrument blanks prior to analysis. Used for isotope dilution methods.
False Negative	A result that fails to identify (detect) an analyte to be present.
False Positive	A result that erroneously identifies (detects) an analyte to be present.
Field Blank	A blank sample prepared in the field by filling a clean container with reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken.
Field Measurement	Testing performed in the field outside of a fixed-laboratory or outside of an enclosed structure that meets the requirements of a mobile laboratory.
Flame Atomic Absorption Spectrometer (FAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the fact that ground state metals absorb light at different wavelengths. Metals in a solution are converted to the atomic state by use of a flame.
Flame Ionization Detector (FID)	A type of gas detector used in GC analysis where samples are passed through a flame which ionizes the sample so that various ions can be measured.
Gas Chromatography (GC)	Instrumentation which utilizes a mobile carrier gas to deliver an environmental sample across a stationary phase with the intent to separate compounds out and measure their retention times.
Gas Chromatograph/ Mass Spectrometry (GC/MS)	In conjunction with a GC, this instrumentation utilizes a mass spectrometer which measures fragments of compounds and determines their identity by their fragmentation patterns (mass spectra).
Gasoline Range Organics (GRO)	A range of compounds that denote all the characteristic compounds that make up gasoline (range can be state or program specific).
Graphite Furnace Atomic Absorption Spectrometry (GFAA)	Instrumentation used to measure the concentration of metals in an environmental sample based on the absorption of light at different wavelengths that are characteristic of different analytes.
High Pressure Liquid Chromatography (HPLC)	Instrumentation used to separate, identify, and quantitate compounds based on retention times which are dependent on interactions between a mobile phase and a stationary phase.
Holding Time	The maximum time specified in a reference method that can elapse between analytical activities.
Homogeneity	The degree to which a property or substance is uniformly distributed throughout a sample.
Incremental Sampling Method (ISM)	A sampling method designed to provide an accurate characterization of contamination in soils at a site through collection of numerous portions of soil that are combined, processed, and subsampled in order to reduce the effects of soil heterogeneity.
Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES)	Analytical technique used for the detection of trace metals which uses plasma to produce excited atoms that emit radiation of characteristic wavelengths.
Inductively Coupled Plasma- Mass Spectrometry (ICP/MS)	An ICP that is used in conjunction with a mass spectrometer so that the instrument is not only capable of detecting trace amounts of metals and non-metals but is also capable of monitoring isotopic speciation for the ions of choice.
Infrared Spectrometer (IR)	An instrument that uses infrared light to identify compounds of interest.

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Initial Calibration (ICAL)	The process of analyzing standards, prepared at specified concentrations, to define the quantitative response relationship of the instrument to the analytes of interest.
Initial Calibration Blank (ICB)	A blank sample used to monitor the cleanliness of an analytical system at a frequency determined by the analytical method.
Initial Calibration Verification (ICV)	A standard obtained or prepared from a source independent of the source of the initial calibration standards to verify the initial calibration.
Instrument Blank	An analyte free matrix processed through the instrumental steps of the measurement process; use to determine instrument contamination.
Interference	The suppression or elevation of result of an analyte of interest due to the presence of other compounds.
Internal Standard	A known amount of standard added to a test portion of a sample as a reference for evaluating and controlling the precision and bias of the applied analytical method.
International Organization for Standardization (ISO)	An international standard-setting body composed of representatives from various national standards organizations.
Intermediate Standard Solution	Reference solutions prepared by dilution of the stock solutions with an appropriate solvent.
International System of Units (SI)	The coherent system of units adopted and recommended by the General Conference on Weights and Measures.
Ion Chromatography (IC)	Instrumentation or process that allows the separation of ions and molecules based on the charge properties of the molecules.
Isomer	One of two or more compounds, radicals, or ions that contain the same number of atoms of the same element but differ in structural arrangement and properties. For example, hexane (C6H14) could be n-hexane, 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutane.
Laboratory	A facility that performs testing, whether in-house or in the field.
Laboratory Control Sample (LCS)	Also known as laboratory fortified blank (LFB): A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, which is taken through all sample preparation and analytical steps of the procedure.
Laboratory Information Management System (LIMS)	The entirety of an electronic data system (including hardware and software) that collects, analyzes, stores, and archives electronic records and documents.
Legal Chain-of-Custody Protocols	Industry defined protocol that is sometimes used for evidentiary or legal purposes that establishes an intact, continuous record of the physical possession, storage, and disposal of samples, including sample aliquots and prepared samples.
Limit(s) of Detection (LOD)	The smallest concentration of a substance that must be present in a sample in order to be detected at the DL with 99% confidence.
Limit(s) of Quantitation (LOQ)	The minimum level, concentration, or quantity of a target analyte that can be reported with a specified degree of confidence.
Lower Limit of Quantitation (LLOQ)	The concentration of the lowest calibration standard included in the calibration curve.
Linear Dynamic Range	Concentration range where the instrument provides a linear response.
Liquid chromatography/ tandem mass spectrometry (LC/MS/MS)	Instrumentation that combines the physical separation techniques of liquid chromatography with the mass analysis capabilities of mass spectrometry.
Lot	A definite amount of material produced during a single manufacturing cycle and intended to have uniform character and quality.
Matrix	The substrate of a test sample.

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Matrix Duplicate	A replicate matrix prepared in the laboratory and analyzed to obtain a measure of precision.
Matrix Spike (MS)	A sample prepared, taken through all sample preparation and analytical steps of the procedure, to which a known amount of target analyte is added to a specified amount of sample for which an independent test result of target analyte concentration is available.
Matrix Spike Duplicate (MSD)	A replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.
Measurement System	A method, as implemented at a particular laboratory, and which includes the equipment used to perform the test and the operator(s).
Measurement Uncertainty	An estimate of the error in a measurement often stated as a range of values that contain the true value within a certain confidence level.
Method	A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification).
Method Blank	A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures.
Method Detection Limit (MDL)	The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.
Method of Standard Additions	A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects.
Minimum Detectable Activity (MDA)	Estimate of the smallest true activity that ensures a specified high confidence, $1 - \beta$, of detection above the Critical Value, and a low probability β of false negatives below the Critical Value. For radiometric methods, β is often set at 0.05.
Negative Control	A sample included in the method that is treated the same as all others but is expected to have a detection of analytes of interest.
Nonconformance	An indication or judgment that a product or service has not met the requirement of the relevant specifications, contract, or regulation; also, the state of failing to meet the requirements.
Positive Control	A sample included in the method that is treated the same as all others and is expected to have a detection of the analytes of interest.
Practical Quantitation Limit (PQL)	Another term for a method reporting limit.
Precision	The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.
Preservation	Any conditions under which a sample must be kept in order to maintain chemical, physical, and/or biological integrity prior to analysis.
Primary Accreditation Body (Primary AB)	The TNI-approved state agency responsible for assessing a laboratory's total quality system, on-site assessment, and PT performance tracking for fields of accreditation.
Procedure	Written instructions detailing the performance of a task, process, or other laboratory activity.
Proficiency Testing (PT)	A means to evaluate a laboratory's performance under controlled conditions relative to a given set of criteria, through analysis of unknown samples provided by an external source.
Proficiency Testing Reporting Limit (PTRL)	A statistically derived value that represents the lowest acceptable concentration for an analyte in a PT sample, if the analyte is spiked into the PT sample. The PTRLs are specified in the TNI FoPT tables.
Qualitative Analysis	Analysis designed to identify the components of a substance or mixture.
Quality Assurance (QA)	An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.
Quality Assurance Project Plan (QAPP)	A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved.

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Quality Control (QC)	The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out of control" conditions and ensuring that the results are of acceptable quality.
Quality Control Sample (QCS)	A sample used to assess the performance the measurement system.
Quality Manual	A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.
Quality Management System	A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required quality assurance and quality control activities.
Quantitation Range	The upper and lower limit to which values can be reported without being qualified as estimated.
Quantitative Analysis	Analysis designed to determine the amounts or proportions of the components of a substance.
Raw Data	The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records.
Record	Documentation, data, or other records constituting a piece of evidence about the past, especially an account of an act or occurrence kept in writing or some other permanent form. Records may be in electronic or hardcopy media. Unless otherwise specified, the term "record" refers to both electronic and hardcopy media.
Reference Material	Material or substance one or more of whose property values are sufficiently homogenized and well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.
Reference Method	A published method issued by an organization recognized as competent to do so. (When the ISO language refers to a "standard method," that term is equivalent to "reference method").
Reference Standard	Standard used for the calibration of working measurement standards in a given organization or at a given location.
Relative Percent Difference (RPD)	A measure of precision defined as the difference between two measurements divided by the average concentration of the two measurements.
Reporting Limit (RL)	The value to which test results are reported as detected or not detected.
Representativeness	A quality element related to the ability to collect a sample reflecting the characteristics of the part of the environment to be assessed.
Retention Time	The time between sample injection and the appearance of a solute peak at the detector.
Sample	The item submitted for testing.
Sampling	Activity related to obtaining a representative sample for analysis.
Selected Ion Monitoring (SIM)	A mode of analysis in mass spectrometry where the detector is set to scan over a very small mass range, typically one mass unit.
Selectivity	The ability of a method to correctly determine an analyte without interference from other compounds.
Sensitivity	The smallest amount of substance in a sample that can accurately be measured.
Serial Dilution	The stepwise dilution of a substance in a solution.
Standard Operating Procedure (SOP)	A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps.
Standard Reference Material (SRM)	A certified reference material produced by the US NIST or other equivalent organization and characterized for absolute content, independent of analytical method.

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Stock Standard	A concentrated reference solution containing one or more analytes prepared in the laboratory using an assayed reference compound or purchased from a reputable commercial source.
Storage Blank	A sample of analyte-free media prepared by the laboratory and retained in the sample storage area.
Suitability	The quality of being appropriate for a particular purpose.
Surrogate	A substance with properties that mimic the analyte of interest, and unlikely to be found in environmental samples.
Systems Audit	An on-site inspection or assessment of a laboratory's quality system.
Target Analytes	Analytes or chemicals of primary concern identified by the customer on a project-specific basis.
Technology	A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
Traceability	The ability to trace the history of measuring equipment to national or international standards, primary standards, basic physical conditions or properties, or reference materials.
Trip Blank	A cleaned sample container filled with laboratory reagent water that is stored, shipped, and analyzed with its associated samples to detect sample contamination during transport and storage of the sample.
Tuning	A check and/or adjustment of instrument performance for mass spectrometry as required by the method.
Validation	The confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled.
Verification	Confirmation by examination and objective evidence that specified requirements have been met. In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation, or specification peculiar to the management of the measuring equipment.
Working Standard	Dilution made of a stock or intermediate standard used in the analytical process.

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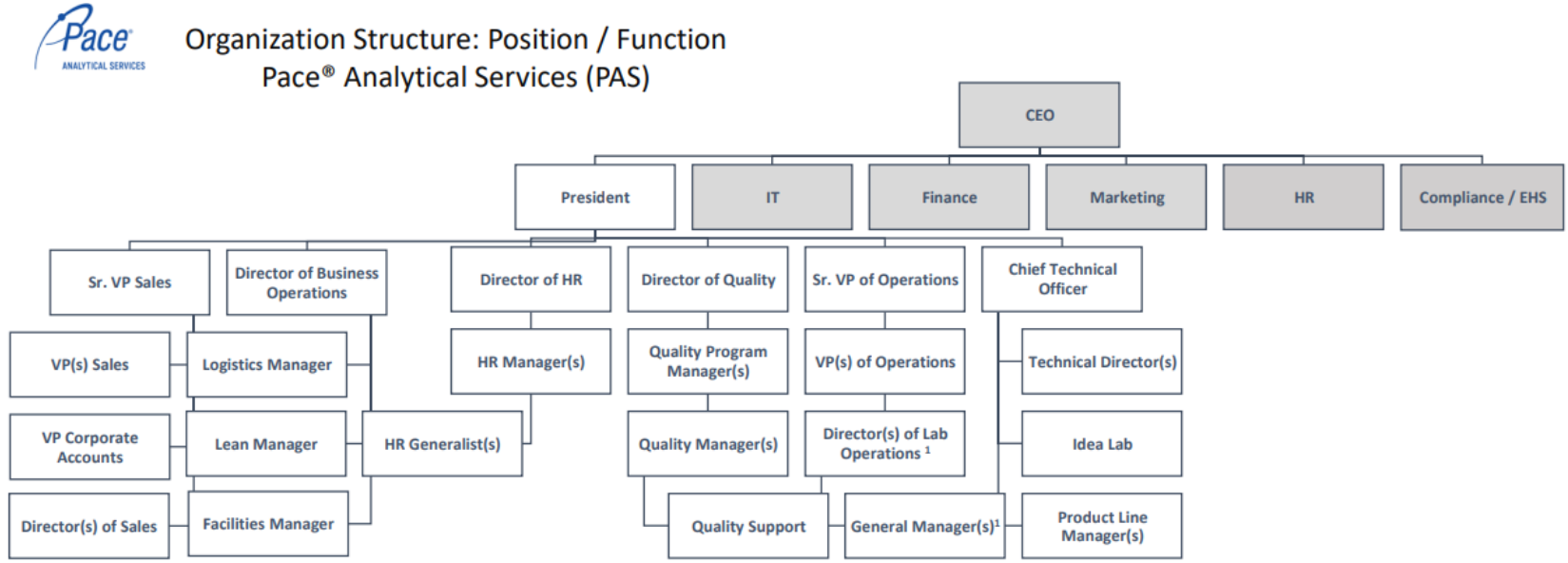
7.4 Appendix D: Organization Chart(s)

7.4.1 PAS Corporate Organization Chart(s)

The information in this organization chart is subject to change. Please contact PAS for the most up-to-date version.



White Box = PAS Positions / Functions
Grey Box = Pace®Corporate Positions / Functions
1= Positions not Assigned to all Locations – see location specific organization charts

Effective 12.18.23
Information is subject to change, please contact PAS for most up-to-date version.

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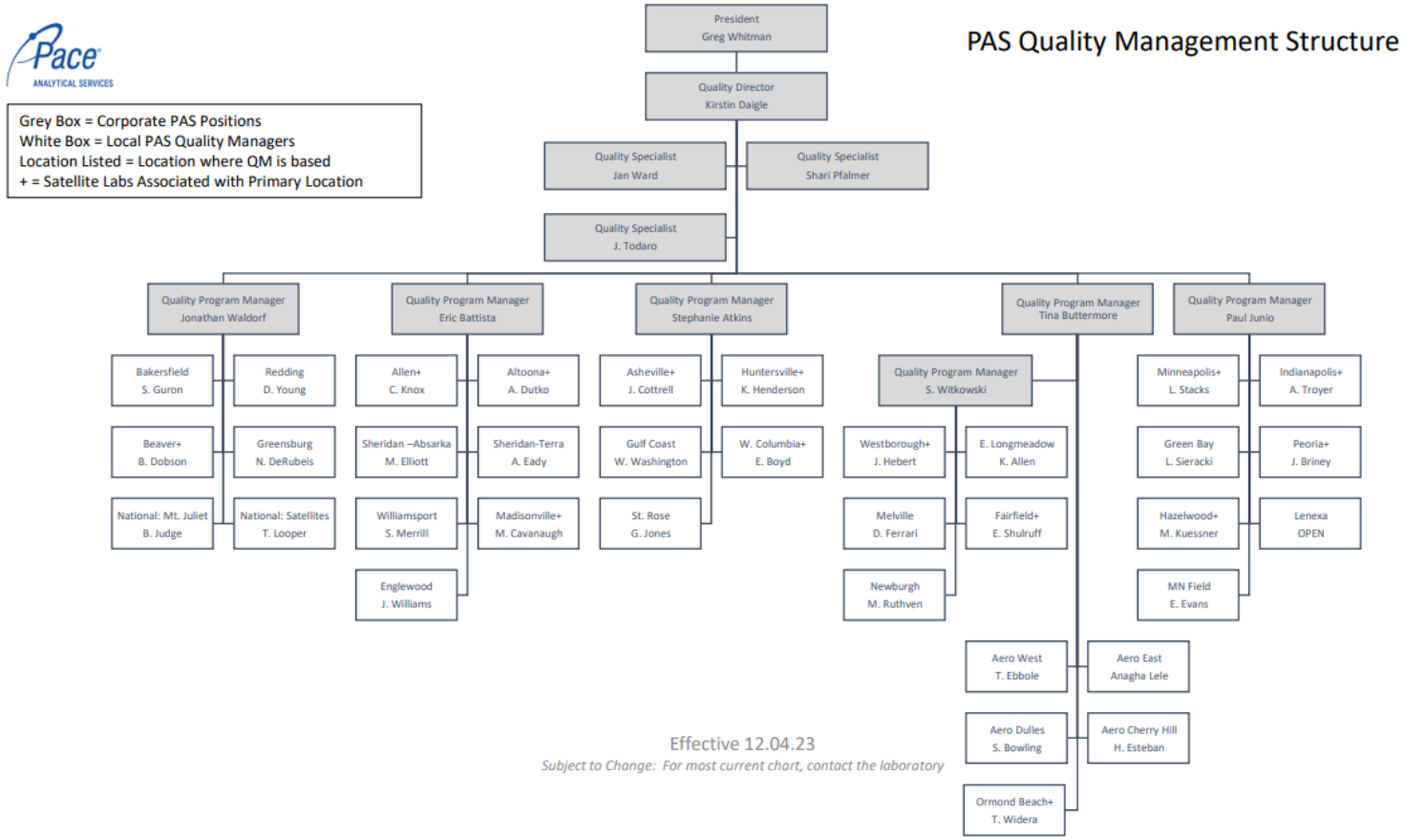
7.4.2 PAS Quality Systems Management Organization Chart

The information in this organization chart is subject to change. Please contact PAS for the most up-to-date version.



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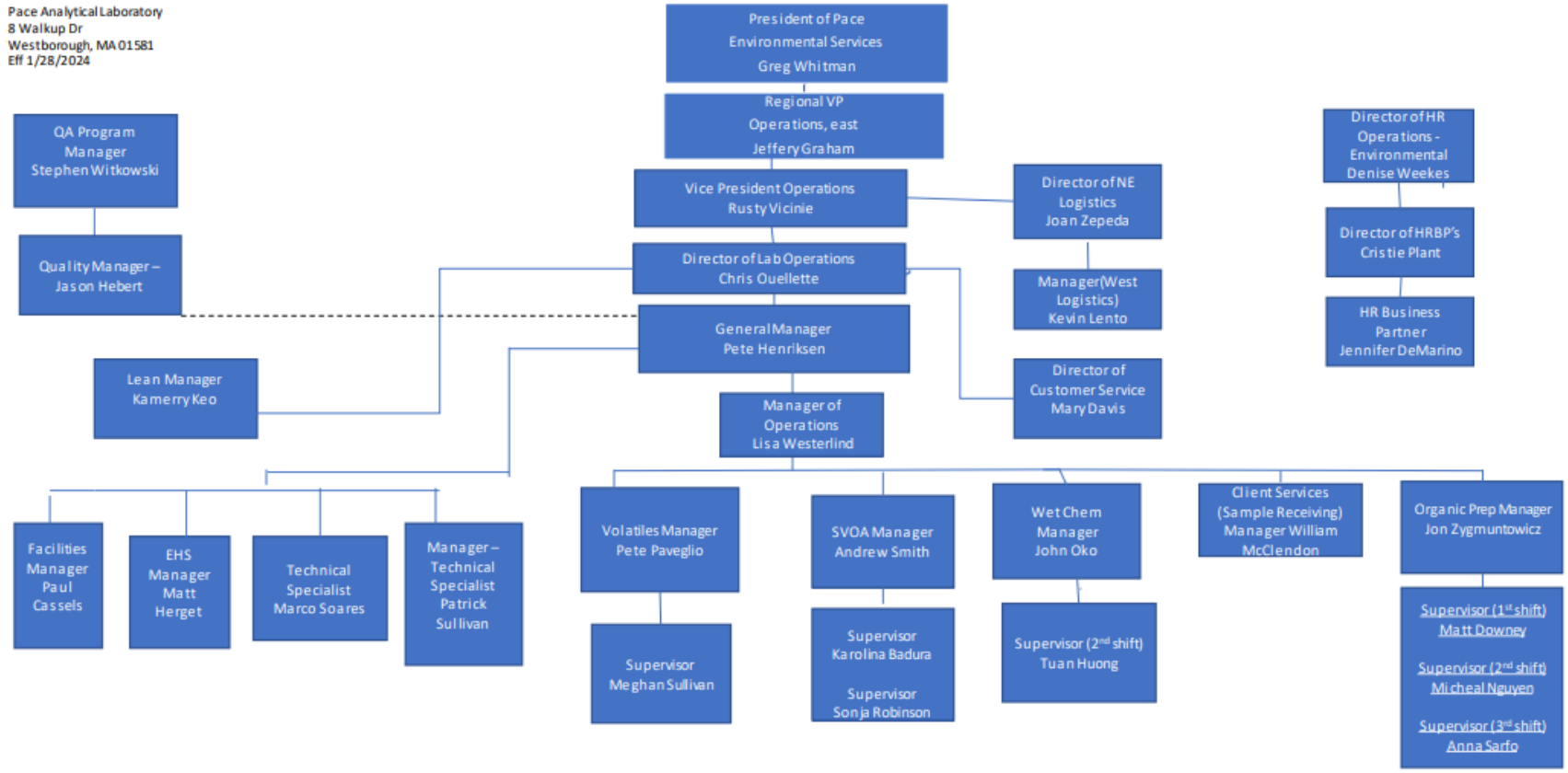
7.4.3 PAS-WES2 – Organization Chart

The information in this organization chart is subject to change. Please contact PAS for the most up-to-date version.



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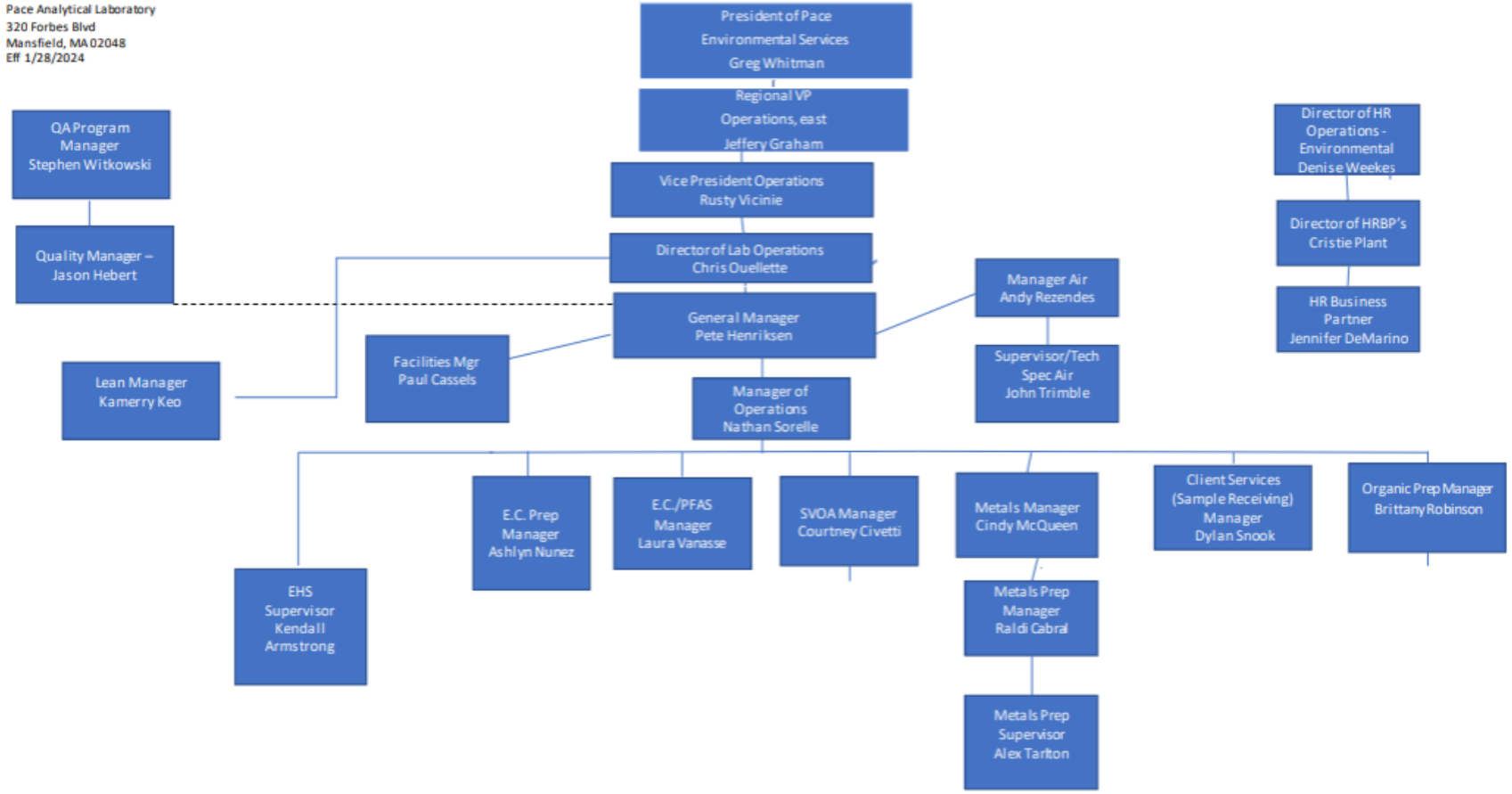
7.4.4 PAS-MANS 320 Forbes Blvd, Mansfield, MA – Organization Chart

The information in this organization chart is subject to change. Please contact PAS for the most up-to-date version



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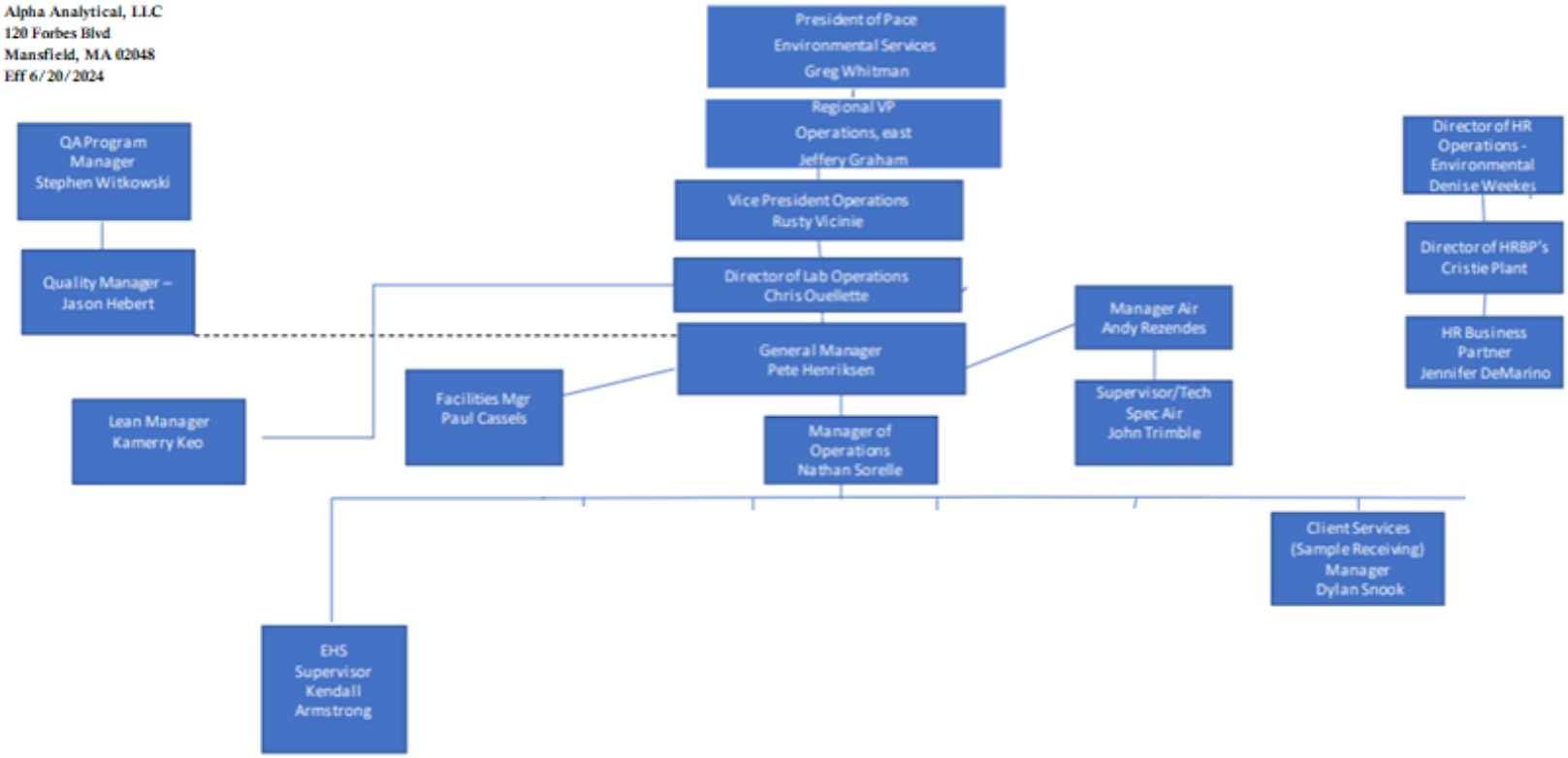
7.4.5 PAS-MANS 120 Forbes Blvd. Mansfield, MA – Organization Chart



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7.5 Appendix E: Equipment Listing

The equipment listed represents equipment were held by each location on the effective date of this manual. This information is subject to change without notice. External parties should contact the location for the most current information.

7.5.1 PAS-WES2

Equipment List: PAS-WES2 8 Walkup Drive, Westborough, MA

Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Adsorption Module	Mitsubishi Chemical Analytech	TXA-04	D9D00104	5/1/2015	New	WB Wet Chemistry	2281	QA Backup/Team
Ammonia Distillation Unit	Buchi	K-355	10000323097	12/19/2018	New	WB Wet Chemistry	3496	QA Backup/Team
Anemometer	Control Company	33500-096	130078984	8/9/2013	New	WB Wet Chemistry	2004	QA Backup/Team
Auto Boat Controller	Mitsubishi Chemical Analytech	ABC-201	D8CA0054	3/1/2015	New	WB Wet Chemistry	2280	QA Backup/Team
Auto Sampler	EST Analytical	Centurion	CENT371060 314	9/1/2015	UNK	WestVOA	2308	QA Backup/Team
Autoclave	Harvey	ST75925	1.28E+12	6/25/1905	UNK	WB Microbiology	734	QA Backup/Team
Autoclave	Tuttnauer	2340E	9612771	1/1/2001	UNK	WB Microbiology	2011	QA Backup/Team
Autoclave	Tuttnauer	3870EA	1202281	3/28/2016	Used	WB Wet Chemistry	2391	QA Backup/Team
Automated IS Dispener	Pace Ideas Lab	Viper	1	6/1/2023	New	WB SVOA	4846	QA Backup/Team
Autosampler	Agilent	7693	CN17020085	UNK	UNK	WB SVOA	3070	QA Backup/Team
Autosampler	Agilent	7693A	CN19340010	8/27/2019	New	WB SVOA	3714	QA Backup/Team
Autosampler	Agilent	G2614A	CN85252207	UNK	UNK	WB SVOA	1082	QA Backup/Team
Autosampler	Agilent	G2614A	US83301526	UNK	UNK	WB SVOA	1071	QA Backup/Team
Autosampler	Agilent	G2614A	US90403246	UNK	UNK	WB SVOA	1080	QA Backup/Team
Autosampler	Agilent	G2614A	CN20920296	UNK	UNK	WB SVOA	1351	QA Backup/Team
Autosampler	Agilent	G2614A	CN34927347	UNK	UNK	WB SVOA	1355	QA Backup/Team
Autosampler	Agilent	G2614A	US12211768	UNK	UNK	WB SVOA	1060	QA Backup/Team
Autosampler	Agilent	G2614A	US12712062	UNK	UNK	WB SVOA	1101	QA Backup/Team
Autosampler	Agilent	G2614A	CN85352302	UNK	UNK	WB SVOA	1107	QA Backup/Team
Autosampler	Agilent	G2614A	CN42229080	UNK	UNK	WB SVOA	1089	QA Backup/Team
Autosampler	Agilent	G2614A	CN72244178	UNK	UNK	WB SVOA	2034	QA Backup/Team
Autosampler	Agilent	G2614A	CN71543674	UNK	UNK	WB SVOA	1086	QA Backup/Team
Autosampler	Agilent	G2614A	US92605580	UNK	UNK	WB SVOA	1509	QA Backup/Team
Autosampler	Agilent	G2614A	US14213136	UNK	UNK	WB SVOA	1111	QA Backup/Team
Autosampler	Agilent	G2614A	CN51132503	UNK	UNK	WB SVOA	1114	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Autosampler	Agilent	G2614A	CN71743823	UNK	UNK	WB SVOA	839	QA Backup/Team
Autosampler	Agilent	G2614A	CN22020701	UNK	UNK	WB SVOA	1369	QA Backup/Team
Autosampler	Agilent	G2614A	CN80146891	UNK	UNK	WB SVOA	1137	QA Backup/Team
Autosampler	Agilent	G2614A	CN34626993	UNK	UNK	WB SVOA	3781	QA Backup/Team
Autosampler	Agilent	G4514A	CN12420010	UNK	UNK	WB SVOA	3780	QA Backup/Team
Autosampler	Agilent	G4514A	CN10130029	UNK	UNK	WB SVOA	1405	QA Backup/Team
Autosampler	Agilent	G4514A	CN15060082	UNK	UNK	WB SVOA	2313	QA Backup/Team
Autosampler	Agilent	G4514A	CN13150014	UNK	UNK	WB SVOA	2132	QA Backup/Team
Autosampler	Agilent	G4514A	CN15320105	UNK	UNK	WB SVOA	3073	QA Backup/Team
Autosampler	Agilent	G4514A	CN17020081	UNK	UNK	WB SVOA	3216	QA Backup/Team
Autosampler	Agilent	G4514A	CN18170257	UNK	New	WB SVOA	3416	QA Backup/Team
Autosampler	Agilent	G4514A	CN17020085	UNK	UNK	WB SVOA	3212	QA Backup/Team
Autosampler	Agilent	G4514A	CN10210007	UNK	UNK	WB SVOA	1136	QA Backup/Team
Autosampler	Agilent	G4514A	CN10130053	UNK	UNK	WB SVOA	1359	QA Backup/Team
Autosampler	Agilent	G4514A	CN10340019	UNK	UNK	WB SVOA	1143	QA Backup/Team
Autosampler	Agilent	G4514A	CN10130053	UNK	UNK	WB SVOA	1410	QA Backup/Team
Autosampler	Agilent	G4514A	CN10230053	UNK	UNK	WB SVOA	1124	QA Backup/Team
Autosampler	Agilent	G4514A	CN13440032	UNK	UNK	WB SVOA	2089	QA Backup/Team
Autosampler	Agilent	G4514A	CN18430060	UNK	UNK	WB SVOA	3782	QA Backup/Team
Autosampler	Agilent	G4514A	cn15490113	UNK	New	WB SVOA	3102	QA Backup/Team
Autosampler	Agilent	G4514A	cn16150057	UNK	New	WB SVOA	3111	QA Backup/Team
Autosampler	Agilent	G4514A	CN10470031	UNK	UNK	WB SVOA	1075	QA Backup/Team
Autosampler	Agilent	G4514A	CN10260029	UNK	UNK	WB SVOA	1378	QA Backup/Team
Autosampler	Agilent	G4514A	CN10200045	UNK	UNK	WB SVOA	1447	QA Backup/Team
Autosampler	Agilent	G4514A	CN10210005	UNK	UNK	WB SVOA	1419	QA Backup/Team
Autosampler	Agilent	G4514A	CN13430014	UNK	UNK	WB SVOA	2086	QA Backup/Team
Autosampler	Agilent	G4514A	CN13430061	UNK	UNK	WB SVOA	2104	QA Backup/Team
Autosampler	Agilent	G4514A	CN16150002	UNK	UNK	WB SVOA	3106	QA Backup/Team
Autosampler	Agilent	G4514A	CN10380042	UNK	UNK	WB SVOA	1123	QA Backup/Team
Autosampler	Agilent	G4514A	cn16150002	UNK	New	WB SVOA	3106	QA Backup/Team
Autosampler	Agilent	G4567A	CN18520009	UNK	UNK	WB SVOA	3700	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Autosampler	EST Analytical	CENT w/s	cents417070915	UNK	New	WestVOA	3065	QA Backup/Team
AutoSampler	EST Analytical	CENT w/s	CENTS556041018	4/1/2018	New	WestVOA	3378	QA Backup/Team
Autosampler	EST Analytical	Cent WS	CENTS144031610	UNK	UNK	WestVOA	1029	QA Backup/Team
Autosampler	EST Analytical	Cent WS	CENTS270060112	UNK	New	WestVOA	3757	QA Backup/Team
Autosampler	EST Analytical	Cent WS	CENTS140531810	UNK	UNK	WestVOA	1020	QA Backup/Team
Autosampler	EST Analytical	Cent WS	CENTS237071411	UNK	UNK	WestVOA	1036	QA Backup/Team
Autosampler	EST Analytical	Cent WS	CENTS246100411	UNK	New	WestVOA	3753	QA Backup/Team
Autosampler	EST Analytical	Cent WS	CENTS505051217	UNK	New	WestVOA	3755	QA Backup/Team
Autosampler	EST Analytical	Cent WS	CENTS156051410	UNK	UNK	WestVOA	1007	QA Backup/Team
Autosampler	EST Analytical	Cent WS	CENTS370060314	UNK	New	WestVOA	3771	QA Backup/Team
Autosampler	EST Analytical	Cent WS	CENTS359031014	UNK	New	WestVOA	3758	QA Backup/Team
Autosampler	EST Analytical	Cent WS	CENTS330101713	UNK	New	WestVOA	3754	QA Backup/Team
Autosampler	EST Analytical	Cent WS	CENTS3490122140	UNK	UNK	WestVOA	2047	QA Backup/Team
Autosampler	EST Analytical	Cent WS	CENTS605020419	UNK	New	WestVOA	3770	QA Backup/Team
Autosampler	EST Analytical	Cent WS	CENTS276071712	UNK	New	WestVOA	3772	QA Backup/Team
Autosampler	EST Analytical	Centurion	CENT219110606	UNK	New	WestVOA	3756	QA Backup/Team
Autosampler	EST Analytical	Centurion	4.16E+11	UNK	New	WestVOA	3359	QA Backup/Team
Autosampler	EST Analytical	Centurion	CENT239072409	UNK	UNK	WestVOA	845	QA Backup/Team
Autosampler	EST Analytical	Centurion	CENTS220113006	UNK	UNK	WestVOA	821	QA Backup/Team
Autosampler	EST Analytical	Centurion WS	CENTS367050514	6/1/2014	UNK	WestVOA	2093	QA Backup/Team
AutoSampler	EST Analytical	EST Centurion	CENT480010317	1/1/2017	New	WestVOA	3079	QA Backup/Team
Autosampler	HP	18596B	3215A2800	UNK	UNK	WB SVOA	1442	QA Backup/Team
Autosampler	HP	G2614A	US81900879	UNK	UNK	WB SVOA	1128	QA Backup/Team
Autosampler	Tekmar	ASX-7200HR	121951A7200HR	12/1/2019	New	WestVOA	3802	QA Backup/Team
Autosampler	Teledyne Tekmar	Aquatek LVA	US21208005	9/1/2021	New	WestVOA	4273	QA Backup/Team
AutoSampler	THERMO	AS-DV PN#068888	170512365	UNK	New	WB Wet Chemistry	3353	QA Backup/Team
Autosampler (Lachat)	Hach	ASX-560	101952A560	12/27/2019	New	WB Wet Chemistry	3775	QA Backup/Team
Autosampler Controller	Agilent	7673	US71801886	UNK	UNK	WB SVOA	1065	QA Backup/Team
AutoSampler Pump	Mantech	PC-1000-75	MT-1H3-267	10/22/2013	New	WB Wet Chemistry	2257	QA Backup/Team
Autosampler Tray	Agilent	G4514A	CN14100106	8/18/2014	New	WB SVOA	2123	QA Backup/Team
Autosampler/Purge&Trap	Tekmar	Atomx XYZ	US20014001	12/1/2019	New	WestVOA	3794	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Autosampler/Purge&Trap	Teledyne Tekmar	Atomx XYZ	US18065019	5/8/2018	New	WestVOA	3773	QA Backup/Team
Autosampler/Purge&Trap	Teledyne Tekmar	Atomx XYZ	US21222017	9/1/2021	New	WestVOA	4277	QA Backup/Team
Auto-shaker	Glas-Col	099A VS5504T	407598	N/A	New	WB Organic Prep	986	QA Backup/Team
Auto-shaker	Glas-Col	099A VS5504T	407597	N/A	New	WB Organic Prep	3408	QA Backup/Team
Auto-shaker	Glas-Col	099A VS5504T	407596	N/A	New	WB Organic Prep	3407	QA Backup/Team
Auto-shaker	Glas-Col	099A VS5504T	11347043	N/A	New	WB Organic Prep	3386	QA Backup/Team
Balance	A&D	HR120	12330253	8/12/2011	New	WB Microbiology	1001	QA Backup/Team
Balance	AND	GF-2000	UNK	UNK	New	WB Organic Prep	3061	QA Backup/Team
Balance	AnD	GF2002A	T2105461	1/7/2021	New	WB Login	4040	QA Backup/Team
Balance	AND	GF-2002A	UNK	UNK	New	WB Organic Prep	4289	QA Backup/Team
Balance	AND	GF-2002A	UNK	UNK	New	WB Organic Prep	4851	QA Backup/Team
Balance	AND	GF-2002A	UNK	UNK	New	WB Organic Prep	4857	QA Backup/Team
Balance	AND	GF-2002AN	UNK	UNK	New	WB Organic Prep	4856	QA Backup/Team
Balance	Mettler Toledo	30133522	C130219766	9/3/2021	New	WB Wet Chemistry	4267	QA Backup/Team
Balance	Mettler Toledo	30133522	C130219767	9/3/2021	New	WB Wet Chemistry	4268	QA Backup/Team
Balance	Mettler Toledo	AE 200	H23588	UNK	UNK	WB SVOA	242	QA Backup/Team
Balance	Mettler Toledo	AX205	1125430398	11/2/2004	UNK	WB Wet Chemistry	732	QA Backup/Team
Balance	Mettler Toledo	ML104	B145441300	2/28/2012	New	WB Login	1798	QA Backup/Team
Balance	Mettler Toledo	MS104TS/00	1100496882	UNK	New	WB Wet Chemistry	3707	QA Backup/Team
Balance	Mettler Toledo	MS104TS/00	b923778218	UNK	New	WB Wet Chemistry	3708	QA Backup/Team
Balance	Mettler Toledo	UNK	UNK	UNK	Used	WB Organic Prep	4637	QA Backup/Team
Balance	Ohaus	EP214	J26211263419 52	9/19/2005	Used	WB Microbiology	2003	QA Backup/Team
Balance	Ohaus	EX223	BO33261336	7/1/2012	UNK	WestVOA	1871	QA Backup/Team
Balance	Ohaus	SP-202	B424698330	10/23/2014	UNK	WestVOA	2392	QA Backup/Team
Balance	VWR	1002E	12132021032	12/1/2022	New	WB Wet Chemistry	4833	QA Backup/Team
Balance	VWR	VWR-123P	6232017223	UNK	New	WB Wet Chemistry	3383	QA Backup/Team
Balance	VWR	VWR-500P	12252015398	6/2/2016	New	WB Wet Chemistry	3094	QA Backup/Team
Balance	VWR	VWR-500P	6152015336	3/20/2018	New	WestVOA	3363	QA Backup/Team
Balance	VWR	VWR-500P Balance	7282022110	6/15/2023	New	WB Login	4423	QA Backup/Team
Barometer	Control Company	15551-024	130739731	UNK	UNK	WB Wet Chemistry	2266	QA Backup/Team
Barometer	VWR	10510-922	170391456	UNK	UNK	WB Wet Chemistry	3286	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
BOD Analyzer	SEAL	ML1200	8694	8/22/2024	New	WB Microbiology	4714	QA Backup/Team
BOD Auto Sampler	Mantech	automax122	261H3N227	10/22/2013	New	WB Wet Chemistry	2255	QA Backup/Team
BOD Incubator	Fisher	ISO-Temp	94167062	UNK	UNK	WB Microbiology	481	QA Backup/Team
BOD Incubator	Sheldon	SRI20P	8046419	9/11/2019	New	WB Microbiology	3716	QA Backup/Team
BOD Incubator	VWR	2020	300795	4/24/1995	UNK	WB Microbiology	80	QA Backup/Team
BOD Incubator	VWR	414004-632	0411302125M008	4/1/2013	New	WB Microbiology	1933	QA Backup/Team
BOD Pump	ManSci	PC-1000-443	MT-1K6-220	UNK	UNK	WB Microbiology	QA Backup/Team	
BOD Pump	ManSci	PC-1000-443	MT-1K6-220	4/17/2015	New	WB Microbiology	3185	QA Backup/Team
BOD Pump	ManSci	PC-1000-443	MT-1H3-177	7/28/2014	New	WB Microbiology	3186	QA Backup/Team
Chiller	Buchi	F-108	997813	10/6/2013	New	WB Organic Prep	2026	QA Backup/Team
Chiller	Buchi	F-108	997613	10/10/2013	New	WB Organic Prep	3736	QA Backup/Team
Chiller	Buchi	F-108	10006176655	8/13/2014	New	WB Organic Prep	2127	QA Backup/Team
Chiller	Buchi	F-108	1000172485	8/13/2014	New	WB Organic Prep	3737	QA Backup/Team
Chiller	Buchi	F-108	1000200054	6/19/2015	New	WB Organic Prep	2293	QA Backup/Team
Chiller	Buchi	F-308	1636816	1/1/2019	New	WB Organic Prep	3690	QA Backup/Team
Chiller	Neslab	M-33	103357029	UNK	UNK	WB Wet Chemistry	1185	QA Backup/Team
Chiller	Neslab	M75	102128080	UNK	UNK	WB Wet Chemistry	1225	QA Backup/Team
Chiller	VWR	1172	614617	UNK	UNK	WB Wet Chemistry	1211	QA Backup/Team
Chiller	VWR	1177MD	107700434	UNK	UNK	WB Wet Chemistry	1230	QA Backup/Team
Chiller	VWR	1177PD	5L12A0727	10/19/2012	New	WB Organic Prep	3735	QA Backup/Team
Chiller	WKL	1000	LWG47304-0012	UNK	UNK	WB Wet Chemistry	1193	QA Backup/Team
Chiller	Buchi	F-108	UNK	UNK	UNK	WB Organic Prep	4713	QA Backup/Team
Chiller	Buchi	F-108	UNK	UNK	UNK	WB Organic Prep	4718	QA Backup/Team
Chiller	Buchi	F-108	UNK	UNK	UNK	WB Organic Prep	4427	QA Backup/Team
Chiller	VWR	1177PD	UNK	UNK	UNK	WB Organic Prep	3733	QA Backup/Team
Chiller	VWR	1177PD	6U1551245	UNK	UNK	WB Organic Prep	QA Backup/Team	
Chromatography Oven	Dionex	LC30	4120631	2/2/2005	UNK	WestVOA	747	QA Backup/Team
Cl Titrimeter-Amperometric	HACH	19299	9.81E+12	6/20/1905	UNK	WB Wet Chemistry	579	QA Backup/Team
COD Reactor	Environmental Express	B3000	2018CODW127	12/10/2018	New	WB Wet Chemistry	3491	QA Backup/Team
COD Reactor	HACH	45600	910404502	UNK	UNK	WB Microbiology	1227	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
COD Reactor	HACH	DRB-200	115261	Fall, 2006	UNK	WB Microbiology	807	QA Backup/Team
COD Reactor	HACH	LTG082	14100C0075	6/17/2015	New	WB Wet Chemistry	2298	QA Backup/Team
Colony Counter	Quebec	3330	UNK	UNK	UNK	WB Microbiology	1261	QA Backup/Team
Concentrator	Buchi	Q-101	1000176503	6/19/2015	New	WB Organic Prep	3388	QA Backup/Team
Concentrator	Buchi	Q-101	100170193	7/6/1905	New	WB Organic Prep	2002	QA Backup/Team
Concentrator	Buchi	Q-101	1000176504	7/6/1905	New	WB Organic Prep	3785	QA Backup/Team
Concentrator	Buchi	Q-101	1000149181	10/1/2013	New	WB Organic Prep	3387	QA Backup/Team
Concentrator	Buchi	Q-101	1000151471	10/9/2013	New	WB Organic Prep	2029	QA Backup/Team
Concentrator	Buchi	Q-101	1000153955	10/5/2013	New	WB Organic Prep	2025	QA Backup/Team
Concentrator	Buchi	Q-101	1000176507	8/13/2014	New	WB Organic Prep	3409	QA Backup/Team
Concentrator	Buchi	Q-101	1000176500	8/13/2014	New	WB Organic Prep	2128	QA Backup/Team
Concentrator	Buchi	Q-101	1000180892	1/1/2019	New	WB Organic Prep	3498	QA Backup/Team
Concentrator	EST Analytical	Encon	529112106	UNK	New	WestVOA	3764	QA Backup/Team
Concentrator	EST Analytical	Encon	559091207E	9/1/2007	UNK	WestVOA	846	QA Backup/Team
Concentrator	EST Analytical	Encon	560091207E	9/1/2007	UNK	WestVOA	847	QA Backup/Team
Concentrator	EST Analytical	ENCON	581013108	UNK	UNK	WestVOA	1035	QA Backup/Team
Concentrator	EST Analytical	Encon Evolution	EV24032410	UNK	UNK	WestVOA	1028	QA Backup/Team
Concentrator	EST Analytical	Encon Evolution	EV701092115	UNK	New	WestVOA	3778	QA Backup/Team
Concentrator	EST Analytical	Encon Evolution	EV375082911	UNK	UNK	WestVOA	3798	QA Backup/Team
Concentrator	EST Analytical	Encon Evolution	EV584060314	UNK	New	WestVOA	3765	QA Backup/Team
Concentrator	EST Analytical	Encon Evolution	EV218082009	UNK	UNK	WestVOA	1048	QA Backup/Team
Concentrator	EST Analytical	Encon Evolution	EV373080211	UNK	New	WestVOA	3761	QA Backup/Team
Concentrator	EST Analytical	Encon Evolution	EV250251410	UNK	UNK	WestVOA	1008	QA Backup/Team
Concentrator	EST Analytical	Encon Evolution	EV245040910	UNK	UNK	WestVOA	1021	QA Backup/Team
Concentrator	EST Analytical	Encon Evolution	EV429073112	UNK	New	WestVOA	3766	QA Backup/Team
Concentrator	EST Analytical	ENCON Evolution	EV516101713	10/1/2013	UNK	WestVOA	2035	QA Backup/Team
Concentrator	EST Analytical	Encon Evolution	EV737032416	UNK	New	WestVOA	3762	QA Backup/Team
Concentrator	EST Analytical	Encon Evolution	EV736032416	UNK	New	WestVOA	3763	QA Backup/Team
Concentrator	EST Analytical	Encon Evolution	EVX1038020419	UNK	UNK	WestVOA	3774	QA Backup/Team
Concentrator	EST Analytical	EV	EV952041018	4/1/2018	New	WestVOA	3377	QA Backup/Team
Conductivity Cell	Dionex	CD20	4120779	2/2/2005	UNK	WB SVOA	744	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Conductivity Meter	Corning	441	1262	11/1/1998	UNK	WB Wet Chemistry	471	QA Backup/Team
Conductivity Meter	Hanna Instruments	HI 9033	UNK	6/1/1996	UNK	WB Wet Chemistry	340	QA Backup/Team
Conductivity Meter		HI 99300	H0028302	UNK	New	WB Wet Chemistry	3084	QA Backup/Team
Controller	Buchi	V-855	1000129622	10/3/2013	New	WB Organic Prep	2023	QA Backup/Team
Controller	Buchi	V-855	1000144338	10/7/2013	New	WB Organic Prep	2027	QA Backup/Team
Controller	Buchi	V-855	1000144331	10/11/2013	New	WB Organic Prep	2031	QA Backup/Team
Controller	Buchi	V-855	1000171223	8/13/2014	New	WB Organic Prep	2125	QA Backup/Team
Controller	Buchi	V-855	1000174547	8/13/2014	New	WB Organic Prep	2129	QA Backup/Team
Controller	Buchi	V-855	1000202566	6/19/2015	New	WB Organic Prep	2292	QA Backup/Team
Controller	Horizon	3000	71403	UNK	UNK	WB Wet Chemistry	1209	QA Backup/Team
Controller	Horizon	1000/3000XL	1002	UNK	UNK	WB Wet Chemistry	1210	QA Backup/Team
Controller	Horizon	SPE-DEX 3000	71398	UNK	UNK	WB Wet Chemistry	1323	QA Backup/Team
Controller	V&P Scientific Inc	KBMB	710332	8/14/2012	New	WB Wet Chemistry	1926	QA Backup/Team
Controller (PID)	OI Analytical	4430	C142530284	UNK	UNK	WestVOA	3768	QA Backup/Team
Controller (PID)	OI Analytical	4430	UNK	UNK	UNK	WestVOA	1037	QA Backup/Team
Controller (PID)	OI Analytical	4430	C234530245	UNK	UNK	WestVOA	3769	QA Backup/Team
Controller (PID)	OI Analytical	4430	UNK	UNK	UNK	WestVOA	1034	QA Backup/Team
Datalogger	Dickson	DWE	16462703	3/20/2022	New	WB Login	4581	QA Backup/Team
Datalogger	Dickson	DWE	16456788	3/20/2022	New	WB Login	4582	QA Backup/Team
Datalogger	Dickson	DWE	16456796	3/20/2022	New	WB Login	4583	QA Backup/Team
Datalogger	Dickson	DWE	16461534	3/20/2022	New	WB Organic Prep	4584	QA Backup/Team
Datalogger	Dickson	DWE	16461470	3/20/2022	New	WB Organic Prep	4585	QA Backup/Team
Datalogger	Dickson	DWE	16456784	3/20/2022	New	WB Organic Prep	4586	QA Backup/Team
Datalogger	Dickson	DWE	16461447	3/20/2022	New	WB Organic Prep	4587	QA Backup/Team
Datalogger	Dickson	DWE	16461383	3/20/2022	New	WB Organic Prep	4588	QA Backup/Team
Datalogger	Dickson	DWE	16462549	3/20/2022	New	WB Organic Prep	4589	QA Backup/Team
Datalogger	Dickson	DWE	16461446	3/20/2022	New	WB Wet Chemistry	4611	QA Backup/Team
Datalogger	Dickson	DWE	16461405	3/20/2022	New	WB Wet Chemistry	4612	QA Backup/Team
Datalogger	Dickson	DWE	16462516	3/20/2022	New	WB Wet Chemistry	4613	QA Backup/Team
Datalogger	Dickson	DWE	16461546	3/20/2022	New	WB Wet Chemistry	4614	QA Backup/Team
Datalogger	Dickson	DWE	16456759	3/20/2022	New	WB Wet Chemistry	4615	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Datalogger	Dickson	DWE	16462550	3/20/2022	New	WB Wet Chemistry	4616	QA Backup/Team
Datalogger	Dickson	DWE	16462443	3/20/2022	New	WB Wet Chemistry	4617	QA Backup/Team
Datalogger	Dickson	DWE	16456792	3/20/2022	New	WB Wet Chemistry	4618	QA Backup/Team
Datalogger	Dickson	DWE	16462437	3/20/2022	New	WB Wet Chemistry	4619	QA Backup/Team
Datalogger	Dickson	DWE	16462558	3/20/2022	New	WB Wet Chemistry	4620	QA Backup/Team
Datalogger	Dickson	DWE	16462767	3/20/2022	New	WB Wet Chemistry	4621	QA Backup/Team
Datalogger	Dickson	DWE	16462770	3/20/2022	New	WB Wet Chemistry	4607	QA Backup/Team
Datalogger	Dickson	DWE	16462494	3/20/2022	New	WB Wet Chemistry	4622	QA Backup/Team
Datalogger	Dickson	DWE	16462440	3/20/2022	New	WB Wet Chemistry	4623	QA Backup/Team
Datalogger	Dickson	DWE	16462455	3/20/2022	New	WB Wet Chemistry	4624	QA Backup/Team
Datalogger	Dickson	DWE	16462612	3/20/2022	New	WB Wet Chemistry	4625	QA Backup/Team
Datalogger	Dickson	DWE	16462585	3/20/2022	New	WB Wet Chemistry	4631	QA Backup/Team
Datalogger	Dickson	DWE	16461388	3/20/2022	New	WB Wet Chemistry	4610	QA Backup/Team
Datalogger	Dickson	DWE	16456797	3/20/2022	New	WB Wet Chemistry	4609	QA Backup/Team
Datalogger	Dickson	DWE	16461541	3/20/2022	New	WB Wet Chemistry	4608	QA Backup/Team
Datalogger	Dickson	DWE	16456784	3/20/2022	New	WB Wet Chemistry	4627	QA Backup/Team
Datalogger	Dickson	DWE	16462602	3/20/2022	New	WB Wet Chemistry	4634	QA Backup/Team
Datalogger	Dickson	DWE	16461398	3/20/2022	New	WB Wet Chemistry	4633	QA Backup/Team
Datalogger	Dickson	DWE	16462541	3/20/2022	New	WB Wet Chemistry	4632	QA Backup/Team
Datalogger	Dickson	DWE	16461522	3/20/2022	New	WB Wet Chemistry	4626	QA Backup/Team
Datalogger	Dickson	DWE	16461424	3/20/2022	New	WB SVOA	4591	QA Backup/Team
Datalogger	Dickson	DWE	16461464	3/20/2022	New	WB SVOA	4592	QA Backup/Team
Datalogger	Dickson	DWE	16462534	3/20/2022	New	WB SVOA	4593	QA Backup/Team
Datalogger	Dickson	DWE	16461483	3/20/2022	New	WB SVOA	4594	QA Backup/Team
Datalogger	Dickson	DWE	16461509	3/20/2022	New	WestVOA	4595	QA Backup/Team
Datalogger	Dickson	DWE	16461497	3/20/2022	New	WestVOA	4596	QA Backup/Team
Datalogger	Dickson	DWE	16462595	3/20/2022	New	WestVOA	4597	QA Backup/Team
Datalogger	Dickson	DWE	16462717	3/20/2022	New	WestVOA	4598	QA Backup/Team
Datalogger	Dickson	DWE	16461495	3/20/2022	New	WestVOA	4599	QA Backup/Team
Datalogger	Dickson	TWE	16374979	3/20/2022	New	WB Login	4601	QA Backup/Team
Datalogger	Dickson	TWE	16374958	3/20/2022	New	WB Login	4602	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Datalogger	Dickson	TWE	16375067	3/20/2022	New	WB Organic Prep	4603	QA Backup/Team
Datalogger	Dickson	TWE	16374353	3/20/2022	New	WB Organic Prep	4590	QA Backup/Team
Datalogger	Dickson	TWE	16375259	3/20/2022	New	WestVOA	4604	QA Backup/Team
Datalogger	Dickson	TWE	16375268	3/20/2022	New	WestVOA	4606	QA Backup/Team
Datalogger	Dickson	TWE	16374897	3/20/2022	New	WestVOA	4605	QA Backup/Team
Dessicator	Boekel	Sm Stainless	None	UNK	UNK	WB Wet Chemistry	1215	QA Backup/Team
Dessicator	Boekel	Sm Stainless	None	UNK	UNK	WB Wet Chemistry	1215	QA Backup/Team
Dessicator	Fisher	Old Stainless	None	UNK	UNK	WB Wet Chemistry	1164	QA Backup/Team
Dessicator	Fisher	Old Stainless	None	UNK	UNK	WB Wet Chemistry	1165	QA Backup/Team
Dessicator	Fisher	Sm Stainless	None	N/A	N/A	WB Organic Prep	1246	QA Backup/Team
Dessicator	Sanplatec	DryKeeper	None	N/A	N/A	WB Organic Prep	1248	QA Backup/Team
Dessicator	Sanplatec	DryKeeper	None	UNK	UNK	WB Wet Chemistry	1206	QA Backup/Team
Dessicator	Terra Universal	1911-26A	1911-26A11885811	UNK	UNK	WB Wet Chemistry	1161	QA Backup/Team
Digestion Block	Environmental Express	SC151	UNK	1/1/2019	New	WB Wet Chemistry	3724	QA Backup/Team
Diluter	Lachat	2900-000	2000-361	2/1/1997	UNK	WB Wet Chemistry	401	QA Backup/Team
Discrete Analyzer	Seal	AQ2	90856	2/19/2013	New	WB Wet Chemistry	1932	QA Backup/Team
Discrete Analyzer	SEAL	AQ400	341247	UNK	New	WB Wet Chemistry	4290	QA Backup/Team
Dishwasher	Labconco	4420431	191084550G	12/30/2019	New	WB Organic Prep	3747	QA Backup/Team
Dishwasher	Labconco	4420431	191287365G	2/5/2020	New	WB Organic Prep	3814	QA Backup/Team
Dishwasher	Miele	G7783	12/53084579	UNK	UNK	WB Wet Chemistry	1194	QA Backup/Team
Dishwasher	Miele	G7783	44/074359852	7/2/2012	UNK	WB Wet Chemistry	1927	QA Backup/Team
Dishwasher	UNK	UNK	UNK	UNK	UNK	WB Wet Chemistry	1200	QA Backup/Team
Distillation Block	Environmental Express	EMD1920-106	2245	12/21/2024	New	WB Wet Chemistry	3948	QA Backup/Team
Distillation Block	Environmental Express	EMD1920-107	2244	12/21/2024	New	WB Wet Chemistry	3949	QA Backup/Team
Distillation System MIDI	Andrews	110-10-R	A1V10062	UNK	UNK	WB Wet Chemistry	1185	QA Backup/Team
Distillation System MIDI	Andrews	110-10-R	A6R0209	10/6/1996	UNK	WB Wet Chemistry	1187	QA Backup/Team
Distillation System MIDI	Andrews	110-10-R	Unknown	UNK	UNK	WB Wet Chemistry	1195	QA Backup/Team
Distillation System MIDI	Andrews	110-10-R	Unknown	UNK	UNK	WB Wet Chemistry	1196	QA Backup/Team
Distillation System MIDI	UNK	Unknown	Unknown	1996	UNK	WB Wet Chemistry	1188	QA Backup/Team
Distillation Unit	Environmental Express	7100-1320-107	2033	6/23/1905	UNK	WB Wet Chemistry	694	QA Backup/Team
Distillation Unit	Environmental Express	emd1920-106	2395	1/3/2020	New	WB Wet Chemistry	3730	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Distillation Unit	Environmental Express	EMD-1920-106	2308	2/4/2013	New	WB Wet Chemistry	1925	QA Backup/Team
Distillation Unit	Environmental Express	EMD-1920-106	2309	2/4/2013	New	WB Wet Chemistry	1924	QA Backup/Team
Distillation Unit	Environmental Express	UNK	9378	1/1/2019	New	WB Wet Chemistry	3726	QA Backup/Team
Distillation Unit	Environmental Express	UNK	2384	1/1/2109	New	WB Wet Chemistry	3727	QA Backup/Team
Distillation Unit	Foss	2100	520026801	UNK	UNK	WB Wet Chemistry	1179	QA Backup/Team
Distillation Unit	Glastron	emd1920-106	2331	6/1/2014	UNK	WB Wet Chemistry	2143	QA Backup/Team
Distillation Unit	Kjeltec	8100	91712470	10/11/2011	UNK	WB Wet Chemistry	1922	QA Backup/Team
Distillation Unit	Kjeltec	2200	44021	12/1/1997	UNK	WB Wet Chemistry	399	QA Backup/Team
DO Meter	YSI	5000	97F0722AC	UNK	UNK	WB Microbiology	669	QA Backup/Team
DO Meter	YSI	5100-115	13D100734	10/22/2013	New	WB Wet Chemistry	2256	QA Backup/Team
Eluent Generator	Dionex	EG50	5010177	2/2/2005	UNK	WB SVOA	746	QA Backup/Team
Flash Tester	Koehler	K16200	R07002918B	1/27/2011	UNK	WB Wet Chemistry	1930	QA Backup/Team
Florisil System	Supelco	None	None	N/A	N/A	WB Organic Prep	999	QA Backup/Team
Florisil System	Supelco	None	None	N/A	N/A	WB Organic Prep	996	QA Backup/Team
Fluke Thermometer	Fluke	52 II	57130039MV	10/25/2022	New	West QA	4834	WEST QA
Fluoride Meter	Hanna	HI5222	F0022641	3/27/2017	New	WB Wet Chemistry	3128	QA Backup/Team
Freezer	Beverage Air	HF2-1S	11803668	9/29/2017	UNK	WB Login	3294	QA Backup/Team
Freezer	Beverage Air	HF2-1S	13005852	7/16/2019	New	WB Login	3710	QA Backup/Team
Freezer	Beverage Air	HF2-1S	11803668	UNK	New	WestVOA	3294	QA Backup/Team
Freezer	Electrolux Home Products Inc	FGCH25M8LW1	WB32046880	UNK	Used	WB Login	4262	QA Backup/Team
Freezer	Horizon	AFV-20	VWR-WB83460777-1810	10/23/2018	New	WB Login	3083	QA Backup/Team
Freezer	Kenmore	235.2104241	WB3758093	UNK	New	WB Login	3731	QA Backup/Team
Freezer	Kenmore	253.2104241	WB62354772	7/18/2016	New	WestVOA	3099	QA Backup/Team
Freezer	Kenmore	Unknown	Unknown	UNK	Used	WestVOA	2069	QA Backup/Team
Freezer	NorthLAke Scientific	LF141WWW/0HC	NLS-8A10336552-2105	5/26/221	New	WestVOA	4049	QA Backup/Team
Freezer	Sears	253.2104241	WB61755744	61/16	New	WestVOA	3096	QA Backup/Team
Freezer	ThermoScientific	ULT430A	1.12E+15	10/31/2019	New	WestVOA	3722	QA Backup/Team
Freezer	VWR	AFV-14	VWR-WB85059039-1901	1/8/2019	New	WestVOA	3495	QA Backup/Team
Freezer	VWR	AFV-17	VWR-WB72553667-1710	New	WB SVOA	3296	QA Backup/Team	

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Freezer	VWR	HCUCFS-0420	VWR-041905161153-1908	11/15/2019	New	WB Login	3721	QA Backup/Team
Freezer	TRUE	T-23F	1-3454087	UNK	UNK	WestVOA	1058	QA Backup/Team
Gas Chromatograph	Agilent	8890	US2125A063	9/1/2021	New	WestVOA	4271	QA Backup/Team
Gas Chromatograph	Agilent	8890	US2125A060	9/1/2021	New	WestVOA	4275	QA Backup/Team
Gas Chromatograph	Agilent	8890	US2150A036	12/21/2024	New	WestVOA	4286	QA Backup/Team
GC	Agilent	6890	US10203098	8/1/2004	UNK	WB SVOA	1488	QA Backup/Team
GC	Agilent	6890	CN10439073	11/1/2004	UNK	WestVOA	716	QA Backup/Team
GC	Agilent	6890	US00029138	UNK	UNK	WestVOA	1102	QA Backup/Team
GC	Agilent	6890	US000034651	6/1/2003	UNK	WestVOA	710	QA Backup/Team
GC	Agilent	7890	CN11351196	9/1/2011	UnUsed	WestVOA	2015	QA Backup/Team
GC	Agilent	5977B	US18523035	UNK	New	WB SVOA	3701	QA Backup/Team
GC	Agilent	5977B	US1715R024	4/1/2017	New	WestVOA	3268	QA Backup/Team
GC	Agilent	6890N	US10203095	2-Mar	UNK	WB SVOA	677	QA Backup/Team
GC	Agilent	6890N	CN10525090	5-Sep	UNK	WB SVOA	837	QA Backup/Team
GC	Agilent	6890N	US10128056	1-Jul	UNK	WB SVOA	663	QA Backup/Team
GC	Agilent	6890N	US10137101	UNK	UNK	WB SVOA	1068	QA Backup/Team
GC	Agilent	6890N	CN10625123	UNK	UNK	WB SVOA	1084	QA Backup/Team
GC	Agilent	6890N	US 10641076	4/1/2007	UNK	WB SVOA	824	QA Backup/Team
GC	Agilent	6890N	CN10413083	4-May	UNK	WB SVOA	718	QA Backup/Team
GC	Agilent	6890N	CN10444076	3/15/2005	UNK	WestVOA	737	QA Backup/Team
GC	Agilent	6890N	CN10433067	11/1/2004	UNK	WestVOA	714	QA Backup/Team
GC	Agilent	6890N	CN10733012	9/1/2007	UNK	WestVOA	844	QA Backup/Team
GC	Agilent	6890N	US10143049	1/1/2003	UNK	WestVOA	693	QA Backup/Team
GC	Agilent	6890N (G1530N)	CN10413009	4-Feb	UNK	WB SVOA	750	QA Backup/Team
GC	Agilent	7890A	CN10842068	UNK	UNK	WB SVOA	1121	QA Backup/Team
GC	Agilent	7890A	CN10715071	8/1/2013	Used	WB SVOA	2006	QA Backup/Team
GC	Agilent	7890A	CN13411007	10/2/2013	New	WB SVOA	2033	QA Backup/Team
GC	Agilent	7890A	CN12471113	2014	UNK	WB SVOA	2334	QA Backup/Team
GC	Agilent	7890A	CN10021057	8/2/2010	New	WB SVOA	1133	QA Backup/Team
GC	Agilent	7890A	CN10261101	UNK	UNK	WB SVOA	1127	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
GC	Agilent	7890A	CN10803048	8-May	UNK	WB SVOA	852	QA Backup/Team
GC	Agilent	7890A	US10828037	UNK	UNK	WB SVOA	1140	QA Backup/Team
GC	Agilent	7890A	CN10181023	UNK	UNK	WB SVOA	1352	QA Backup/Team
GC	Agilent	7890A	CN10847125	6/5/2010	Used	WB SVOA	1407	QA Backup/Team
GC	Agilent	7890A	CN10511068	UNK	UNK	WB SVOA	1073	QA Backup/Team
GC	Agilent	7890A	CN10341144	10/1/2012	UNK	WB SVOA	1375	QA Backup/Team
GC	Agilent	7890A	CN10221049	UNK	UNK	WB SVOA	1444	QA Backup/Team
GC	Agilent	7890A	CN10031123	UNK	UNK	WB SVOA	1416	QA Backup/Team
GC	Agilent	7890A	CB10804039	4/1/2008	UNK	WestVOA	834	QA Backup/Team
GC	Agilent	7890A	CN10031129	UNK	UNK	WestVOA	1026	QA Backup/Team
GC	Agilent	7890A	CN11311129	UNK	UNK	WestVOA	1033	QA Backup/Team
GC	Agilent	7890A	CN10181119	1/1/2011	Used	WestVOA	1047	QA Backup/Team
GC	Agilent	7890A	CN10051030	Spring 2010	UNK	WestVOA	1006	QA Backup/Team
GC	Agilent	7890A	CN10843110	UNK	UNK	WestVOA	1018	QA Backup/Team
GC	Agilent	7890A	CN10521070	UNK	Used	WestVOA	1390	QA Backup/Team
GC	Agilent	7890A	CN10391176	UNK	UNK	WestVOA	1356	QA Backup/Team
GC	Agilent	7890A	CN10141099	5/1/2010	New	WestVOA	1402	QA Backup/Team
GC	Agilent	7890B	CN13443026	5/14/2014	UNK	WB SVOA	2088	QA Backup/Team
GC	Agilent	7890B	CN15483113	UNK	New	WB SVOA	3071	QA Backup/Team
GC	Agilent	7890B	CN17123159	UNK	New	WB SVOA	3218	QA Backup/Team
GC	Agilent	7890B	CN18233173	UNK	New	WB SVOA	3418	QA Backup/Team
GC	Agilent	7890B	US19363002	8/27/2019	New	WB SVOA	3713	QA Backup/Team
GC	Agilent	7890B	CN15283138	UNK	UNK	WB SVOA	2312	QA Backup/Team
GC	Agilent	7890B	CN15443075	UNK	UNK	WB SVOA	3068	QA Backup/Team
GC	Agilent	7890B	CN15483138	UNK	New	WB SVOA	3702	QA Backup/Team
GC	Agilent	7890B	CN13523172	5/1/2014	UNK	WB SVOA	2084	QA Backup/Team
GC	Agilent	7890B	CN13503187	7/1/2014	UNK	WB SVOA	2102	QA Backup/Team
GC	Agilent	7890B	CN18023166	UNK	UNK	WB SVOA	3698	QA Backup/Team
GC	Agilent	7890B	US19523001	19-Dec	New	WB SVOA	3789	QA Backup/Team
GC	Agilent	7890B	us16453037	11/1/2016	UNK	WB SVOA	3116	QA Backup/Team
GC	Agilent	7890B	CN17123083	UNK	UNK	WB SVOA	3215	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
GC	Agilent	7890B	CN13463132	1/1/2014	UNK	WestVOA	2045	QA Backup/Team
GC	Agilent	7890B	CN14703033	6/1/2014	UNK	WestVOA	2095	QA Backup/Team
GC	Agilent	7890B	CN15143068	9/1/2015	UNK	WestVOA	2310	QA Backup/Team
GC	Agilent	7890B	CN16503164	1/1/2017	New	WestVOA	3184	QA Backup/Team
GC	Agilent	7890b	CN17493168	12/1/2018	New	WestVOA	3695	QA Backup/Team
GC	Agilent	7890B	CN19513025	12/1/2019	New	WestVOA	3792	QA Backup/Team
GC	Agilent	7890B (G3442B)	CN16123112	UNK	New	WB SVOA	3100	QA Backup/Team
GC	Agilent	7890B (G3442B)	cn16293097	UNK	New	WB SVOA	3108	QA Backup/Team
GC	Agilent	7890B (G3442B)	cn16293093	UNK	New	WB SVOA	3104	QA Backup/Team
GC	Agilent	7890B/G3442 B	CN15483138	UNK	New	WestVOA	3063	QA Backup/Team
GC	Agilent	7890B/G3442 B	CN17513094	4/1/2018	New	WestVOA	3375	QA Backup/Team
GC	Agilent	G1530N	CN10528088	10/10/2005	UNK	WB SVOA	766	QA Backup/Team
GC	Agilent	G1530N	CN10528089	10/10/2005	UNK	WB SVOA	765	QA Backup/Team
GC	Agilent	G3442B	CN16293093	UNK	UNK	WB SVOA	3104	QA Backup/Team
GC	HP	6890	US00029050	11/1/1999	UNK	WestVOA	3795	QA Backup/Team
GC	HP	6890N	US10151112	2-Jun	UNK	WB SVOA	692	QA Backup/Team
GC	HP	6890N	US10225135	5/1/2003	UNK	WestVOA	689	QA Backup/Team
GC	HP	G1530A	US00008152	6/5/1997	UNK	WB SVOA	366	QA Backup/Team
GC	HP	HP 6890 series	US00020499	May-98	UNK	WB SVOA	423	QA Backup/Team
GC	HP	HP6890 series	US00021266	Jun-98	UNK	WB SVOA	452	QA Backup/Team
GC	HP	HP6890 Series	US00032120	4/1/2000	UNK	WestVOA	3797	QA Backup/Team
GC	Agilent	7890B	CN14173019	8/18/2014	New	WB SVOA	3717	QA Backup/Team
GC	Agilent	7890A	CN10371137	UNK	UNK	WB SVOA	1115	QA Backup/Team
GC Fan	Zip Scientific	GC Chaser	UNK	7-Feb	UNK	WB SVOA	814	QA Backup/Team
GC Fan	Zip Scientific	GC Chaser	1215545	UNK	UNK	WB SVOA	1063	QA Backup/Team
GC Fan	Zip Scientific	GC Chaser	UNK	7-Jan	UNK	WB SVOA	812	QA Backup/Team
GC Fan	Zip Scientific	GC Chaser	1215526	UNK	UNK	WB SVOA	1147	QA Backup/Team
GC Fan	Zip Scientific	GC Chaser	1215525	4/1/2008	UNK	WestVOA	835	QA Backup/Team
GC Fan	Zip Scientific	GC Chaser	1215524	4/1/2008	UNK	WestVOA	836	QA Backup/Team
GC Fan	Zip Scientific	GC Chaser	1215576	UNK	UNK	WestVOA	1057	QA Backup/Team
Gilson Frac Unit	Gilson	GX274	261M8N261	12/19/2018	New	WB Organic Prep	3691	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Gilson Frac Unit	Gilson	GX274 Aspec	261A6N021	12/1/2016	New	WB Organic Prep	3273	QA Backup/Team
Gilson Pump	Gilson	Verity 4260	641B6Q013	N/A	N/A	WB Organic Prep	3275	QA Backup/Team
Gilson Pump	Gilson	Verity 4260	641B6Q012	N/A	N/A	WB Organic Prep	3274	QA Backup/Team
Gilson Pump	Gilson	Verity 4260	641L8Q098	12/19/2018	New	WB Organic Prep	3692	QA Backup/Team
Gilson Pump	Gilson	Verity 4260	641L8Q099	12/19/2018	New	WB Organic Prep	3693	QA Backup/Team
Grinding Mill	IKA Works	T25BS1	3026403	UNK	UNK	WB Wet Chemistry	157	QA Backup/Team
Headspace Concentrator/sampler	Teledyne tekmar	HT3	US15016001	UNK	New	WestVOA	3751	QA Backup/Team
Headspace Concentrator/sampler	Teledyne tekmar	HT3	US1512004	UNK	New	WestVOA	3751	QA Backup/Team
Headspace Concentrator/sampler	Teledyne tekmar	HT3	US16074007	UNK	New	WestVOA	3748	QA Backup/Team
Headspace Concentrator/sampler	Teledyne tekmar	HT3	US16074007	UNK	New	WestVOA	3749	QA Backup/Team
Headspace Concentrator/sampler	Teledyne tekmar	HT3	US18096003	4/1/2018	New	WestVOA	3799	QA Backup/Team
Headspace Concentrator/sampler	Teledyne tekmar	HT3	US19114002	UNK	New	WestVOA	3750	QA Backup/Team
Heat Block, Digital	VWR	UNK	None	UNK	UNK	WestVOA	1012	QA Backup/Team
Helium Manifold	Concoa	5267-133-57-590	05301LAT	2005	UNK	WB SVOA	779	QA Backup/Team
Hood-Benchtop	Captairflex	M481	Enc1008731	3/8/2016	New	WB Login	2340	QA Backup/Team
Hood-Benchtop HEPA	Labconco	398032	080383851C	UNK	UNK	WB Login	1212	QA Backup/Team
Hot Block	Environmental Express	SC154	8462CECW3635	UNK	UNK	WB Wet Chemistry	3264	QA Backup/Team
Hot Block Controller	V&P Scientific	VP 710 Series	710806	1/1/2019	New	WB Wet Chemistry	3723	QA Backup/Team
Hot Block/Stir Plate	Environmental Express	SC 151	7021CECW3334	8/14/2012	UNK	WB Wet Chemistry	1928	QA Backup/Team
Hot Plate	Barnstead	HP46225	2.37E+11	N/A	N/A	WB Organic Prep	3840	QA Backup/Team
Hot Plate	Barnstead	HP46225	2878307	UNK	UNK	WB Wet Chemistry	1171	QA Backup/Team
Hot Plate	Barnstead	HP47135	6.12E+11	UNK	UNK	WB Wet Chemistry	1172	QA Backup/Team
Hot Plate	Thermo Scientific	HPA2240MQ	C1707150619208	N/A	New	WB Organic Prep	3399	QA Backup/Team
Hot Plate	Thermo Scientific	HPA2240MQ	C1707150411634	N/A	New	WB Organic Prep	3397	QA Backup/Team
Hot Plate	Thermo Scientific	HPA2240MQ	C1707150411637	N/A	New	WB Organic Prep	2301	QA Backup/Team
Hot Plate	Thermo Scientific	HPA2240MQ	C1707150619205	N/A	New	WB Organic Prep	2302	QA Backup/Team
Hot Plate	Thermo Scientific	HPA4440M	C1707140203963	4/2/2014	New	WB Organic Prep	2078	QA Backup/Team
Hot Plate	Thermolyne	Cinarec2	UNK	UNK	UNK	WB Wet Chemistry	1175	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
HotPlate/Stir	Barnstead	Multi-magnastir 1268				WB Wet Chemistry	1174	QA Backup/Team
HotPlate/Stir	Barnstead	Multi-magnastir 1288R	0902-1079	UNK	UNK	WB Wet Chemistry	1173	QA Backup/Team
HotPlate/Stir	Corning	PC-320	unknown	UNK	UNK	WB Wet Chemistry	1184	QA Backup/Team
HotPlate/Stir	Corning	PC-520	3010478	UNK	UNK	WB Wet Chemistry	1183	QA Backup/Team
HotPlate/Stir	Lab-Line	Pyro Multi-magnastir	1.66E+12	UNK	UNK	WB Wet Chemistry	1170	QA Backup/Team
HotPlate/Stir	VWR	12620-986	70206006	UNK	UNK	WB Wet Chemistry	1176	QA Backup/Team
HotPlate/Stir	VWR	12620-986	70206002	UNK	UNK	WB Wet Chemistry	1177	QA Backup/Team
HotPlate/Stir	VWR	12620-986	70206005	UNK	UNK	WB Wet Chemistry	1178	QA Backup/Team
HPLC	Shimadzu	LC-2030C-3D	L21455300850	5/12/2014	New	WB Wet Chemistry	3124	QA Backup/Team
Hydrogen Generator	PARKER	H2PEMD-1300-100	15PLD5036	4/1/2015	New	WB SVOA	2276	QA Backup/Team
Hydrogen Generator	Parker	H2PEMPD-1300-100	15PLD5035	4/1/2015	New	WB SVOA	2277	QA Backup/Team
Ice Machine	Scotsman	CME806AS-32F	6.02E+12	8/20/2006	UNK	WB Login	790	QA Backup/Team
Incubator	Barnstead	120	1.46E+12	UNK	UNK	WB Wet Chemistry	1221	QA Backup/Team
Incubator	Fisher	650D	412N0382	5/2/2005	UNK	WB Wet Chemistry	742	QA Backup/Team
Incubator	ShelLab	SRI20P	5010117	5/5/2017	New	WB Wet Chemistry	3265	QA Backup/Team
Incubator	ShelLab	SRI20P	1003218	UNK	New	WB Microbiology	3299	QA Backup/Team
Incubator	Thermo Electron Corp.	6LM	102711	4/1/2006	UNK	WB Microbiology	789	QA Backup/Team
Incubator	VWR	2005	9003704	11/2/2004	UNK	WB Wet Chemistry	731	QA Backup/Team
Incubator	VWR Sheldon	1545	1011505	5/2/2005	UNK	WB Microbiology	741	QA Backup/Team
Interface Module	Mantech	Automax 122	MT-1H3-205	10/22/2013	New	WB Wet Chemistry	2258	QA Backup/Team
Ion Chromatograph	Thermo	22153-60315 Dionex HPLC	17070793	UNK	New	WB Wet Chemistry	3352	QA Backup/Team
Ionization Gauge Controller	Agilent	59864B	US6010073	UNK	UNK	WB SVOA	1062	QA Backup/Team
Ionization Gauge Controller	Agilent	59864B	US60101846	UNK	UNK	WB SVOA	1067	QA Backup/Team
Ionization Gauge Controller	HP	59864A	UNK	UNK	UNK	WestVOA	534	QA Backup/Team
IR Gun	Omega	OS542	141008955	UNK	UNK	WB Login	3088	QA Backup/Team
Kiln	Paragon DTC 100	TNF823	283662	UNK	UNK	WB Wet Chemistry	276	QA Backup/Team
Kiln	Select Fire	ex 270sf	7385	6/1/2019	N/A	WB Organic Prep	3123	QA Backup/Team
Lachat	HACH	Quickchem QC8500 Series 2	1.91E+11	12/27/2019	New	WB Wet Chemistry	3777	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Lachat Analyzer	Lachat	Quick Chem 8500 Series 2	90300001086	4/30/2009	UNK	WB Wet Chemistry	865	QA Backup/Team
Lachat Analyzer	Lachat	Quickchem QC8500 Series 2	1.40E+11	5/28/2014	New	WB Wet Chemistry	2114	QA Backup/Team
Lachat Autosampler	Lachat	ASX-520	031445A520	5/28/2014	New	WB Wet Chemistry	2113	QA Backup/Team
Lachat Autosampler	Lachat	ASX-520 series	90300001856	4/30/2009	UNK	WB Wet Chemistry	864	QA Backup/Team
Lachat Diluter	Lachat	PDS 200	90300000509	4/30/2009	UNK	WB Wet Chemistry	867	QA Backup/Team
Lachat Diluter	Lachat	PDS 200	1.41E+11	5/28/2014	New	WB Wet Chemistry	2115	QA Backup/Team
Lachat Pump	Lachat	ISM1135	A82000-3058	5/28/2014	New	WB Wet Chemistry	2112	QA Backup/Team
LC/MS/MS	ABI/Sciex	API-2000	B7590411	2/2/2005	UNK	WB SVOA	743	QA Backup/Team
Line Voltage Transformer	SCIEX	14179	117255104	UNK	UNK	WB SVOA	1095	QA Backup/Team
Mass Spectrometer	Agilent	5977B	US2131R033	9/1/2021	New	WestVOA	4272	QA Backup/Team
Mass Spectrometer	Agilent	5977B	US2131R022	9/1/2021	New	WestVOA	4276	QA Backup/Team
Mass Spectrometer	Agilent	5977b	US2144R051	12/21/2024	New	WestVOA	4285	QA Backup/Team
Microscope	Fisher	Stereomaster Z 5	1891	UNK	UNK	WB Microbiology	137	QA Backup/Team
Microwave	CEM	91900	1891421	10/1/2012	New	WB Organic Prep	1887	QA Backup/Team
Microwave	CEM	910900	MJ2060	N/A	N/A	WB Organic Prep	3611	QA Backup/Team
Microwave	CEM	910900	MJ6008	4/15/2015	New	WB Organic Prep	4041	QA Backup/Team
Microwave	CEM	Mars 6	MJ 9687	12/26/2018	New	WB Organic Prep	3085	QA Backup/Team
Microwave	CEM	Mars 6	AC1020	8/29/2019	New	WB Organic Prep	3711	QA Backup/Team
Microwave	CEM	R907501	MD4731	10/28/2016	N/A	WB Organic Prep	3115	QA Backup/Team
MS	Agilent	5973	US10461505	UNK	UNK	WB SVOA	1069	QA Backup/Team
MS	Agilent	5973	US43110455	3/15/2005	UNK	WestVOA	738	QA Backup/Team
MS	Agilent	5973	US43110414	11/1/2004	UNK	WestVOA	715	QA Backup/Team
MS	Agilent	5973	US90160009	6/1/2003	UNK	WestVOA	711	QA Backup/Team
MS	Agilent	5975	US52420453	5-Sep	UNK	WB SVOA	838	QA Backup/Team
MS	Agilent	5973 Network	BN30464483	2-Mar	UNK	WB SVOA	679	QA Backup/Team
MS	Agilent	5973N	US10451041	1-Jul	UNK	WB SVOA	664	QA Backup/Team
MS	Agilent	5975C	US11023714	UNK	UNK	WB SVOA	1074	QA Backup/Team
MS	Agilent	5975C	US10373606	9/14/2010	UNK	WB SVOA	1376	QA Backup/Team
MS	Agilent	5975C	US10213435	UNK	UNK	WB SVOA	1445	QA Backup/Team
MS	Agilent	5975C	US10223405	UNK	UNK	WB SVOA	1417	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
MS	Agilent	5975C	US11093732	UNK	UNK	WB SVOA	1371	QA Backup/Team
MS	Agilent	5975C	US80818631	4/1/2008	UNK	WestVOA	833	QA Backup/Team
MS	Agilent	5975C	US10033003	UNK	UNK	WestVOA	1027	QA Backup/Team
MS	Agilent	5975C	US71215997	9/1/2007	UNK	WestVOA	843	QA Backup/Team
MS	Agilent	5975C	US10113007	6/7/2010	UNK	WestVOA	1046	QA Backup/Team
MS	Agilent	5975C	US10133009	Spring 2010	New	WestVOA	1005	QA Backup/Team
MS	Agilent	5975C	US83110164	UNK	UNK	WestVOA	1019	QA Backup/Team
MS	Agilent	5975C	US11093709	UNK	Used	WestVOA	1391	QA Backup/Team
MS	Agilent	5975C	US10393819	UNK	UNK	WestVOA	1357	QA Backup/Team
MS	Agilent	5975C	US10163303	5/10/2024	New	WestVOA	1403	QA Backup/Team
MS	Agilent	5977A	US1410L222	5/1/2014	UNK	WB SVOA	2085	QA Backup/Team
MS	Agilent	5977A	US1419L202	7/1/2014	UNK	WB SVOA	2101	QA Backup/Team
MS	Agilent	5977A	US139L217	1/1/2014	UNK	WestVOA	2046	QA Backup/Team
MS	Agilent	5977A	US14118L211	6/1/2014	UNK	WestVOA	2096	QA Backup/Team
MS	Agilent	5977B	US1850R041	UNK	UNK	WB SVOA	3699	QA Backup/Team
MS	Agilent	5977B	US1850R037	12/1/2018	New	WestVOA	3696	QA Backup/Team
MS	Agilent	5977B/G7081B	US1952R015	19-Dec	New	WB SVOA	3790	QA Backup/Team
MS	Agilent	5977B/G7081B	US1609R019	UNK	New	WestVOA	3062	QA Backup/Team
MS	Agilent	5977B/G7081B	US1814R010	4/1/2018	New	WestVOA	3376	QA Backup/Team
MS	Agilent	5977B/G7081B	US1952R008	12/1/2019	New	WestVOA	3793	QA Backup/Team
MS	Agilent	G3170A	US65115053	UNK	UNK	WB SVOA	825	QA Backup/Team
MS	Agilent	G7077B	CN2+25M013	UNK	New	WB SVOA	3105	QA Backup/Team
MS	Agilent	G7077B	us1630r005	UNK	New	WB SVOA	3109	QA Backup/Team
MS	Agilent	G7077B	us1630r007	UNK	New	WB SVOA	3105	QA Backup/Team
MS	HP	5973	US80221319	May-98	UNK	WB SVOA	424	QA Backup/Team
MS	HP	5973	US92522665	11/1/1999	UNK	WestVOA	3796	QA Backup/Team
MS	HP	G1098A	US71410498	6/5/1997	UNK	WB SVOA	367	QA Backup/Team
MS	Agilent	5977A	US1426l317	8/18/2012	New	WB SVOA	2120	QA Backup/Team
MS	Agilent	5977A	US1513423	9/1/2015	UNK	WestVOA	2311	QA Backup/Team
MS	Agilent	7890B	CN17083153	4/1/2017	New	WestVOA	3269	QA Backup/Team
MS	Agilent	Agilent MS 5977B	US1701R001	1/1/2017	New	WestVOA	3077	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Muffle Furnace	Thermolyne	Type 6000	1.06E+12	12/1/1999	UNK	WB Wet Chemistry	621	QA Backup/Team
Orbital Shaker	Lab-Line	3627	0402-6479	5/1/2003	UNK	WB Wet Chemistry	769	QA Backup/Team
Oven	Fisher	6925	610666-291	UNK	UNK	WB Wet Chemistry	1888	QA Backup/Team
Oven	Fisher	3510FS	1.88E+12	UNK	UNK	WB Login	1213	QA Backup/Team
Oven	Fisher	655G	109982 or 11000174	UNK	UNK	WB Wet Chemistry	1162	QA Backup/Team
Oven	Fisher	Iso-Temp 655G	UNK	UNK	UNK	WestVOA	541	QA Backup/Team
Oven	Fisher	655F	90400206	UNK	UNK	WB Wet Chemistry	1166	QA Backup/Team
Oven	ShelLab	SMO28-2	5014617	6/8/2017	New	WB Wet Chemistry	3266	QA Backup/Team
Oven	VWR	1690	3015012	11/16/2015	New	WB Login	2332	QA Backup/Team
Oven	VWR	1350GM	3059606	5/1/2006	UNK	WB Wet Chemistry	811	QA Backup/Team
Oven	VWR	1370F	700698	UNK	UNK	WB Wet Chemistry	1163	QA Backup/Team
Oven	VWR	89511-404	41854416	3/8/2016	New	WB Organic Prep	2336	QA Backup/Team
Oven	VWR Sheldon	1680	7064804	11/1/2004	UNK	WB Wet Chemistry	728	QA Backup/Team
pH Meter	Hanna	HI 5521	F0022641	UNK	New	WB Wet Chemistry	3128	QA Backup/Team
pH Meter	Hanna	HI5521-01	3130023991	11/26/2018	New	WB Organic Prep	3086	QA Backup/Team
pH Meter	Hanna Instruments	HI 5521	F0011343	10/30/2015	New	WB Wet Chemistry	2329	QA Backup/Team
pH Meter	Hanna Instruments	HI2002-01	C0322019	UNK	New	WB Wet Chemistry	3267	QA Backup/Team
pH Meter	Hanna Instruments	HI2002-01	CO813071	12/21/2024	New	WB Wet Chemistry	3947	QA Backup/Team
pH Meter	Hanna Instruments	HI2020-01	C051079	UNK	New	WB Wet Chemistry	3705	QA Backup/Team
pH Meter	Hanna Instruments	Hl2002-01	CO3261C3	5/2/2017	New	WestVOA	3219	QA Backup/Team
pH Meter	Hanna Insturments	H198128	7390442121	UNK	New	WB Wet Chemistry	4288	QA Backup/Team
pH Meter	Orion	940	RT89A	UNK	UNK	WB Wet Chemistry	675	QA Backup/Team
pH Meter	Orion	210A+	21847	9/1/2005	UNK	WB Wet Chemistry	761	QA Backup/Team
pH Meter	Orion	555A	17857	4/1/2005	UNK	WB Wet Chemistry	810	QA Backup/Team
pH/Conductivity Meter	Oakton	35413-00	2918627	1/8/2020	New	WB Wet Chemistry	3746	QA Backup/Team
Pump	Buchi	V-700	1000143237	10/4/2013	New	WB Organic Prep	2024	QA Backup/Team
Pump	Buchi	V-700	1000153942	10/8/2013	New	WB Organic Prep	2028	QA Backup/Team
Pump	Buchi	V-700	1000153941	10/12/2013	New	WB Organic Prep	2032	QA Backup/Team
Pump	Buchi	V-700	1000175456	8/13/2014	New	WB Organic Prep	2126	QA Backup/Team
Pump	Buchi	V-700	1000176883	8/13/2014	New	WB Organic Prep	2130	QA Backup/Team
Pump	Buchi	V-700	1000204369	6/19/2015	New	WB Organic Prep	2294	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Pump	Cole-Palmer	07522-20	F17005271	UNK	New	WestVOA	3760	QA Backup/Team
Pump	Pfeiffer	DUO 2.5, PDK41062G	22033282	4/1/2017	New	WestVOA	3271	QA Backup/Team
Pump (Lachat)	HACH	12Ch PVD-115V	W19005570	12/27/2019	New	WB Wet Chemistry	3776	QA Backup/Team
Purge & Trap Concentrator	EST Analytical	Centurion	CENTS496033117	4/1/2017	New	WestVOA	3272	QA Backup/Team
Purge & Trap Concentrator	EST Analytical	Encon Evolution	EV550012214	1/1/2014	UNK	WestVOA	2048	QA Backup/Team
Purge & Trap Concentrator	EST Analytical	Encon Evolution	EV574050514	6/1/2014	UNK	WestVOA	2094	QA Backup/Team
Purge & Trap Concentrator	EST Analytical	Encon Evolution	EV585060314	9/1/2015	UNK	WestVOA	2309	QA Backup/Team
Purge & Trap Concentrator	EST Analytical	Encon Evolution	EV829033117	4/1/2017	New	WestVOA	3270	QA Backup/Team
Purge & Trap Concentrator	EST Analytical	EST Evolution	EV804010317	1/1/2017	New	WestVOA	3078	QA Backup/Team
Purge & Trap Concentrator	EST/PTS Analytical, Inc.	Encon Evolution	EV376091911	9/1/2011	UnUsed	WestVOA	2014	QA Backup/Team
Purge & Trap Concentrator	Tekmar	Lumin	US19361008	12/1/2019	New	WestVOA	3801	QA Backup/Team
Purge and trap concentrator	EST Analytical	Evolution	4.16E+11	UNK	New	WestVOA	3358	QA Backup/Team
Purge and trap concentrator	Teledyne Tekmar	Lumin	US21222005	9/1/2021	New	WestVOA	4274	QA Backup/Team
QA Reference weight set	Rice Lake		2305	3/14/2024	New	West QA	2305	WEST QA
QuantiTray Sealer	IDEXX	89-20894-04	05267-08-023	4/10/2013	New	WB Wet Chemistry	2261	QA Backup/Team
Refrigerator	Asber	ARMD 49	8101980025	6/3/2021	New	WB Login	4261	QA Backup/Team
Refrigerator	Asber	AWRR-23F	8102136498	4/16/2021	New	WB Wet Chemistry	4046	QA Backup/Team
Refrigerator	Avanti	ARBC17T2PG	A5810191178 1862800068	3/4/2019	New	WB Organic Prep	3697	QA Backup/Team
Refrigerator	Avanti	ARBC17T2PG	A5810191178 1852900050	N/A	New	WB Organic Prep	3706	QA Backup/Team
Refrigerator	Beverage Air	LV45-1-B	9503809	UNK	UNK	WB Login	1252	QA Backup/Team
Refrigerator	Beverage Air	LV45-1-B	9702500	UNK	Used	WB Login	1597	QA Backup/Team
Refrigerator	Beverage Air	LV45-1-B-LED		4/24/2024	New	WB Login	2271	QA Backup/Team
Refrigerator	Beverage Air	LV45-1-B-LED	11602214	8/20/2015	New	WB Login	2303	QA Backup/Team
Refrigerator	Beverage Air	LV45-1-W	9705029	5/6/2011	New	WB Login	1256	QA Backup/Team
Refrigerator	Beverage Air	LV45-1-W	9803502	6/22/2011	New	WB Login	1052	QA Backup/Team
Refrigerator	Beverage Air	LV45-1-W	9410518	UNK	Used	WB Login	1596	QA Backup/Team
Refrigerator	Beverage Air	LV45-1-W	9803502	UNK	UNK	WestVOA	1052	QA Backup/Team
Refrigerator	Beverage Air	LV45-1-W-LED	12104441	5/15/2017	New	WB Login	3732	QA Backup/Team
Refrigerator	Beverage Air	LV45HC-1-Wq	12402812	9/18/2017	New	WB Login	3292	QA Backup/Team
Refrigerator	Blue Air	BKGM49-HC	KGM49-HC-V11-0548	6/3/2021	New	WB Login	4260	QA Backup/Team
Refrigerator	Della	048-GM-48313	1805000096	7/27/2018	New	WB Wet Chemistry	3419	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Refrigerator	Frigidaire	EFMIS9000	A2203420230001879	9/22/2024	New	WB Organic Prep	4716	QA Backup/Team
Refrigerator	Frigidaire	EFMIS9000	A2202412030000042	9/22/2024	New	WB Organic Prep	4717	QA Backup/Team
Refrigerator	Frigidaire	FRU17B2JW20	WA01002558	UNK	UNK	WB Microbiology	1222	QA Backup/Team
Refrigerator	Haier	HRC2736BWB	HH647591	UNK	UNK	WB Wet Chemistry	3412	QA Backup/Team
Refrigerator	Haier	HRC2736BWB	HH647657	UNK	UNK	WB Wet Chemistry	3413	QA Backup/Team
Refrigerator	Haier	HRC2736BWB	FH638434	UNK	UNK	WB Wet Chemistry	3414	QA Backup/Team
Refrigerator	Kenmore	106.762494	ET2516230	11/6/2024	UNK	WestVOA	1595	QA Backup/Team
Refrigerator	Kenmore	253-60722007	WA93700459	1/7/2010	New	WB Microbiology	1224	QA Backup/Team
Refrigerator	Kenmore	795-73259300	305MR00109	UNK	UNK	WB SVOA	1145	QA Backup/Team
Refrigerator	Kenmore	unknown	unknown	UNK	UNK	WB Wet Chemistry	1167	QA Backup/Team
Refrigerator	Kenmore	unknown	unknown	1/1/2001	UNK	WB Microbiology	1223	QA Backup/Team
Refrigerator	Powers	BS103GD	B033878	N/A	N/A	WB Organic Prep	1235	QA Backup/Team
Refrigerator	Powers	BS103GD4	B050961	4/1/2005	N/A	WB Organic Prep	735	QA Backup/Team
Refrigerator	Powers	BS33SD	D947046	UNK	UNK	WestVOA	297	QA Backup/Team
Refrigerator	Powers	BS33SD	C946508	UNK	UNK	WestVOA	299	QA Backup/Team
Refrigerator	Sears	253-67189504	4A63621623	UNK	UNK	WB Wet Chemistry	1167	QA Backup/Team
Refrigerator	True Refrigeration	GDM-45	6880875	UNK	Used	WB Login	1595	QA Backup/Team
Refrigerator/Freezer	Frigidaire	FFTR1814QW9B	A7121956	4/24/2017	New	WestVOA	3211	QA Backup/Team
Refrigerator/Freezer	VWR	scrf-20a	vwr-4a74903508-1805	New	WB SVOA	3374	QA Backup/Team	
RO Generator	Evoqua	MFXR21003A	1615580	3/1/2017	New	WB Wet Chemistry	3260	QA Backup/Team
RPM Meter	Monarch	TACH-4A	1133816	UNK	New	West QA	751	WEST QA
Sensor and Stir Control	ManTech	Automax122	MT-1H3-161	UNK	New	WB Wet Chemistry	2260	QA Backup/Team
Shaker/Incubator	Lab-Line	3627	0402-6479	UNK	UNK	WB Wet Chemistry	769	QA Backup/Team
Solid Phase Extractor	Horizon	3000XL	72088	UNK	UNK	WB Wet Chemistry	1208	QA Backup/Team
Solid Phase Extractor	Horizon	3000XL	72087	UNK	UNK	WB Wet Chemistry	1207	QA Backup/Team
Solid Phase Extractor	Horizon	SPE-DEX 3000XL	13-1926	6/3/2013	New	WB Wet Chemistry	2010	QA Backup/Team
Solid Phase Extractor Controller	Horizon	Oil & Grease Controller	13-1340	6/3/2013	New	WB Wet Chemistry	2009	QA Backup/Team
Solvent Evaporation System	Horizon	SpeedVap II 9000	99-167	6/1/2024	UNK	WB Wet Chemistry	668	QA Backup/Team
Solvent Evaporation System	Horizon	SpeedVapIII	456002	UNK	UNK	WB Wet Chemistry	1173	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Solvent Evaporator	Organimation	406	57409	N/A	N/A	WB Organic Prep	3080	QA Backup/Team
Solvent Evaporator	Organimation	406	57968	N/A	N/A	WB Organic Prep	3081	QA Backup/Team
Solvent Evaporator	Organimation	16169	62481	N/A	N/A	WB Organic Prep	3415	QA Backup/Team
Solvent Evaporator	Organimation	OA-Sys	61619	N/A	N/A	WB Organic Prep	3279	QA Backup/Team
Solvent Evaporator	Organomation	13165	63969	7/16/2021	New	WB Organic Prep	4266	QA Backup/Team
Solvent Evaporator	Organomation	14169	58990	5/1/2013	New	WB Organic Prep	3369	QA Backup/Team
Solvent Evaporator	Organomation	14169	58371	5/2/2013	New	WB Organic Prep	3372	QA Backup/Team
Solvent Evaporator	Organomation	14169	58376	5/1/2013	New	WB Organic Prep	3368	QA Backup/Team
Solvent Evaporator	Organomation	14169	58374	5/1/2013	New	WB Organic Prep	3743	QA Backup/Team
Solvent Evaporator	Organomation	14169	58372	5/1/2013	New	WB Organic Prep	3373	QA Backup/Team
Solvent Evaporator	Organomation	16165	64428	3/1/2022	New	WB Organic Prep	4291	QA Backup/Team
Solvent Evaporator	Organomation	N/A	57183	N/A	N/A	WB Organic Prep	3739	QA Backup/Team
Solvent Evaporator	Organomation	N-EVAP 112	57409	4/15/2012	New	WB Organic Prep	1913	QA Backup/Team
Solvent Evaporator	Organomation	N-EVAP 116	57318	11/1/1997	N/A	WB Organic Prep	3361	QA Backup/Team
Solvent Evaporator	Organomation	N-EVAP 116	54344	N/A	N/A	WB Organic Prep	1912	QA Backup/Team
Sonicator	Qsonica	Q500	71932AD+11-12	11/30/2012	New	WB Organic Prep	3393	QA Backup/Team
Sonicator	BRANSON	M1800	BGA081823140B	10/19/2018	New	WB SVOA	3497	QA Backup/Team
Spectrophotometer	HACH	DR/2000	900409085	2/19/2016	Used	WB Wet Chemistry	2338	QA Backup/Team
Spectrophotometer	HACH	DR/2000 Direct Read	9.61E+11	UNK	UNK	WB Wet Chemistry	482	QA Backup/Team
Spectrophotometer	Spectronic	Genesys 5	3Volatiles141004	12/18/1998	UNK	WB Wet Chemistry	472	QA Backup/Team
Spectrophotometer	Thermo Scientific	G10S UV-VIS	2L6S254202	11/17/2014	New	WB Wet Chemistry	3350	QA Backup/Team
Spectrophotometer	Thermo Scientific	Genesys 50	9A3Y2500056	12/16/2020	New	WB Wet Chemistry	4265	QA Backup/Team
Spectrophotometer	Thermofisher Scientific	G10s UV-VIS	2L5T315224	11/19/2015	New	WB Wet Chemistry	2328	QA Backup/Team
Speed Vap	Horizon	Speed Vap 3	07-0651	7/7/2007		WB Wet Chemistry	1929	QA Backup/Team
Stir Plate	VWR	MultiStir 9	160421001	4/28/2016	New	WB Organic Prep	2339	QA Backup/Team
Stirbase for Hotblock	Environmental Express	SC100	2016Stir100	1/1/2019	New	WB Wet Chemistry	3725	QA Backup/Team
TCLP Tumbler	Analytical Testing Corp.	DC-20B Rotary Agitator	N/A	9/8/2011	New	WB Wet Chemistry	1004	QA Backup/Team
TCLP Tumbler	Associated Design	3740-12-BRE	None	UNK	UNK	WestVOA	1014	QA Backup/Team
TCLP Tumbler	Dayton	None	None	UNK	UNK	WestVOA	1016	QA Backup/Team
TCLP/SPLP Tumbler	Environmental Express	LE1002	39564698003	2/19/2014	New	WB Organic Prep	2074	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
TCLP/SPLP Tumbler	Environmental Express	LE1002	39564698010	2/19/2014	New	WB Organic Prep	2073	QA Backup/Team
TCLP/SPLP Tumbler	Environmental Express	LE1002	39564698007	2/19/2014	New	WB Organic Prep	2072	QA Backup/Team
TCLP/SPLP Tumbler	Environmental Express	LE1002	39564698001	2/19/2014	New	WB Organic Prep	2071	QA Backup/Team
TCLP/SPLP Tumbler	N/A	N/A	N/A	N/A	N/A	WB Organic Prep	2049	QA Backup/Team
TCLP/SPLP Tumbler	N/A	N/A	N/A	N/A	N/A	WB Organic Prep	2058	QA Backup/Team
TCLP/SPLP Tumbler	N/A	N/A	N/A	N/A	N/A	WB Organic Prep	2059	QA Backup/Team
TCLP/SPLP Tumbler	N/A	N/A	N/A	N/A	N/A	WB Organic Prep	2060	QA Backup/Team
TCLP/SPLP Tumbler	N/A	N/A	N/A	N/A	N/A	WB Organic Prep	2050	QA Backup/Team
TCLP/SPLP Tumbler	N/A	N/A	N/A	N/A	N/A	WB Organic Prep	2051	QA Backup/Team
TCLP/SPLP Tumbler	N/A	N/A	N/A	N/A	N/A	WB Organic Prep	2052	QA Backup/Team
TCLP/SPLP Tumbler	N/A	N/A	N/A	N/A	N/A	WB Organic Prep	2053	QA Backup/Team
TCLP/SPLP Tumbler	N/A	N/A	N/A	N/A	N/A	WB Organic Prep	2054	QA Backup/Team
TCLP/SPLP Tumbler	N/A	N/A	N/A	N/A	N/A	WB Organic Prep	2055	QA Backup/Team
TCLP/SPLP Tumbler	N/A	N/A	N/A	N/A	N/A	WB Organic Prep	2056	QA Backup/Team
TCLP/SPLP Tumbler	N/A	N/A	N/A	N/A	N/A	WB Organic Prep	2057	QA Backup/Team
TCN Distillation Unit Blue	Galstron	Midi-Dist	2357	?	?	WB Wet Chemistry	3229	QA Backup/Team
Teactor	Gerhardt	Vapodest 20S	1/7370 13 0004	12/11/2015	New	WB Wet Chemistry	3187	QA Backup/Team
Thermocouple Reader	Control Company	127U00	36313	6/4/2015	New	WB Wet Chemistry	2286	QA Backup/Team
Thermometer	Klein Tools	unknown	N/A	unknown	New	WB Login	4913	QA Backup/Team
Thermometer	Klein Tools	unknown	N/A	unknown	New	WB Login	4914	QA Backup/Team
Thermometer	Omega	OS542	141008955	5/15/2015	New	WB Login	3088	QA Backup/Team
Thermometer	Omega	OS542	140409823		New	WB Login	3492	QA Backup/Team
Thermometer	Omega	OS542	121205819	8/29/2013	New	WB Wet Chemistry	2109	QA Backup/Team
Titration Rinse Pump	Mantech	TitraRinse/ A Module Automax 122	MT-1H3-504	10/22/2013	New	WB Wet Chemistry	2259	QA Backup/Team
Titrator	HACH		9.90E+11			WB Wet Chemistry	1203	QA Backup/Team
Titrator	Mettler Toledo	T70	B526103250	8/1/2015	New	WB Wet Chemistry	2316	QA Backup/Team
Titrator Control	Mettler Toledo	N/A	B526103250	8/1/2015	New	WB Wet Chemistry	2318	QA Backup/Team
Titrator Dosing Unit	Mettler Toledo	Dosing Unit	B522989237	8/1/2015	New	WB Wet Chemistry	2317	QA Backup/Team
Titrator Dosing Unit	Mettler Toledo	N/A	B522989237	8/1/2015	New	WB Wet Chemistry	2317	QA Backup/Team
TOC Analyzer	Shimadzu	TOC-V WP	638-91116-42	5/1/2016	New	WB Wet Chemistry	3093	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
TOC Analyzer	Shimadzu	TOC-VWP	H5171470004 5NK			WB Wet Chemistry	1219	QA Backup/Team
TOC Analyzer	Shimadzu	TOC-VWP	H5172570038 2	3/1/2020	New	WB Wet Chemistry	3816	QA Backup/Team
TOC Analyzer	Shimadzu	TOC-VWP	H5172590064 8 NK	2/17/2022	New	WB Wet Chemistry	4292	QA Backup/Team
TOC Autosampler	Shimadzu	ASI-V	H5212520008 5 SA	9/22/2015	UNK	WB Wet Chemistry	3126	QA Backup/Team
TOC Autosampler	Shimadzu	ASI-V	H5210480404 0125A	UNK	UNK	WB Wet Chemistry	1220	QA Backup/Team
TOC Autosampler	Shimadzu	ASI-V	H5212570044 5	3/1/2020	New	WB Wet Chemistry	3817	QA Backup/Team
Tower	Agilent	7683B	UNK	UNK	UNK	WB SVOA	3779	QA Backup/Team
Tower	Agilent	7683B	CN70238232	UNK	UNK	WB SVOA	1131	QA Backup/Team
Tower	Agilent	7683B	CN82450119	UNK	UNK	WB SVOA	2007	QA Backup/Team
Tower	Agilent	7683B	CN53427173	UNK	UNK	WB SVOA	1116	QA Backup/Team
Tower	Agilent	7683B	CN84653785	UNK	UNK	WB SVOA	1354	QA Backup/Team
Tower	Agilent	7683B	CN71940440	UNK	UNK	WB SVOA	1507	QA Backup/Team
Tower	Agilent	7693A	CN19310024	8/27/2019	New	WB SVOA	3715	QA Backup/Team
Tower	Agilent	CAN'T SEE	CAN'T SEE	UNK	UNK	WB SVOA	1130	QA Backup/Team
Tower	Agilent	G2613A	US12419330	UNK	UNK	WB SVOA	1129	QA Backup/Team
Tower	Agilent	G2613A	CN42437240	UNK	UNK	WB SVOA	1490	QA Backup/Team
Tower	Agilent	G2613A	CN54128256	UNK	UNK	WB SVOA	1119	QA Backup/Team
Tower	Agilent	G2613A	US11618650	UNK	UNK	WB SVOA	1081	QA Backup/Team
Tower	Agilent	G2613A	US41121685	UNK	UNK	WB SVOA	1059	QA Backup/Team
Tower	Agilent	G2613A	CN23326700	UNK	UNK	WB SVOA	3357	QA Backup/Team
Tower	Agilent	G2613A	CN42537313	UNK	UNK	WB SVOA	1472	QA Backup/Team
Tower	Agilent	G2613A	CN14222765	UNK	UNK	WB SVOA	1108	QA Backup/Team
Tower	Agilent	G2613A	CN22125486	UNK	UNK	WB SVOA	1109	QA Backup/Team
Tower	Agilent	G2613A	CN41536146	UNK	UNK	WB SVOA	1112	QA Backup/Team
Tower	Agilent	G2613A	CN14122490	UNK	UNK	WB SVOA	1367	QA Backup/Team
Tower	Agilent	G2613A	US12419337	UNK	UNK	WB SVOA	1117	QA Backup/Team
Tower	Agilent	G2913A	CN54128256	UNK	UNK	WB SVOA	1070	QA Backup/Team
Tower	Agilent	G2913A	CN85255133	UNK	UNK	WB SVOA	1139	QA Backup/Team
Tower	Agilent	G2913A	CN43920908	UNK	UNK	WB SVOA	1079	QA Backup/Team
Tower	Agilent	G2913A	CN84854097	UNK	UNK	WB SVOA	1100	QA Backup/Team
Tower	Agilent	G4513A	CN13040010	UNK	UNK	WB SVOA	1122	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Tower	Agilent	G4513A	CN10150101	UNK	UNK	WB SVOA	1404	QA Backup/Team
Tower	Agilent	G4513A	CN13470207	UNK	UNK	WB SVOA	2090	QA Backup/Team
Tower	Agilent	G4513A	CN12420101	UNK	UNK	WB SVOA	2133	QA Backup/Team
Tower	Agilent	G4513A	CN15420129	UNK	UNK	WB SVOA	3072	QA Backup/Team
Tower	Agilent	G4513A	CN17120046	UNK	UNK	WB SVOA	3217	QA Backup/Team
Tower	Agilent	G4513A	CN18120050	UNK	New	WB SVOA	3417	QA Backup/Team
Tower	Agilent	G4513A	CN17120043	UNK	New	WB SVOA	3214	QA Backup/Team
Tower	Agilent	G4513A	CN10240069	UNK	UNK	WB SVOA	1134	QA Backup/Team
Tower	Agilent	G4513A	CN10150014	UNK	UNK	WB SVOA	1408	QA Backup/Team
Tower	Agilent	G4513A	cn10240058	UNK	New	WB SVOA	3356	QA Backup/Team
Tower	Agilent	G4513A	CN10240056	UNK	UNK	WB SVOA	1125	QA Backup/Team
Tower	Agilent	G4513A	CN10240058	UNK	UNK	WB SVOA	1126	QA Backup/Team
Tower	Agilent	G4513A	CN10520150	UNK	UNK	WB SVOA	1392	QA Backup/Team
Tower	Agilent	G4513A	CN10020007	UNK	UNK	WB SVOA	1142	QA Backup/Team
Tower	Agilent	G4513A	cn10020011	UNK	New	WB SVOA	3345	QA Backup/Team
Tower	Agilent	G4513A	cn1015024	UNK	New	WB SVOA	3355	QA Backup/Team
Tower	Agilent	G4513A	CN10020014	UNK	UNK	WB SVOA	1141	QA Backup/Team
Tower	Agilent	G4513A	CN15110128	UNK	UNK	WB SVOA	2315	QA Backup/Team
Tower	Agilent	G4513A	CN15450269	UNK	UNK	WB SVOA	3069	QA Backup/Team
Tower	Agilent	G4513A	CN15450270	UNK	UNK	WB SVOA	3067	QA Backup/Team
Tower	Agilent	G4513A	CN18510067	UNK	New	WB SVOA	3703	QA Backup/Team
Tower	Agilent	G4513A	CN18510076	UNK	New	WB SVOA	3704	QA Backup/Team
Tower	Agilent	G4513A	CN15110120	6/30/2018	New	WB SVOA	2314	QA Backup/Team
Tower	Agilent	G4513A	cn16010036	UNK	New	WB SVOA	3103	QA Backup/Team
Tower	Agilent	G4513A	cn16230177	UNK	New	WB SVOA	3110	QA Backup/Team
Tower	Agilent	G4513A	CN16230177	UNK	UNK	WB SVOA	3110	QA Backup/Team
Tower	Agilent	G4513A	CN10310057	UNK	UNK	WB SVOA	1377	QA Backup/Team
Tower	Agilent	G4513A	CN10510119	UNK	UNK	WB SVOA	1372	QA Backup/Team
Tower	Agilent	G4513A	CN14290139	UNK	UNK	WB SVOA	3784	QA Backup/Team
Tower	Agilent	G4513A	CN16290215	UNK	UNK	WB SVOA	3119	QA Backup/Team
Tower	Agilent	G4513A	CN10020006	UNK	UNK	WB SVOA	1358	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Tower	Agilent	G4513A	CN14240243	8/18/2014	New	WB SVOA	2122	QA Backup/Team
Tower	Agilent	G4513A	CN10170067	UNK	New	WB SVOA	1446	QA Backup/Team
Tower	Agilent	G4513A	CN10170034	UNK	UNK	WB SVOA	1418	QA Backup/Team
Tower	Agilent	G4513A	CN14030252	UNK	UNK	WB SVOA	3783	QA Backup/Team
Tower	Agilent	G4513A	CN16290213	UNK	UNK	WB SVOA	3118	QA Backup/Team
Tower	Agilent	G4513A	CN17120044	UNK	UNK	WB SVOA	3213	QA Backup/Team
Tower	Agilent	G4513A	cn16320183	UNK	New	WB SVOA	3107	QA Backup/Team
Tower	Agilent	G4567A	CN19500018	19-Dec	New	WB SVOA	3791	QA Backup/Team
Tower	HP	7683	NA	UNK	UNK	WB SVOA	1113	QA Backup/Team
TOX-300 Analyzer	Mitsubishi Chemical Analytech	TOX-300	H0AA0064	3/1/2015	New	WB Wet Chemistry	2279	QA Backup/Team
Turbidimeter	HACH	2100AN	9.41E+11	UNK	UNK	WB Wet Chemistry	304	QA Backup/Team
Turbidimeter	HACH	at1122	00-0E-1C-04-8C-4C	2/5/2016	New	WB Wet Chemistry	3125	QA Backup/Team
Turbidimeter	HACH	HI83414	E0039034	11/26/2014	New	WB Wet Chemistry	3114	QA Backup/Team
Turbo Vap	Biotage	TurboVap II	TV1634N21603	N/A	N/A	WB Organic Prep	3113	QA Backup/Team
Turbo Vap	Biotage	TurboVap II	TV163421605	N/A	N/A	WB Organic Prep	3112	QA Backup/Team
UF GC Analyzer	Electronic Sensor Tech	7100	UNK	UNK	UNK	WestVOA	1011	QA Backup/Team
UV-lamp	Spectroline	Q-22S	281009	UNK	UNK	WB Microbiology	1262	QA Backup/Team
Vaccum Pump	Pfeiffer	pk d41 062 G	22046576	4/1/2018	New	WestVOA	3379	QA Backup/Team
Vacuum Pump	Buchi	V-300	10028595988	1/1/2019	New	WB Organic Prep	3499	QA Backup/Team
Vortexer	VWR	10153-838	151112045	4/14/2016	New	WestVOA	3067	QA Backup/Team
Vortexer	VWR	58816-121	110307051	N/A	N/A	WB Organic Prep	1268	QA Backup/Team
Vortexer	VWR	89399-880	151007001	UNK	New	WestVOA	2331	QA Backup/Team
Vortexer	VWR	89399-880	151007002	UNK	New	WestVOA	2330	QA Backup/Team
Vortexer	VWR	9453VWALUS	C206510378	1/1/2022	New	WB Organic Prep	4628	QA Backup/Team
Vortexer	VWR	9453VWALUS	C210803066	1/1/2022	New	WB Organic Prep	4629	QA Backup/Team
Vortexer	VWR	97043-564	N/A	10/1/2020	New	WB SVOA	3944	QA Backup/Team
Vortexer	VWR	G-560	2-128130	UNK	UNK	WB SVOA	1094	QA Backup/Team
Water Bath	Fisher	2335	117204-263	N/A	N/A	WB Organic Prep	3410	QA Backup/Team
Water Bath	Fisher	Iso Temp 205	411N 0901	8/1/2005	UNK	WB Wet Chemistry	756	QA Backup/Team
Water Bath	Precision	51221036	604021381	3/1/2004	UNK	WB Microbiology	696	QA Backup/Team
Water Bath	Precision	808	202176-109	UNK	UNK	WB Microbiology	2868	QA Backup/Team

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Description	Manufacturer	Model	Serial Number	Date in Service	Condition	Location	Alpha Asset Tag	Location of Manual
Water Deionization System	Barnstead	Nanopure 2	8806030	UNK	UNK	WB Wet Chemistry	152	QA Backup/Team
Water Deionization System	Millipore	Milli-Q Direct-8	F2CIA76293 B	4/27/2015	New	WestVOA	2272	QA Backup/Team
Water Treatment Controller	Walchem	W600Series WCN610PSN NN-NN	1501080475	1/19/2015	UNK	WB Wet Chemistry	2254	QA Backup/Team
WebAlert WA500 Series	Walchem	WA500-PNN	1501080523	1/19/2015	UNK	WB Wet Chemistry	2253	QA Backup/Team
ZHE Tumbler	Analytical Testing Corp	DC-20B/DC	N/A	UNK	New	WestVOA	3284	QA Backup/Team
ZHE Tumbler	Analytical Testing Corp	DC-20B/DC	N/A	UNK	New	WestVOA	3285	QA Backup/Team
ZHE Tumbler	Analytical Testing Corp	DC-20B/DC	N/A	UNK	New	WestVOA	3282	QA Backup/Team
ZHE Tumbler	Analytical Testing Corp	DC-20B/DC	N/A	UNK	New	WestVOA	3281	QA Backup/Team
ZHE Tumbler	Analytical Testing Corp	DC-20B/DC	N/A	UNK	New	WestVOA	3280	QA Backup/Team
ZHE Tumbler	Analytical Testing Corp	DC-20B/DC	N/A	UNK	New	WestVOA	3283	QA Backup/Team

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7.5.2 PAS-MANS

Equipment List: PAS-MANS 320 Forbes Blvd. Mansfield, MA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Arsenic analyzer	PS Analytical	Millenium Excalibur 10.055	201	2/7/2007	New	MF Metals	1342	WB Tech
Autosampler	Agilent	7693	CN17040056	UNK	New	MF Forensics	3576	WB Tech
Autosampler	Agilent	G1530N	CN10652042	UNK	New	MF Forensics	1432	WB Tech
Autosampler	Agilent	G2614A	CN82549202	UNK	New	MF Forensics	3572	WB Tech
Autosampler	Agilent	G2614A	US82201034	UNK	New	MF Forensics	3573	WB Tech
Autosampler	Agilent	G2614A	CN92453237	UNK	New	MF Forensics	3577	WB Tech
Autosampler	Agilent	G4514A	R019500089	12/17/2019	New	MF Forensics	3788	WB Tech
Autosampler	Cetac	ASX-520	017566A520	UNK	New	MF Metals	3582	WB Tech
Autosampler	Cetac	ASX-520	031527A520	UNK	New	MF Metals	3583	WB Tech
Autosampler	Cetac	ASX-520	071276A520	UNK	New	MF Metals	3584	WB Tech
Autosampler	Cetac	ASX-520	120759A520	UNK	New	MF Metals	3585	WB Tech
Autosampler	Cetac	ASX-560	071736A560	UNK	New	MF Metals	3578	WB Tech
Autosampler	Cetac	ASX-560	121564A560	UNK	New	MF Metals	3579	WB Tech
Autosampler	Cetac	ASX-560	061917A560	UNK	New	MF Metals	3580	WB Tech
Autosampler	CETAC Technologies	ASX-520	1011121A120	UNK	New	MF Metals	4283	WB Tech
Autosampler	CETAC Technologies	ASX-520	041503A520	UNK	New	MF Metals	4284	WB Tech
Autosampler	PS Analytical	20.4	4703A11585	UNK	New	MF Metals	1340	WB Tech
Autosampler	SCIEX	ExionLC 2.0 Autosampler	FZC212510014	9/22/2024	New	MF EC	3870	WB Tech
Autosampler	Sciex	ExionLC 2.0 Autosampler	FZC213810002	22-Oct	New	MF EC	4642	WB Tech
Autosampler	SCIEX	ExionLC 2.0 Autosampler	FZC212510014	9/22/2024	New	MF EC	3870	WB Tech
Autosampler	Sciex	ExionLC 2.0 Autosampler	FZC213810002	22-Oct	New	MF EC	4642	WB Tech
Autosampler	SCIEX	ExionLC AD Autosampler	AB3AC5771320	12/5/2019	New	MF EC	3592	WB Tech
Autosampler	SCIEX	ExionLC AD Autosampler	AB3AC5771319	12/5/2019	New	MF EC	3598	WB Tech
Autosampler	SCIEX	ExionLC AD Autosampler	AB3AC5972606	12/21/2024	New	MF EC	4215	WB Tech
Autosampler	SCIEX	ExionLC AD Autosampler	AB3AC5972604	12/21/2024	New	MF EC	4210	WB Tech
Autosampler	SCIEX	ExionLC AD Autosampler	AB3AC5771320	12/5/2019	New	MF EC	3592	WB Tech
Autosampler	SCIEX	ExionLC AD Autosampler	AB3AC5771319	12/5/2019	New	MF EC	3598	WB Tech
Autosampler	SCIEX	ExionLC AD Autosampler	AB3AC5972606	12/21/2024	New	MF EC	4215	WB Tech
Autosampler	SCIEX	ExionLC AD Autosampler	AB3AC5972604	12/21/2024	New	MF EC	4210	WB Tech
Autosampler (LEAP)	CTC Analytics	GC PAL	1211847	5/1/1997	New	MF Forensics	1346	WB Tech

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Autosampler Controller	Agilent	7673	US71301482	UNK	New	MF Forensics	1439	WB Tech
Autosampler Controller	Agilent	7673	A000064524	UNK	New	MF Forensics	1443	WB Tech
Autosampler Tower	Agilent	G4513A	CN14240243	8/18/2014	New	MF Forensics	2122	WB Tech
Autosampler Tray	Agilent	G4514A	CN14100106	8/18/2014	New	MF Forensics	2123	WB Tech
Balance	A&D	GF2000	T0337841	4/5/2013	New	MF Metals	1934	WB Tech
Balance	AND	GF1202A	T2108383	6/28/2021	New	MF EC	4017	WB Tech
Balance	AND	GF1202A	T2108383	6/28/2021	New	MF EC	4017	WB Tech
Balance	Mettler-Toledo	PB 302	1116383231	6/10/2011	New	MF EC	1907	WB Tech
Balance	Mettler-Toledo	PB 302	1116383231	6/10/2011	New	MF EC	1907	WB Tech
Balance	Ohaus	AX523	B748064593	3/7/2018	New	MF Metals	3173	WB Tech
Balance	Ohaus	AX523	B902049357	4/19/2024	New	MF Metals	3514	WB Tech
Balance	Ohaus	AX525	C101869817	4/19/2021	New	MF Metals	4013	WB Tech
Balance	Ohaus	SJX1502N/E	CO51719854	11/15/2021	New	MF Metals	4258	WB Tech
Balance	Ohaus	V11P3	20931060	5/7/2014	New	MF Metals	2079	WB Tech
Binary Pump	Sciex	ExionLC 2.0 Binary Pump	FBT215010002	10/1/2022	New	MF EC	4641	WB Tech
Binary Pump	Sciex	ExionLC 2.0 Binary Pump	FBT215010002	10/1/2022	New	MF EC	4641	WB Tech
Chiller	Neslab	Thermo 2500	1.27E+14	9/10/2014	New	MF Metals	2139	WB Tech
Chiller	Thermo Fisher Scientific	ThermoFlex 2500	111056013	UNK	New	MF Metals	1152	WB Tech
Chiller	Thermo Fisher Scientific	ThermoFlex 900	UNK	UNK	New	MF Metals	1157	WB Tech
Chiller	Thermo Scientific	1.01E+14	1.11E+14	UNK	New	MF EC	3486	WB Tech
Chiller	Thermo Scientific	1.01E+14	1.11E+14	UNK	New	MF EC	3486	WB Tech
Chiller	Thermo Scientific	Thermoflex 9000	1.17E+15	UNK	New	MF Metals	4298	WB Tech
Chiller	Thermo Scientific	Thermoflex 9000	1.17E+15	UNK	New	MF Metals	4299	WB Tech
Chiller	Thermo Scientific	Thermoflex 9000	1.21E+14	UNK	New	MF Metals	3860	WB Tech
Chiller	Thermo Scientific	Thermoflex 9000	1.28E+15	UNK	New	MF Metals	3861	WB Tech
Chiller	Thermo Scientific	Thermoflex 9000	1.12E+15	UNK	New	MF Metals	3862	WB Tech
Chiller	Thermo Scientific	Thermoflex 9000	1.17E+15	UNK	New	MF Metals	3863	WB Tech
Chiller	Thermo Scientific	Thermoflex 9000	1.11E+15	UNK	New	MF Metals	3864	WB Tech
Chiller	Thermo Scientific	Thermoflex 9000	1.17E+15	UNK	New	MF Metals	3865	WB Tech
CHNS/O Analyzer	Perkin Elmer	2400 Series II	241N8102003	6/1/2002	New	MF Metals	1328	WB Tech
Circulator	VWR	1177PD	108601065	UNK	New	MF Metals	1156	WB Tech
Clean Benches, Class 100	Atmos Tech Industries	EL222	4132	1/1/1993	New	MF Metals	1611	WB Tech

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Clean Benches, Class 100	Laminire	LP-24	Unknown	1/1/1999	New	MF Metals	1613	WB Tech
Clean Benches, Class 100	Unknown	Unknown	Unknown	UNK	New	MF Metals	1612	WB Tech
Cleanup System	Miura	GO-6EHT	JP83260105-4	UNK	New	MF EC	3549	WB Tech
Cleanup System	Miura	GO-6EHT	JP83260105-4	UNK	New	MF EC	3549	WB Tech
Column Oven	SCIEX	ExionLC 2.0 column oven	FCC214510004	9/22/2024	New	MF EC	3871	WB Tech
Column Oven	Sciex	ExionLC 2.0 column oven	FCC214710010	10/1/2022	New	MF EC	3879	WB Tech
Column Oven	SCIEX	ExionLC 2.0 column oven	FCC214510004	9/22/2024	New	MF EC	3871	WB Tech
Column Oven	Sciex	ExionLC 2.0 column oven	FCC214710010	10/1/2022	New	MF EC	3879	WB Tech
Column Oven	SCIEX	ExionLC AD column oven	AB3C15770954	12/5/2019	New	MF EC	3589	WB Tech
Column Oven	SCIEX	ExionLC AD column oven	AB3C15770953	12/5/2019	New	MF EC	3595	WB Tech
Column Oven	SCIEX	ExionLC AD column oven	AB3CT5971355	12/21/2024	New	MF EC	4217	WB Tech
Column Oven	SCIEX	ExionLC AD column oven	ABC3CT5971363	12/21/2024	New	MF EC	4212	WB Tech
Column Oven	SCIEX	ExionLC AD column oven	AB3C15770954	12/5/2019	New	MF EC	3589	WB Tech
Column Oven	SCIEX	ExionLC AD column oven	AB3C15770953	12/5/2019	New	MF EC	3595	WB Tech
Column Oven	SCIEX	ExionLC AD column oven	AB3CT5971355	12/21/2024	New	MF EC	4217	WB Tech
Column Oven	SCIEX	ExionLC AD column oven	ABC3CT5971363	12/21/2024	New	MF EC	4212	WB Tech
Concentrator	Biotage	103187/07	TV1719N21931	UNK	New	MF EC	3502	WB Tech
Concentrator	Biotage	103187/07	TV1719N21931	UNK	New	MF EC	3502	WB Tech
Concentrator	Labanco	7810014	181268080	UNK	New	MF EC	3489	WB Tech
Concentrator	Labanco	7810014	181268080	UNK	New	MF EC	3489	WB Tech
Conditioner	Ametek	ABC15.0-20D41Y	7401564R-1720004	UNK	New	MF EC	3487	WB Tech
Conditioner	Ametek	ABC15.0-20D41Y	7401564R-1720004	UNK	New	MF EC	3487	WB Tech
Controller	SCIEX	ExionLC AD Pump	ABCBM5974510	12/21/2024	New	MF EC	4216	WB Tech
Controller	SCIEX	ExionLC AD Pump	ABCBM5974511	12/21/2024	New	MF EC	4211	WB Tech
Controller	SCIEX	ExionLC AD Pump	ABCBM5974510	12/21/2024	New	MF EC	4216	WB Tech
Controller	SCIEX	ExionLC AD Pump	ABCBM5974511	12/21/2024	New	MF EC	4211	WB Tech
Controller	SCIEX	ExionLC Controller	ASCBM5772286	12/5/2019	New	MF EC	3588	WB Tech
Controller	SCIEX	ExionLC Controller	ASCBM5772285	12/5/2019	New	MF EC	3594	WB Tech
Controller	SCIEX	ExionLC Controller	ASCBM5772286	12/5/2019	New	MF EC	3588	WB Tech
Controller	SCIEX	ExionLC Controller	ASCBM5772285	12/5/2019	New	MF EC	3594	WB Tech
Data Logger	Honeywell	Miniitrend QX	1029Y086	40724	New	MF QA	1905	

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Dishwasher	Miele	G7783	Inaccessable	UNK	New	MF Metals	1269	WB Tech
Dishwasher	Miele	G7783	Inaccessable	UNK	New	MF Metals	1270	WB Tech
Freezer	Kenmore	253.2826281	WB24838847	12/10/2012	New	MF Forensics	1910	WB Tech
GC	Agilent	5890	2950A26298	UNK	New	MF Forensics	1440	WB Tech
GC	Agilent	6890	US10218066	8/1/2002	New	MF Forensics	701	WB Tech
GC	Agilent	6890	CN10426030	8/1/2004	New	MF Forensics	1469	WB Tech
GC	Agilent	6890	CN10517033	6/1/2005	New	MF Forensics	1494	WB Tech
GC	Agilent	7890	CN17093098,	UNK	New	MF Forensics	3170	WB Tech
GC	Agilent	7890	CN10937017	10/1/2009	New	MF Forensics	1426	WB Tech
GC	Agilent	7890	US19523008	12/17/2019	New	MF Forensics	3786	WB Tech
GC	Agilent	6890N	CN10731003	8/1/2009	New	MF Forensics	1506	WB Tech
GC	Agilent	6890N	US10350004	5/1/1997	New	MF Forensics	1459	WB Tech
GC	Agilent	6890N	CN10517034	UNK	New	MF Forensics	1510	WB Tech
GC	Agilent	6890N	CN10547119	10/1/2005	New	MF Forensics	1449	WB Tech
GC	Agilent	6890N	CN10652042	2/1/2007	New	MF Forensics	815	WB Tech
GC	Agilent	6890N	US10123018	1-Jul	New	MF Forensics	666	WB Tech
GC	Agilent	6890N	US10344015	UNK	New	MF Forensics	1366	WB Tech
GC	Agilent	6890N	US10350110	UNK	New	MF Forensics	1348	WB Tech
GC	Agilent	6890N	CN10425029	UNK	New	MF Forensics	1476	WB Tech
GC	Agilent	6890N	CN10606012	3/1/2006	New	MF Forensics	1500	WB Tech
GC	Agilent	6890N	US00008300	5/1/2004	New	MF Forensics	1311	WB Tech
GC	Agilent	6890N	US10218066	UNK	New	MF Forensics	4282	WB Tech
GC	Agilent	7890A	CN11211149	UNK	New	MF Forensics	1344	WB Tech
GC	Agilent	7890A	CN12471036	5/8/2013	New	MF Forensics	1935	WB Tech
GC	Agilent	7890A	CN10850068	4/1/2009	New	MF Forensics	1429	WB Tech
GC	Agilent	7890A	CN10221048	6/10/2024	New	MF Forensics	1421	WB Tech
GC	Agilent	7890A	CN10211018	UNK	New	MF Forensics	1464	WB Tech
GC	Agilent	7890A	CN10341126	UNK	New	MF Forensics	1380	WB Tech
GC	Agilent	7890A	CN10141040	8/18/2014	New	MF Forensics	2117	WB Tech
GC	Agilent	7890A	CN12471113	7/6/1905	New	MF Forensics	2334	WB Tech
GC	Agilent	7890A	CN10929193	8/1/2009	New	MF Forensics	1483	WB Tech

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
GC	Agilent	7890A	CN10141040	5/1/2010	New	MF Forensics	1411	WB Tech
GC	Agilent	7890A	CN11261020	UNK	New	MF Forensics	1361	WB Tech
GC	Agilent	7890B	US18143006	4/1/2018	New	MF Forensics	3380	WB Tech
GC	Agilent	7890B	CN13483104	1/30/2014	New	MF Forensics	2043	WB Tech
GC	Agilent	6890	US00021356	4/1/1998	New	MF Forensics	1436	WB Tech
GC	Agilent	7890	CN10341125	9/14/2010	New	MF Forensics	1385	WB Tech
GC	Agilent	7890A	CN14173019	8/18/2014	New	MF Forensics	2121	WB Tech
Heated Coil	PS Analytical	S570U100	10	UNK	New	MF Metals	1343	WB Tech
Hg System	PS Analytical	Millenium Merlin 10.036	11	2/16/2007	New	MF Metals	1339	WB Tech
Hot Block	Environmental Express	Hotblock 150	OW5516	6/7/2021	New	MF Metals	4016	WB Tech
Hot Block	Environmental Express	SC154	2019CECW5098	6/12/2019	New	MF Metals	3525	WB Tech
Hot Block	Environmental Express	SC154	2002CEC1249	1-Jul	New	MF Metals	725	WB Tech
Hot Block	Environmental Express	SC154	4298CEC5056	3/21/2007	New	MF Metals	3174	WB Tech
Hot Block	Environmental Express	SC154	8462CE CW3635	5/28/2013	New	MF Metals	2001	WB Tech
Hot Block	Environmental Express	SC154	8462CE CW3625	5/28/2013	New	MF Metals	2002	WB Tech
Hot Block	Environmental Express	SC154	7021CECW3303	4/1/2014	New	MF Metals	3176	WB Tech
Hot Block	Environmental Express	SC154	UNK	UNK	New	MF Metals	3521	WB Tech
Hot Block	SCP Science	LS	LSX1014130215	5/1/2014	New	MF Metals	2082	WB Tech
Hot Plate	VWR	986974	130726001	UNK	New	MF Metals	3142	WB Tech
Hydrogen Generator	Parker Balston	HP2EMPD-100-100	15PLD5041	4/15/2015	New	MF Forensics	3148	WB Tech
ICP	Thermo Fisher Scientific	iCAP 6000 DUO	ICP-20081510	8-Apr	New	MF Metals	851	WB Tech
ICP	Thermo Scientific	ICAP 6500 DUO	IC5D20122909	9/1/2015	New	MF Metals	2335	WB Tech
ICP	Thermo Scientific	iCAP 6500 DUO	IC65DU131201	5/13/2013	New	MF Metals	1994	WB Tech
ICP-MS	Thermo Fisher Scientific	ICP-MSQ	03031R	9/10/2014	New	MF Metals	2140	WB Tech
ICP-MS	Thermo Fisher Scientific	ICP-MSQ	ICAP01717	UNK	New	MF Metals	3581	WB Tech
ICP-MS	Thermo Fisher Scientific	Xseries2	01925C	UNK	New	MF Metals	1150	WB Tech
ICP-MS	Thermo Scientific	iCap RQ	iCapRQ01904	UNK	New	MF Metals	3570	WB Tech
ICP-OES	Thermo Scientific	BRE731404	iCAPPRO60606	22-Nov	New	MF Metals	3876	WB Tech
Injector	Agilent	7683B	CN84459462	UNK	New	MF Forensics	3574	WB Tech
Injector	Agilent	G4513A	CN17280100	UNK	New	MF Forensics	3575	WB Tech
Ion Gauge	Agilent	59864A	1218	UNK	New	MF Forensics	1461	WB Tech

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Ion Gauge	Agilent	59864B	Unknown	UNK	New	MF Forensics	1514	WB Tech
Ion Gauge	Agilent	59864B	US60111985	UNK	New	MF Forensics	1471	WB Tech
Ion Gauge	Agilent	59864B	US60101708	UNK	New	MF Forensics	1503	WB Tech
LCMS	SCIEX	Triple Quad 4500	EB253951909	12/5/2019	New	MF EC	3587	WB Tech
LCMS	SCIEX	Triple Quad 4500	EB253891909	12/5/2019	New	MF EC	3593	WB Tech
LCMS	SCIEX	Triple Quad 4500	EB253951909	12/5/2019	New	MF EC	3587	WB Tech
LCMS	SCIEX	Triple Quad 4500	EB253891909	12/5/2019	New	MF EC	3593	WB Tech
LCMS	SCIEX	Triple Quad 5500+	EX232132111	12/21/2024	New	MF EC	4183	MF QA
LCMS	SCIEX	Triple Quad 5500+	EX232182111	12/21/2024	New	MF EC	4182	WB Tech
LCMS	SCIEX	Triple Quad 5500+	EX232132111	12/21/2024	New	MF EC	4183	MF QA
LCMS	SCIEX	Triple Quad 5500+	EX232182111	12/21/2024	New	MF EC	4182	WB Tech
LCMS	SCIEX	Triple Quad 5500+	EX234802207	9/22/2024	New	MF EC	3872	WB Tech
LCMS	SCIEX	Triple Quad 5500+	EX234802207	9/22/2024	New	MF EC	3872	WB Tech
LCMSMS	Waters	Column Manager	E16CMP407G	11/1/2016	New	MF EC	3181	WB Tech
LCMSMS	Waters	Column Manager	G18CMP447G	9/18/2018	New	MF EC	3152	WB Tech
LCMSMS	Waters	Column Manager Aux	H16CMX8446	11/1/2016	New	MF EC	3180	WB Tech
LCMSMS	Waters	Column Manager Aux	E18CMX435G	9/18/2018	New	MF EC	3153	WB Tech
LCMSMS	waters	Quaternary Solvent Manager	G16QSM637A	11/1/2016	New	MF EC	3183	WB Tech
LCMSMS	Waters	Quaternary Solvent Manager	G18QSP719A	9/18/2018	New	MF EC	3150	WB Tech
LCMSMS	Waters	sample manager FTN	g16sdi3096	11/1/2016	New	MF EC	3182	WB Tech
LCMSMS	Waters	sample manager FTN	F18FPT673G	9/18/2018	New	MF EC	3151	WB Tech
LCMSMS	Waters	TQ-S Micro	QEA0276	11/1/2016	New	MF EC	3139	WB Tech
LCMSMS	Waters	TQ-S Micro	QEB0050	9/18/2018	New	MF EC	3154	WB Tech
Liquid Limit Set	Humboldt	H-4226	2104226	8/1/2003	New	MF Metals	3523	WB Tech
Mass Spectrometer	Sciex	5500+	EX235302209	22-Oct	New	MF EC	3878	WB Tech
Mass Spectrometer	Sciex	5500+	EX235302209	22-Oct	New	MF EC	3878	WB Tech
Mercury Analyzer	CETAC Technologies	M-8000 CVAF	071201QM8	12/3/2012	New	MF Metals	1895	WB Tech
Mercury Analyzer	NIC	RA-4300A	18830145	8/7/2019	New	MF Metals	3543	WB Tech
Mercury Analyzer	Nippon Instruments Corp	RA-4300A	20830193	7/26/2021	New	MF Metals	4019	WB Tech
Mercury Analyzer	Teledyne Insturments	QuickTrace M-8000	US21193004	9/17/2021	New	MF Metals	4256	WB Tech

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Microwave	CEM	910900	MJ7964	UNK	New	MF EC	3488	WB Tech
Microwave	CEM	910900	MJ7964	UNK	New	MF EC	3488	WB Tech
Microwave	CEM	910900	MY1048	UNK	New	MF Metals	3544	WB Tech
Microwave	CEM	MARS Xpress	MD8680	7-Jan	New	MF Metals	861	WB Tech
Microwave	CEM	Mars6	MJ1475	3/1/2013	New	MF Metals	2039	WB Tech
MS	Agilent	5973	US52020185	UNK	New	MF Forensics	1511	WB Tech
MS	Agilent	5973	US80221425	6/22/2005	New	MF Forensics	772	WB Tech
MS	Agilent	5973N	US10442382	8/1/2002	New	MF Forensics	1495	WB Tech
MS	Agilent	5973N	US60542296	UNK	New	MF Forensics	1501	WB Tech
MS	Agilent	5975B	US62714677	2/1/2007	New	MF Forensics	816	WB Tech
MS	Agilent	5975C	US83111241	4/1/2009	New	MF Forensics	1430	WB Tech
MS	Agilent	5975C	US10223404	UNKJ	New	MF Forensics	1465	WB Tech
MS	Agilent	5975C	US11333920	UNK	New	MF Forensics	1386	WB Tech
MS	Agilent	5975C	US10363617	UNK	New	MF Forensics	1381	WB Tech
MS	Agilent	5975C	US92012831	8/1/2009	New	MF Forensics	1484	WB Tech
MS	Agilent	5975C	US10163301	5/1/2010	New	MF Forensics	1412	WB Tech
MS	Agilent	5975C	?US11283937	UNK	New	MF Forensics	1362	WB Tech
MS	Agilent	5977A	US1350L211	1/30/2014	New	MF Forensics	2044	WB Tech
MS	Agilent	5973	US35130153	UNK	New	MF Forensics	1455	WB Tech
MS	Agilent	5975C	US10203305	10-Jun	New	MF Forensics	1422	WB Tech
MS	Agilent	3171	US1723R002	UNK	New	MF Forensics	3171	WB Tech
MS	Agilent	5973	US33220217	5/1/1997	New	MF Forensics	1460	WB Tech
MS	Agilent	5973	US52020194	6/1/2005	New	MF Forensics	1398	WB Tech
MS	Agilent	5973 FID	US40646577	8/1/2004	New	MF Forensics	1470	WB Tech
MS	Agilent	5973N	US41746584	8/1/2004	New	MF Forensics	1477	WB Tech
MS	Agilent	5977A	US10163301	8/18/2012	New	MF Forensics	2116	WB Tech
MS	Agilent	5973	US52421036	10/1/2005	New	MF Forensics	1450	WB Tech
N-Evap	Organomation	N-EVAP 116	N3435264	UNK	New	MF EC	3547	WB Tech
N-Evap	Organomation	N-EVAP 116	N3435264	UNK	New	MF EC	3547	WB Tech
N-Evap	Organomation	N-Evap 116 (13165)	63648	12/1/2020	New	MF EC	3619	WB Tech
N-Evap	Organomation	N-Evap 116 (13165)	63648	12/1/2020	New	MF EC	3619	WB Tech
N-Evap	Organomation	N-EVAP 116, 13165	62798	UNK	New	MF EC	3537	WB Tech

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
N-Evap	Organomation	N-EVAP 116, 13165	62798	UNK	New	MF EC	3537	WB Tech
N-Evap	Organomation	OA-SYS	Unknown	UNK	New	MF EC	3177	WB Tech
N-Evap	Organomation	OA-SYS	Unknown	UNK	New	MF EC	3177	WB Tech
Non touch Data Logger	Dickson	DWE	16462806	4/5/2022	New	MF EC	4339	WB Tech
Non touch Data Logger	Dickson	DWE	16462707	4/5/2022	New	MF EC	4340	WB Tech
Non touch Data Logger	Dickson	DWE	16462545	4/5/2022	New	MF EC	4351	WB Tech
Non touch Data Logger	Dickson	DWE	16461525	4/5/2022	New	MF EC	4295	WB Tech
Non touch Data Logger	Dickson	DWE	16462806	4/5/2022	New	MF EC	4339	WB Tech
Non touch Data Logger	Dickson	DWE	16462707	4/5/2022	New	MF EC	4340	WB Tech
Non touch Data Logger	Dickson	DWE	16462545	4/5/2022	New	MF EC	4351	WB Tech
Non touch Data Logger	Dickson	DWE	16461525	4/5/2022	New	MF EC	4295	WB Tech
Non touch Data Logger	Dickson	DWE	16462583	4/5/2022	New	MF Forensics	4354	WB Tech
Non touch Data Logger	Dickson	DWE	16461526	4/5/2022	New	MF Forensics	4355	WB Tech
Non touch Data Logger	Dickson	DWE	16462779	4/5/2022	New	MF Metals	4338	WB Tech
pH Meter	Hanna Instruments	HI 2210-01	8491385	10/14/2011	New	MF Metals	1793	WB Tech
pH Meter	Orion	920	TX79A	UNK	New	MF Metals	1237	WB Tech
pH/MV/Temperature meter	Hach	H260G	150514062	UNK	New	MF Metals	2320	WB Tech
Plastic Limit tester	Humboldt	H-4226	N/A	5/3/2019	New	MF Metals	3522	WB Tech
Power Conditioner	Powervar	95208-74R	95208-74R1420012	9/10/2014	New	MF Metals	2137	WB Tech
PSA Millenium Analyzer	PSA Millenium	10.055	669	11/1/2021	New	MF Metals	4257	WB Tech
Pump	SCIEX	ExionLC 2.0 Binary Pump	FBT213510007	22-Sep	New	MF EC	4359	WB Tech
Pump	SCIEX	ExionLC 2.0 Binary Pump	FBT213510007	22-Sep	New	MF EC	4359	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5773193	12/5/2019	New	MF EC	3590	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5773194	12/5/2019	New	MF EC	3591	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5773191	12/5/2019	New	MF EC	3596	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5773192	12/5/2019	New	MF EC	3597	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5976629	21-Dec	New	MF EC	4218	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5976622	21-Dec	New	MF EC	4219	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5976630	21-Dec	New	MF EC	4213	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5676631	21-Dec	New	MF EC	4214	WB Tech

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Pump	SCIEX	ExionLC AD Pump	AB3AD5773193	12/5/2019	New	MF EC	3590	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5773194	12/5/2019	New	MF EC	3591	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5773191	12/5/2019	New	MF EC	3596	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5773192	12/5/2019	New	MF EC	3597	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5976629	21-Dec	New	MF EC	4218	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5976622	21-Dec	New	MF EC	4219	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5976630	21-Dec	New	MF EC	4213	WB Tech
Pump	SCIEX	ExionLC AD Pump	AB3AD5676631	21-Dec	New	MF EC	4214	WB Tech
Refrigerator	Frigidaire	FFRU17B2Q WD	WA94302118	1/8/2020	New	MF EC	3586	WB Tech
Refrigerator	Frigidaire	FFRU17B2Q WD	WA94302118	1/8/2020	New	MF EC	3586	WB Tech
Refrigerator	Frigidaire	FRC03L2DB1	RA62430491	10/1/2013	New	MF Forensics	2020	WB Tech
Refrigerator	Frigidaire	FRC252GB	KA90601040	10/1/2013	New	MF Forensics	2019	WB Tech
Refrigerator	Haier	BC76B	BA0142E0400BD5 5G3848	10/1/2013	New	MF Forensics	2018	WB Tech
Refrigerator	Haier	BC76B	BA0142E0400BD5 5G3848	10/1/2013	New	MF Forensics	2018	WB Tech
Refrigerator	Haier	HUM013EA	J2010600544	7/1/2002	New	MF Forensics	1518	WB Tech
Refrigerator	Kenmore	253.6041261E	BA91302482	4/15/2019	New	MF Forensics	3513	WB Tech
Refrigerator	Magic Chef	HMR440WE	YOW2004HMR44 0WE0907	9/2/2021	New	MF EC	4251	WB Tech
Refrigerator	Magic Chef	HMR440WE	YOW2004HMR44 0WE0907	9/2/2021	New	MF EC	4251	WB Tech
Refrigerator	Magic Chef	MCBR445B1	YOW1205MCBR4 45B10195	11/28/2012	New	MF Forensics	1918	WB Tech
Refrigerator	Magic Chef	MCBR445W1	0906MCBR445W1 1923	UNK	New	MF Forensics	1517	WB Tech
Refrigerator	Sears	253.6041261E	BA84826476	4/22/2019	New	MF Forensics	3515	WB Tech
Refrigerator	True Refrigeration	GDM-47-HC-LD	9506312	UNK	New	MF EC	3512	WB Tech
Refrigerator	True Refrigeration	GDM-47-HC-LD	9506312	UNK	New	MF EC	3512	WB Tech
Refrigerator	Welbilt	BC-110	UNK	UNK	New	MF Metals	1271	WB Tech
Refrigerator	Whirlpool	WRR56X18F W03	UA4405645	22-Oct	New	MF EC	3873	WB Tech
Refrigerator	Whirlpool	WRR56X18F W03	UA4405645	22-Oct	New	MF EC	3873	WB Tech
Sonicator	Fisher	Solidstate/Ultrasonic FS-28	195737	UNK	New	MF Metals	1622	WB Tech
SPE	Prochrom	SPE-03	202007002	9/1/2021	New	MF EC	4252	WB Tech
SPE	Prochrom	SPE-03	202108011	9/1/2021	New	MF EC	4253	WB Tech
SPE	Prochrom	SPE-03	202108010	9/1/2021	New	MF EC	4254	WB Tech
SPE	Prochrom	SPE-03	202007002	9/1/2021	New	MF EC	4252	WB Tech
SPE	Prochrom	SPE-03	202108011	9/1/2021	New	MF EC	4253	WB Tech

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Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
SPE	Prochrom	SPE-03	202108010	9/1/2021	New	MF EC	4254	WB Tech
Thermocouple	Fluke	51 II	497439393MV	(spare)	New	MF QA	4012	
Thermometer - Digital	SPER SCIENTIFIC	800005	80408354		New	MF Metals	1619	WB Tech
Touchscreen Data Logger	Dickson	TWE	16374983	4/5/2022	New	MF Forensics	4335	WB Tech
Touchscreen Data Logger	Dickson	TWE	16345157	4/5/2022	New	MF Metals	4336	WB Tech
Tower	Agilent	G4513A	CN19390160	12/17/2019	New	MF Forensics	3787	WB Tech
Turbidity meter	Hanna Instruments	HI88703	F0000845	6/25/2019	New	MF Metals	3529	WB Tech
Turbovap	Biotage	415001	192301123		New	MF EC	3548	WB Tech
Turbovap	Biotage	415001	192301123		New	MF EC	3548	WB Tech
Vacuum Pump	Oerlikon Leybold	Sogevac SV40BI	31000983667	9/10/2014	New	MF Metals	2141	WB Tech
Water Deionization System	Barnstead	D4641	1.09E+12	2002	New	MF Metals	868	WB Tech
Water Deionization System	Thermo Scientific	7119	383274-262	9/1/2014	New	MF Metals	2252	WB Tech

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7.5.3 PAS-MANS

Equipment List: PAS-MANS 120 Forbes Blvd. Mansfield, MA

Description	Manufacturer	Model	Serial Number	Service Date	Condition	Location	Internal ID	Location of Manual
Canister Autosampler	EnTech	7650	0080	5/1/2024	Used	120-MANS	N/A	Electronic
Canister Autosampler	EnTech	7016D	N/A	5/1/2024	Used	120-MANS	Airlab23	Electronic
Preconcentrator	EnTech	7200A	00101	5/1/2024	Used	120-MANS	Airlab23	Electronic
MS	Agilent	5977B	CABZG7081B	5/1/2024	Used	120-MANS	Airlab23	Electronic
GC	Agilent	7890B	CN18253111	5/1/2024	Used	120-MANS	Airlab23	Electronic

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8.0 ADDENDUM: PROGRAM REQUIREMENTS

Section 8.0 provides additional requirements the locations covered by this manual are required to follow when performing work under the program. Only requirements that are not covered by the main body of the manual are listed in the addendum.

8.1 DoD/DOE

PAS-MANS maintains accreditation for DoD/DoE Environmental Laboratory Approval Program (ELAP).

This addendum outlines additional policies and processes established by this laboratory to maintain compliance with DoD/DOE program specific requirements as outlined in the DoD/DOE Consolidated Quality Systems Manual (QSM) for Environmental Laboratories. The QSM incorporates ISO/IEC 17025 and the TNI Standard and includes additional program-specific requirements for laboratories that perform analytical testing services for DoD and DOE, and which must be followed for DoD / DOE projects.

Section 4.2.5: Supporting Documents

In addition to the requirements specified in Section 4.2.5, technical SOPs used for DoD/DOE testing must also include instructions for equipment and instrument maintenance, computer software/hardware, and troubleshooting.

The review frequency for technical SOPs used for DoD/DOE testing is annual, instead of every 2 years.

Section 4.4: Review of Analytical Service Requests

If the DoD/DOE customer requests a statement of conformity, the standard used for the decision rule must be communicated to and agreed on with the customer and identified in the final test report.

Laboratory requests to deviate from the requirements specified in the DoD/DOE QSM must be requested on a project-basis and include technical justifications for the deviation. These requests are submitted to and approved by the DoD/DOE project chemist or contractor, however name, in addition to the PAS client.

For DoD / DOE projects, will also seek clarification from the customer when the customer has requested an incorrect, obsolete, or improper method for the intended use of data; the laboratory needs to depart from its test method SOP in order to meet project-specific data quality objectives; information in project planning documents is missing or is unclear,

Section 4.5: Subcontracting

In addition to written client approval of any subcontractor for testing, the customer is notified of the laboratory's intent to use a subcontractor for any management system element (such as data review, data processing, project management or IT support) and consent for subcontracting is obtained approved in writing by the DoD/DOE customer and record of consent kept in the project record.

Section 4.6: Purchasing and Supplies

The laboratory procedure for records of receipt of materials and supplies used in testing also include a specification to record the date opened (DOE only).

Section 4.9.3: Nonconforming Work

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The laboratory's procedure for client notification includes the 15-business day DoD /DOE time for notification of the problem and the 30-business day timeframe for submission of the corrective action plan or corrective actions taken. This procedure also includes the DoD/DOE requirement for AB notification of discovery.

Section 4.13: Control of Records

Technical Records: The laboratory's procedure for logbooks includes measures to prevent the removal of or addition of pages to the logbook (applies to both hardcopy and electronic). Hardcopy logbooks are version controlled, pre-numbered and bound. Initials and entries are signed or initialed and dated by the person making the entry and the entry is made at the time the activity is performed and in chronological order. Each page of the logbook must be closed by the last person making the entry on the page. Closure is recorded by the initial and date of the person making the last entry.

Section 5.4.5.3.3: Limit of Detection

For DoD/DOE the LOD is an estimate of the minimum amount of an analyte that can be reliably detected by an analytical process. For clarification, the LOD is the analyte concentration necessary to distinguish its presence from its absence. The LOD may be used as the lowest concentration for reliably reporting a non-detect (ND). The LOD is specific to each suite of analyte, matrix, and method including sample preparation.

After each DL determination, the laboratory establishes the LOD by spiking a quality system matrix at a concentration of least 2X but no greater than 4X the DL (i.e., 2X DL ≤ LOD Spike ≤ 4X DL). The spike concentration establishes the LOD and the concentration at which the LOD is verified.

The LOD is established during method validation and after major changes to the analytical system or procedure that affects sensitivity of analysis or how the procedure is performed.

An LOD study is not required for any component for which spiking solutions or quality control samples are not available. Additionally, an LOD study is not required if the laboratory does not report data below the LOQ.

The LOD must be verified on a quarterly basis. Each preparation method listed on the scope of accreditation must have quarterly LOD verifications; however, verification of all combinations of preparation and clean-up techniques is not required. Where LOD verifications are not performed on all combinations, the LOD verification is based on the worst-case combination (preparation method with all applicable cleanup steps).

The laboratory's procedure for LOD determination and verification is detailed in SOP ENV-SOP-WES2-0112 Method Detection Limit & Limit of Quantitation.

Section 5.4.5.3.4: Limit of Quantitation

For DoD/DOE, the LOQ is established for each analyte-matrix-method combination, including surrogates. When an LOD is determined or verified by the laboratory, the LOQ must be above the LOD [DL<LOD<LOQ].

At a minimum, the LOQ must be verified quarterly; however, verification of all combinations of preparation and clean-up techniques is not required. Where LOQ verifications are not performed on all combinations, the LOQ verification on the worst-case combination (preparation method with all applicable cleanup steps).

The laboratory's procedure for LOQ determination and verification is detailed in laboratory SOP ENV-SOP-WES2-0112 *Method Detection Limit & Limit of Quantitation*.

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Section 5.4.7: Control of Data

The laboratory will ensure LIMS passwords are changed at least once per year.

An audit of the LIMS will be incorporated into the laboratory's annual internal audit schedule.

The laboratory will have procedures in place to notify DoD/DOE customers of changes to LIMS software or hardware configurations that may impact the customer's integrity of electronic data

Section 5.9.1: Quality Control

For DoD/DOE, storage blanks are essential QC to monitor the storage of samples for volatile organic analysis (VOA). The SOP for storage of VOA samples must include a contamination monitoring program based on the performance of storage blanks. (See QSM 5.3.3)

Section 5.8.5: Sample Disposal

For DOE projects, the record of disposal must also include how the sample was disposed and the name of the person that performed the task.

Appendix E: Support Equipment Calibration

Mechanical Volumetric Pipette: In addition to the quarterly verification check, pipettes used for DoD/DOE projects are checked daily before use using the same procedure and criteria specified for the quarterly check.

Water Purification System: The performance of the water purification system is checked daily prior to use in accordance with SOP ENV-SOP-WES2-0127 *DI Water System*.

Additional: (DOE): Section 6.0 of the QSM outlines additional management system requirements for the management of hazardous and radioactive materials management and health and safety practices. The laboratory, if approved for DOE, will consult with the PAS Health and Safety Director to establish plans, policies and procedures that conform to these comprehensive specifications and incorporate these documents into the QMS.

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8.2 Unregulated Contaminant Monitoring Rule 5 (UCMR5)

PAS-MANS maintains EPA approval to report data for the Unregulated Contaminant Monitoring Rule 5. This addendum outlines additional policies and processes established by this laboratory to maintain compliance with UCMR 5 program specific requirements. These requirements are outlined in the UCMR 5 Laboratory Approval Requirements and Information Document. Data may not be uploaded to the Safe Drinking Water Accession and Review System (SDWARS) if it does not comply with these requirements.

Section 5.8.3: Sample Receipt Checks and Sample Acceptance Policy

Samples cannot be reported to SDWARS for UCMR5 Compliance if any of the following receipt issues are encountered: received in an insecure manner, inappropriate containers, outside the required temperature range, outside the recognized holding time, inadequate identification on sample containers and/or COC, improper preservation.

Section 5.8.3.1: Sample Receipt Checks - Thermal Preservation

Samples must be received at the laboratory within 48 hours of collection and must be ≤ 10˚C. After arrival or within 48 hours of collection, whichever is sooner, the samples must be refrigerated at ≤ 6°C. Samples received more than 48 hours after collection are valid only if they are received at ≤ 6°C and the laboratory can obtain documentation from the client to verify that they were refrigerated between collection and shipment. Temperature requirements apply to all UCMR5 samples with the exception of Lithium by EPA 200.7.

Section 5.9.1.1.3: Method Blank

The method blank will be considered contaminated if the concentration of any target analyte in the method blank is greater than 1/3 the MRL for that analyte. If samples cannot be re-processed, then samples must be re-collected. Data with a failing method blank cannot be reported to SDWARS.

Section 5.9.1.1.4: Laboratory Control Sample (LCS)

If the LCS recovery fails, then all data for the problem analyte are considered invalid for all samples in the Extraction or Digestion Batch. Data associated with a failing LCS must not be reported to SDWARS. If the LCS surrogate or internal standard fails QC criteria, all data for the Extraction or Digestion Batch must not be reported to SDWARS.

Section 5.9.1.1.5: Matrix Spikes (MS/MSD)

A UCMR5 sample must be used as the parent sample for the MS/MSD in batches containing UCMR5 samples. The spike concentration of the MS/MSD must be alternated between a low- and mid-level concentration.

There are no method analyte recovery acceptance criteria specific for MS/MSD results. Data should not be rejected based on the recovery of the fortified target analytes or on the precision between the MS and MSD, provided internal standard and surrogate standard recoveries and all other QC results are valid. If surrogate or internal standard recovery fails criteria in the MS and MSD, do not report results. If one of the fortified samples fails QC, the results of the passing fortified sample should be reported as the MS. The entire analysis batch does not need to be reanalyzed because of a failure of a surrogate or internal standard in MS or MSD. Recollection of the field sample is not required with a surrogate or internal standard fails criteria in the MS/MSD.

Section 5.9.1.1.7: Surrogates

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A sample that fails QC criteria for surrogate recovery should be rerun in case a mis-injection occurred. If the sample continues to fail surrogate QC criteria, the laboratory should process a new aliquot of the sample. Typically, data for samples with failed surrogate recovery must not be reported to SDWARS.

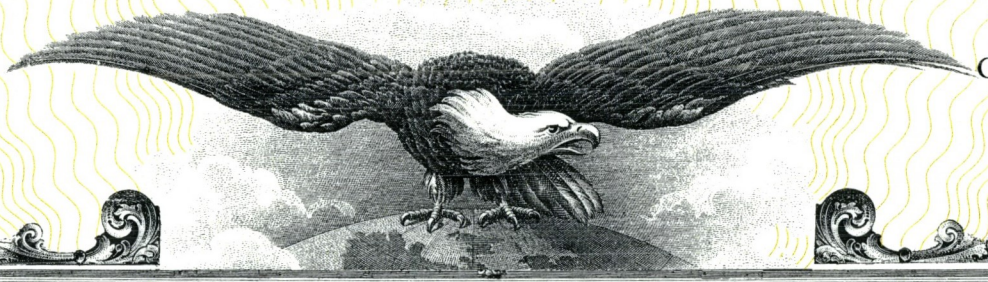
If the surrogate recovery fails the acceptance criteria in a MB or LCS, the MB or LCS may be reanalyzed later in the Analysis Batch. If surrogate recovery fails again, all field sample results for the associated Extraction Batch are invalid and must not be reported. If the surrogate recovery fails in an MB that is needed to pass an Analysis Batch, all data for that Analysis Batch are invalid and must not be reported. If possible, the laboratory should process new aliquots of the affected samples; if this is not possible, the affected field samples must be recollected. One LCS must pass surrogate recovery within a 24-h period of analysis. If not, the samples in the Analysis Batches that correspond to that required LCS, cannot be reported.

Section 5.9.1.1.8: Internal Standards

If the internal standard recovery in a sample fails the acceptance criteria, then the results for the analytes that are associated with the internal standard in that sample are invalid. Data that fail internal standard criteria must not be reported to SDWARS. If possible, the laboratory should process a new aliquot of the sample; if this is not possible, the field sample must be recollected.

For an Extraction Batch or Digestion Batch, if the internal standard recoveries in a MB or LCS fail the acceptance criteria, all data for the analytes that are associated with that internal standard in that Extraction Batch or Digestion Batch are invalid and must not be reported. If the internal standard recoveries in a MB or LCS fail the acceptance criteria in an Analysis Batch, all data for the analytes that are associated with that internal standard in the Analysis Batch are invalid and must not be reported. If possible, the laboratory should process new aliquots of the affected samples.

Accreditation #:
GAI-LAP – 20-96
TEL (610) 522-8440



Geosynthetic Institute
475 Kedron Avenue
Folsom, PA 19033

GeoTesting Express, LLC

Is granted accreditation
for designated geosynthetic test methods in accordance with the
Geosynthetic Accreditation Institute – Laboratory Accreditation Program
(GAI-LAP), as published in its annual directory.
This accreditation is valid until June 30, 2025.

Jamie Roth Koerner
General Manager

George R. Koerner, Ph.D., P.E. & CQA
Director

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Geosynthetic Institute
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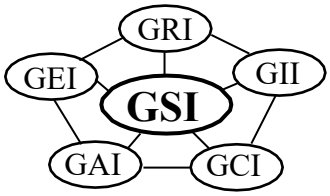


June 25, 2024

Mr. Michael Pratt
Geotesting Express, LLC
125 Nagog Park
Acton, MA 01720

REVISION

Re: GAI-LAP

Dear Mr. Michael Pratt:

The Geosynthetic Institute (GSI) is pleased to acknowledge Geotesting Express LLC on its repertoire of Geosynthetic Accreditation Institute's-Laboratory Accreditation Program (GAI-LAP) accredited tests. This letter should serve as notification that Geotesting Express LLC located in Acton, MA. Geotesting Express is currently accredited for the following sixty-three (63) test methods until June 30, 2025.

1. ASTM D374 Test Methods for Thickness of Solid Electrical Insulation
2. ASTM D413 Test Methods for Rubber Property – Adhesion to Flexible Substrate
3. ASTM D751 Test Methods for Coated Fabrics (thickness)
4. ASTM D792 Test Method for Specific Gravity and Density of Plastics by Displacement
5. ASTM D882 Test Method for Tensile Properties of strip tensile
6. ASTM D1004 Test Method for Initial Tear Resistance of Plastic Film and Sheeting
7. ASTM D1204 Test Method for Linear Dimensional Changes of Nonrigid Thermoplastic Sheeting or Film at Elevated Temperature
8. ASTM D1238 Test Method for Flow Rates of Thermoplastics by Extrusion Plastometer
9. ASTM D1505 Test Method for Density of Plastics by the Density Gradient Technique
10. ASTM D1593 Specification for Nonrigid Vinyl Chloride Plastic Sheeting (thickness)
11. ASTM D1603 Test Method for Carbon Black in Olefin Plastics
12. ASTM D1777 Test Method for Measuring the Thickness of Textile Materials
13. ASTM D3015 Standard of Practice for Microscopic Examination of Pigment Dispersion in Plastic Compounds
14. ASTM D3776 Test Method for Mass Per Unit Area (Weight) or Woven Fabric
15. ASTM D3786 Test Method for Hydraulic Burst Strength of Knitted Goods and Nonwoven Fabrics (Diaphragm Bursting Strength Tester Method)
16. ASTM D3787 Test Method for Bursting strength using CRT Bell Burst Test
17. ASTM D4218 Test Method for Carbon Black in Olefin Plastics (Muffle Furnace)

18. ASTM D4491 Test Methods for Water Permeability of Geotextiles by Permittivity
19. ASTM D4533 Test Method for Index Trapezoidal Tearing Strength of Geotextiles
20. ASTM D4595 Standard Test Method for Tensile Properties of Geotextiles by Wide Width Strip Method
21. ASTM D4632 Test Method for Grab Breaking Load and Elongation of Geotextiles
22. ASTM D4716 Test Method for Determining the (In-Plane) Flow Rate per Unit Width and Hydraulic Transmissivity of a Geosynthetic Using a Constant Head
23. ASTM D4751 Test Method for Determining Apparent Opening Size of a Geotextile
24. ASTM D4833 Test Method for Index Puncture Resistance of Geotextiles, Geomembranes and Related Products
25. ASTM D4884 Test Method for Seam Strength of Sewn Geotextiles
26. ASTM D4885 Test Method for Determining Performance Tensile Strength of Geomembranes Using Wide Strip Testing
27. ASTM D5034 Test Method for Breaking Strength and Elongation (Grab Method)
28. ASTM D5035 Test Method for Breaking Strength and Elongation (Strip Method)
29. ASTM D5199 Test Method for Measuring Nominal Thickness of Geotextiles and Geomembranes
30. ASTM D5261 Test Method for Measuring Mass per Unit Area of Geotextiles
31. ASTM D5321 Test Method Using Direct Sheer Method to Determine Soil and Geosynthetic Friction
32. ASTM D5596 Test Methods for Microscopic Evaluation of the Dispersion of Carbon Black in Polyolefin Geosynthetics
33. ASTM D5884 Test Method for the Tearing Strength of Internally Reinforced Geomembranes
34. ASTM D5887 Test Method to Measure Index Flux of Saturated GCLs
35. ASTM D5890 Standard Test Method for Swell Index of Clay Mineral Component of Geosynthetic Clay Liners
36. ASTM D5891 Standard Test Method for Fluid Loss of Clay Component of GCL
37. ASTM D5993 Test Method for Measuring the Mass Per Unit Area of GCL
38. ASTM D5994 Test Method for Measuring the Core Thickness of Textured Geomembranes
39. ASTM D6140 Test Method to Determine Asphalt Retention of Paving Fabrics used in Asphalt Paving
40. ASTM D6214 Test Method for Determining the Integrity of Field Seams Used in Joining Geomembranes by Chemical Fusion Methods
41. ASTM D6241 Test Method for the Static Puncture Strength of Geotextiles and Geotextile Related Products Using a 50-mm Probe
42. ASTM D6243 Test Method for Determine the Internal and Interface Shear Resistance of Geosynthetic Clay Liners by the Direct Shear Method
43. ASTM D6364 Test Method for Determining the Short-Term Compression Behavior of Geosynthetics
44. ASTM D6392 Test Method for Determining the Integrity of Nonreinforced GM Seams using Thermo Fusion Method

45. ASTM D6496 Test Method to Determine the Average Bonding Peel Strength Between Layers of Needle-Punched GCL.
46. ASTM D6566 Test Method for Measuring Mass per Unit Area of Turf Reinforcement Mats
47. ASTM D6636 Test Method for Determining Ply Adhesion Strength of reinforced Geomembranes
48. ASTM D6637 Test Method for determining Tensile Properties of Geogrids by single or multi rib Tensile Method
49. ASTM D6693 Test Method of Determining Tensile Properties of Nonreinforced Polyethylene and Nonreinforced Flexible Polypropylene GM
50. ASTM D6766 Test Method for Evaluation of Hydraulic Properties of Geosynthetic Clay Liners Permeated with Potentially Incompatible Liquids
51. ASTM D6768 Test Method for Tensile Strength of GCL
52. ASTM D7003 Test Method for Strip Tensile Properties of reinforced GM
53. ASTM D7004 Test Method for Grab Tensile Properties of Reinforced GM
54. ASTM D7005 Test Method for Determining the Bond Strength of Geocomposites
55. ASTM D7179 Test Method for Determining Geonet Breaking Force
56. ASTM D7408 Standard Test for Non-resin PVC geomembrane seams
57. ASTM D7466 Test Method for Measuring the Asperity Height of Textured Geomembranes
58. ASTM D7737 Test Method for Individual Geogrid Junction Strength
59. ASTM D7747 Test Method for Determining Integrity of Seams for reinforced Geomembranes using Strip Tensile Method
60. ASTM D7749 Test Method for Determining Integrity of Seams for reinforced Geomembranes using Grab Method
61. ASTM C165 Standard Test Methods for Measuring Compressive Properties of Thermal Insulations
62. ASTM C203 Standard Test Methods for Breaking Load and Flexural Properties of Block-Type Thermal Insulation
63. ASTM C303 Standard Test Method for Dimensions and Density of Preformed Block and Board-Type Thermal Insulation

Any questions regarding your accreditation should be directed to George Koerner at (610) 522-8440. Once again congratulation and thank you for participating in the GAI-LAP.

Regards,

George R. Koerner, Ph.D., P.E. & CQA
Director

Accredited Laboratory

A2LA has accredited

GEOTESTING EXPRESS, LLC.

Acton, MA

for technical competence in the field of

Geotechnical Testing

This laboratory is accredited in accordance with the recognized International Standard ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories. This accreditation demonstrates technical competence for a defined scope and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated April 2017).

Presented this 29th day of April 2024.

Mr. Trace McInturff, Vice President, Accreditation Services
For the Accreditation Council
Certificate Number 2965.01
Valid to March 31, 2026
Revised September 10, 2024

For the tests to which this accreditation applies, please refer to the laboratory's Geotechnical Scope of Accreditation.

SCOPE OF ACCREDITATION TO ISO/IEC 17025:2017

GEOTESTING EXPRESS, LLC.
125 Nagog Park
Acton, MA 01720
Joe Tomei Phone: 978 635 0424

Valid To: March 31, 2026 Certificate Number: 2965.01

GEOTECHNICAL

In recognition of the successful completion of the A2LA evaluation process, accreditation is granted to this laboratory to perform the following tests under the ASTM recommended practice D3740:

Test Method:	**Test Description:**
Soils:	
AASHTO T307	Determining the Resilient Modulus of Soils and Aggregate Materials
ASTM D421 (Withdrawn 2016)	Dry Preparation of Soil Samples for Particle-Size Analysis and Determination of Soil Constant
ASTM D422 (Withdrawn 2016)	Particle Size Analysis of Soils
ASTM D698	Moisture-Density Relations (Standard Proctor)
ASTM D854	Specific Gravity of Soils
ASTM D1140	Amount of Material in Soils Finer than No. 200 Sieve
ASTM D1557	Moisture-Density Relations (Modified Proctor)
ASTM D1883	CBR (California Bearing Ratio) of Laboratory-Compacted Soils
ASTM D2166/D2166M	Unconfined Compressive Strength of Cohesive Soil
ASTM D2216	Water Content of Soil, Rock & Soil-Aggregate Mixtures
ASTM D2434	Permeability of Granular Soils (Constant Head)
ASTM D2435/D2435M	One-Dimensional Consolidation Properties of Soils
ASTM D2487	Classification of Soils for Engineering Purposes
ASTM D2488	Description and Identification of Soils (Visual-Manual Procedure)
ASTM D2850	Undrained, Unconsolidated Strength in Triaxial Compression
ASTM D2974	Moisture, Ash, and Organic Matter of Peat and Other Organic Soils
ASTM D3080/D3080M	Direct Shear Test of Soils Under Consolidated Drained Conditions
ASTM D3999	Determination of the Modulus and Damping Properties of Soils Using the Cyclic Triaxial Apparatus
ASTM D4186	One-Dimensional Consolidation Properties of Saturated Cohesive Soils Using Controlled-Strain Loading
ASTM D4253	Maximum Index Density and Unit Weight of Soils Using a Vibratory Table
ASTM D4254	Minimum Index Density and Unit Weight of Soils and Calculation of Relative Density
ASTM D4318	Liquid Limit, Plastic Limits & Plasticity Index of Soils
ASTM D4373	Rapid Determination of Carbonate Content of Soils
ASTM D4546	One-Dimensional-Swell or Settlement Properties of Cohesive Soils

5202 Presidents Court, Suite 220 | Frederick, MD 21703-8398 | Phone: 301 644 3248 | Fax: 240 454 9449 | www.A2LA.org

Test Method:	**Test Description:**
ASTM D4718	Correction of Unit Weight and Water Content for Soils Containing Oversize Particles
ASTM D4767	Consolidated Undrained Triaxial Compression Test for Cohesive Soils
ASTM D4829	Expansion Index of Soils
ASTM D4972	pH of Soils
ASTM D5084	Measurement of Hydraulic Conductivity of Saturated Porous Materials Using a Flexible Wall Permeameter
ASTM D5334	Thermal Conductivity of Soil and Soft Rock by Thermal Needle Probe Procedure
ASTM D5311	Load Controlled Cyclic Triaxial Strength of Soil
ASTM D6467	Torsional Ring Shear Test to Determine Drained Residual Shear Strength of Cohesive Soils
ASTM D6528	Consolidated Undrained Direct Simple Shear Testing of Cohesive Soils
ASTM D6913	Particle-Size Distribution (Gradation) of Soil using Sieve Analysis
ASTM D6938	In-Place Density and Water Content of Soil and Soil-Aggregate by Nuclear Methods (Shallow Depth)
ASTM D7181	Consolidated Drained Triaxial Compression Test for Soils
ASTM D7263	Laboratory Determination of Density (Unit Weight) of Soil Specimens
ASTM D7608	Standard Test Method for Torsional Ring Shear test to determine Drained Fully Softened Shear Strength and Nonlinear Strength Envelope of Cohesive Soils for Slope with No Preexisting Shear Surfaces
ASTM D7928	Particle-Size Distribution (Gradation) of Fine-Grained Soils Using the Sedimentation (Hydrometer) Analysis
ASTM D8296	Consolidated Undrained Cyclic Direct Simple Shear Test under Constant Volume with Load Control or Displacement Control
ASTM G51	Standard Test Method for Measuring pH of Soil for Use in Corrosion Testing
ASTM G57	Soil Resistivity Using the Wenner Four-Electrode Method
IEEE-STD-422-17	IEEE Guide for Thermal Resistivity Measurements of Soils and Backfill Materials
ISO/TS 17892-1	Determination of Water Content
ISO/TS 17892-3	Determination of Particle Density - Pycnometer Method
ISO/TS 17892-5	Incremental Loading Oedometer Test
ISO/TS 17892-7	Unconfined Compression Test on Fine-Grained Soil
ISO/TS 17892-9	Consolidated Triaxial Compression Tests on Water-Saturated Soil
ISO/TS 17892-11	Determination of Permeability by Constant and Falling Head
ISO/TS 17892-12	Determination of Atterberg Limits
Rock:	
ASTM D2845	Laboratory Determination of Pulse Velocities and Ultrasonic Elastic Constants of Rock
ASTM D2936	Direct Tensile Strength of Intact Rock Core Specimens
ASTM D3967	Splitting Tensile Strength of Intact Rock Core Specimens
ASTM D4543	Preparing Rock Core as Cylindrical Test Specimens and Verifying Conformance to Dimensional and Shape Tolerances
ASTM D4644	Slake Durability of Shales and Similar Weak Rocks

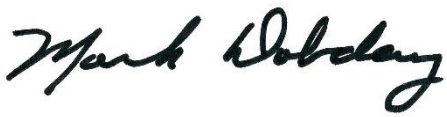
Test Method:	**Test Description:**
ASTM D5607	Performing Laboratory Direct Shear Strength Tests of Rock Specimens Under Constant Normal Force
ASTM D5731	Determination of the Point Load Strength Index of Rock and Application to Rock Strength Classifications
ASTM D5873	Determination of Rock Hardness by Rebound Hammer Method
ASTM D6032	Determining Rock Quality Designation (RQD) of Rock Core
ASTM D7012	Compressive Strength and Elastic Moduli of Intact Rock Core Specimens under Varying States of Stress and Temperature
ASTM D7625	Laboratory Determination of Abrasiveness of Rock Using the CERCHAR Method
Handewith (2000)	Punch Penetration
ISRM Part 1	Water Content of Rock
ISRM Part 2	Porosity/Density
ISRM Part 3	Saturation/Buoyancy

Quality Manual

Revision 14.1 – per ISO 17025:2017

April 8, 2021

CONTROLLED DOCUMENT No QM 14-1-2



_____

Written By: Mark Dobday – Laboratory Consultant

_____

Approved By: Joe Tomei – Vice President & Director of Testing Services

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1 SCOPE

Presented in this document is the Quality Manual for GeoTesting Express, Inc. (GTX). This document compliments other referenced documents which are specific to the laboratory's operation. The quality system outlined in this Quality Manual is designed to meet requirements in ANSI/ISO/IEC 17025:2017. The organization of the GTX Quality Manual follows the organization structure of ANSI/ISO/IEC 17025:2017.

This document may also be used to confirm or recognize the competence of the GTX laboratories by laboratory customers, regulatory authorities, organizations, and schemes using peer-assessment, accreditation bodies, and others.

Created in 1989, GTX is an independent testing laboratory with locations in Acton, MA and Atlanta, GA. GTX is a subsidiary of Geocomp Corporation (Geocomp). GTX is committed to adhering to high standards of quality while providing quick turnaround of test results for soil, rock, geosynthetics, aggregate, concrete, and other "geo" materials. GTX services owners, engineers, contractors, developers, manufacturers, government agencies and other laboratories.

The GTX quality goals can be found in Section 8.

1.1 HEALTH SAFETY AND ENVIRONMENTAL POLICY

It is the policy and practice of the Geocomp and GTX to control hazards in the workplace to the greatest extent feasible. This is to reduce the potential for employee injury and illness as well as to protect potentially affected members of the public, their property, and the environment from loss or damage due to our operations and activities. Geocomp/GTX is committed to the safety and health of all employees and recognizes the need to, at a minimum, comply with regulations governing employee workplace health and safety. When feasible and/or consistent with good management practice, we will always strive to operate in a manner that is "beyond compliance."

The GTX Quality Manual does not address health and safety issues and policies. These matters are addressed by the corporate Health and Safety Program and are beyond the scope of the GTX Quality Manual. Company policies are outlined in the Geocomp Corporation "Health, Safety and Environmental Program."

2 NORMATIVE REFERENCES

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

This quality manual contains references to the following documents:

- ISO/IEC Guide 99, International vocabulary of metrology — Basic and general concepts and associated terms (VIM)1)

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- ISO/IEC 17000-2020, Conformity assessment — Vocabulary and general principles
- ANSI/ASQC Q9001-2015 Quality Management Systems – Requirements
- ISO/IEC Guide 17025:2017 General Requirement for the Competence of Testing and Calibration Laboratories
- ISO/IEC Guide 17025: 2005 General Requirement for the Competence of Testing and Calibration Laboratories
- ASTM D3740 Standard Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction
- American Association of State Highway and Transportation Officials (AASHTO) R 18-17. Standard Recommended Practice for Establishing and Implementing a Quality System for Construction Materials Testing Laboratories
- American Society for Testing and Materials (ASTM) Test Methods and Standards of Practice
- American Association of State Highway and Transportation Officials (AASHTO) Test Methods
- United States Army Corps of Engineers Laboratory Soils Testing Test Standards EM 1110-2-1906 (1970)
- Federal Test Method Standard Number 101C, Method 2065 (1980)
- Geosynthetic Research Institute Standard Test Methods
- A2LA P102 – A2LA Policy on Metrological Traceability
- A2LA P103 - Policy on Estimating Measurement Uncertainty for Testing Laboratories
- A2LA P103d - Annex - Policy on Measurement Uncertainty for Construction Materials and Geotechnical Testing Laboratories.
- A2LA R103 – General Requirements: Proficiency Testing for ISO/IEC 17025 Laboratories
- A2LA R103a – Annex – Proficiency Testing for ISO-IEC 17025 Laboratories
- A2LA R105 – Requirements When Making Reference to A2LA Accredited Status
- A2LA R105 - Supplement - Promoting Accreditation Package
- A2LA G103 – A2LA Guide for Estimation of Uncertainty of Dimensional Calibration and Testing Results
- A2LA G104 – Guide for Estimation of Measurement Uncertainty in Testing
- ASTM International D18 Standards Preparation Manual Revision 3 6/27/2018
- ASTM D653 - Standard Terminology Relating to Soil, Rock, and Contained Fluids
- GeoTesting Express, Inc. Standard Terms and Conditions for Engineering, Testing and Consulting Services 2010

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3 TERMS AND DEFINITIONS

For the purposes of this document, the following terms and definitions given in ISO/IEC 17000 and ISO/IEC Guide 99 and the following apply.

3.1 Impartiality - presence of objectivity.

3.2 Complaint - expression of dissatisfaction by any person or organization to a laboratory (3.6), relating to the activities or results of that laboratory, where a response is expected.

3.3 Interlaboratory Comparison - organization, performance and evaluation of measurements or tests on the same or similar items by two or more laboratories in accordance with predetermined conditions.

3.4 Intralaboratory Comparison - organization, performance and evaluation of measurements or tests on the same or similar items, within the same laboratory (3.6), in accordance with predetermined conditions.

3.5 Proficiency Testing - evaluation of participant performance against pre-established criteria by means of interlaboratory comparisons (3.3).

3.6 Laboratory - body that performs one or more of the following activities: Testing, calibration, and sampling associated with subsequent testing or calibration.

3.7 Decision Rule - rule that describes how measurement uncertainty is accounted for when stating conformity with a specified requirement.

3.8 Verification - Provision of objective evidence that a given item fulfils specified requirements.

3.9 Validation - Verification (3.8) where the specified requirements are adequate for an intended use.

3.10 AASHTO: n – American Association of State Highway and Transportation Officials. AASHTO is a nonprofit, nonpartisan association representing highway and transportation departments in the 50 states, the District of Columbia and Puerto Rico. It provides standard test methods or specifications that have been developed by and meets the approval of the organization.

3.11 ASTM: n – American Society for Testing and Materials. A not-for-profit organization which provides standard test methods or specifications that have been developed within the consensus principles and that meets the approval requirements for the organization.

3.12 Client: n – The company or individual responsible for payment of delivered product or services.

3.13 Contract: n – Formal or verbal agreement between the Client and the supplier with the terms and requirements for the testing services detailed. The contract terms may include specific testing services, along with the agreed price, and delivery schedule.

3.14 Geosynthetic: n – a planar product manufactured from polymeric material used with soil, rock, earth, or other geotechnical engineering related material as an integral part of a man-made project, structure or system.

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3.15 ISO: n – International Standards Organization for Standardization (ISO) which specifies the quality systems requirements.

3.16 IRM: n – Internal Reference Materials are standard materials of known quality or whose quality has previously been established by the laboratory and are used to validate test methods, equipment performance and to qualify technicians.

3.17 Product: n – The final report submitted to the client describing the test results and test data.

3.18 Quality Assurance: n – The system of testing, inspection, review, documentation, etc. to verify the existence, implementation, and maintenance of the Quality System.

3.19 Quality Control: n – The system of testing, inspection, review, and documentation which are designed to verify that the data generated in the laboratory are produced within known limits of accuracy and precision.

3.20 Quality Policy: n – The documented quality commitment and objectives of the supplier.

3.21 Quality System: n – The entire quality program consisting of quality control, quality assurance, and management programs to verify that the company's Quality Policy and requirements of the client are achieved.

3.22 Sample: n – The material delivered to the laboratory as a source of test specimens.

3.23 Specimen: n – A specific portion of a sample upon which a test is performed. Typically, specimens must be specified dimensions, volume or weight.

3.24 In this document, the following verbal forms are used:

— "shall" indicates a requirement;

— "should" indicates a recommendation;

— "may" indicates a permission;

— "can" indicates a possibility or a capability.

3.25 Definitions of technical terminology used by GTX follows ASTM D653 - Standard Terminology Relating to Soil, Rock, and Contained Fluids.

4 GENERAL REQUIREMENTS

4.1 IMPARTIALITY

GTX is an impartial, 3rd party laboratory whose employees are free from undue pressures which could influence their technical decisions. GTX is a subsidiary of Geocomp Corporation. Personnel of GTX are independent from the other divisions of Geocomp. The other divisions of Geocomp do not exert influence upon the GTX laboratory or its employees.

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4.1.1

GTX activities shall be undertaken impartially and structured and managed to safeguard impartiality.

4.1.2

The GTX laboratory management shall be committed to impartiality.

4.1.3

The GTX laboratory shall be responsible for the impartiality of its laboratory activities and shall not allow commercial, financial, or other pressures to compromise impartiality.

4.1.4

The GTX laboratory shall identify risks to its impartiality on an on-going basis. This shall include those risks that arise from its activities, or from its relationships, or from the relationships of its personnel. However, such relationships do not necessarily present a laboratory with a risk to impartiality.

4.1.5

If a risk to impartiality is identified, GTX shall eliminate and minimize such risk as outlined in Section 8.5.4.

4.2 CONFIDENTIALITY

The GTX Standard Terms and Conditions for Engineering, Testing, and Consulting Services relate to ownership and confidentiality of GTX results and reports. Section 11 of Terms and Conditions states the following:

Confidentiality and Subpoenas. We will not tell anybody about this work unless someone's health or safety is in danger, or the Sheriff delivers a demand. Information about this Agreement and GTX's services and information provided by the Client about the services will be maintained in confidence and will not be disclosed to others without the Client's consent, except as GTX believes is necessary to perform its services, comply with professional standards to protect public safety, health, and the environment, and to comply with laws and court orders. GTX will make reasonable efforts to notify the Client prior to any disclosure except as necessary to perform its services. Information available to the public and unprotected information acquired from third parties will not be considered confidential. The Client will reimburse GTX to respond to any subpoena or governmental inquiry or audit related to the services at GTX's standard rates then in effect.

4.2.1

The GTX laboratory shall be responsible, through legally enforceable commitments, for the management of all information obtained or created during the performance of laboratory activities. The laboratory shall inform the client in advance, of the information it intends to place in the public domain. Except for information that the client makes publicly available, or when agreed between the laboratory and the client (e.g. for the purpose of responding to complaints), all other information is considered proprietary information and shall be regarded as confidential.

4.2.2

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When the laboratory is required by law or authorized by contractual arrangements to release confidential information, the client or individual concerned shall, unless prohibited by law, be notified of the information provided.

4.2.3

Information about the client obtained from sources other than the client (e.g. complainant, regulators) shall be confidential between the client and the laboratory. The provider (source) of this information shall be confidential to the laboratory and shall not be shared with the client, unless agreed by the source.

4.2.4

Personnel, including any committee members, contractors, personnel of external bodies, or individuals acting on the laboratory's behalf, shall keep confidential all information obtained or created during the performance of laboratory activities, except as required by law.

5 STRUCTURAL REQUIREMENTS

5.1 GEOCOMP AND GEOTESTING EXPRESS

GeoTesting Express, Inc. is a subsidiary of Geocomp Corporation and is incorporated in the Commonwealth of Massachusetts. GTX's relationship to the Geocomp Corporation and its reporting structure to its parent company is outlined in the organization chart presented in Appendix A, Figure 1.

5.2 GTX ORGANIZATION

The organization chart of GTX is presented in Appendix A, Figure 2. It outlines the management structure and overall laboratory responsibilities. See Section 5.5 for further details of the GTX organization, management, and responsibilities.

5.3 LABORATORY ACTIVITIES

All GTX laboratory activities shall conform with the GTX Quality Manual. A list of GTX laboratory activities can be found in Appendix B. GTX will only claim conformity for its own range of laboratory activities. GTX will not claim conformity to its quality system for any externally provided laboratory activities performed by others.

5.4 REQUIREMENTS FOR LABORATORY ACTIVITIES

All GTX activities shall be carried out in such a way as to meet the requirements of the ISO/IEC 17025 as well as the requirements of GTX clients, relevant regulatory authorities and organizations providing recognition. This shall include GTX activities performed in all its permanent facilities, at sites away from its permanent facilities, in any associated temporary or mobile facilities or at a client's facility.

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5.5 ORGANIZATION

5.5 a)

GTX is a subsidiary of Geocomp Corporation. The Geocomp leadership consists of the Chief Engineer who is also the Chief Executive Officer (CEO) and the owner of the firm, and the Chief Operating Officer (COO.)

GTX's leader is the Director of Testing Services who reports directly to the COO.

5.5 b) Laboratory Personnel, Their Responsibilities, Authority and Interrelationships

5.5.b.1

GTX is committed to providing the necessary resources to achieve its stated goals. This includes:

- Trained personnel to manage and perform testing as outlined by appropriate standards
- Equipment with capabilities, accuracy and precision as prescribed by appropriate standards
- Computers necessary for data acquisition, analysis, report generation, accounting and marketing
- Facilities necessary to conduct testing in appropriate atmosphere

5.5.b.2

All GTX Laboratory personnel are responsible for the following:

Following Health, Safety and Environmental policy and project specific Health and Safety plans

Client confidentiality

Identifying and reporting risk to the GTX quality system, integrity and impartiality.

The specific responsibilities and authorities for each position are outlined in Appendix C as part of the job description for each lab position.

5.5.b.3

Position descriptions for each operational position in the laboratory/field staff can be found in Appendix C. Resumes of the supervisory technical staff (Chief Engineer/CEO, Director of Testing Services and Laboratory Manager(s)) are located with the Director of Testing Services.

5.5 c) Management System

GTX employs a management team appropriate to the volume and nature of laboratory testing conducted. GTX maintains documented procedures which make up the quality system and are located in the Quality Manual and other quality system documents. These procedures have been developed to assure the consistent application of laboratory activities and the validity of the results. The procedures are followed by employees of GTX. The Quality Manager is responsible for implementing the quality system. The Quality Manual is electronically maintained on GTX's server as a read-only protected file. It is available to each employee for viewing. The Quality Manager is responsible for distribution and maintenance of controlled documents related to the quality system.

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5.5.c.1. Management Responsibilities:

Chief Engineer/CEO: Has overall authority on the laboratory and primary technical authority. The Chief Engineer/CEO provides oversight of technical activities and resources.

Chief Operating Officer (COO): The COO works with the Director of Testing Services to develop a business plan and set yearly financial goals for GTX. He/she is also responsible for making sure GTX has whatever resources are necessary to meet those goals.

Director of Testing Services: Is responsible for the day-to-day management of all GTX branches. This person is responsible for both business development, operational activities, and laboratory health and safety policy, within the testing division. This person is responsible for contract review. He/she will perform quality assurance checks on reports generated by GTX. The Director of Testing Services is responsible for ensuring compliance with ISO/IEC 17025, and maintaining client confidentiality. In addition, the Director of Testing Services is responsible for identifying, assessing, evaluating, and controlling risk and opportunities related to the GTX quality system, impartiality, and lab integrity.

Laboratory Manager: Responsible for scheduling, performing, and reporting for the laboratory and field. Responsible for health and safety in the laboratory and is the main client liaison during the testing portion of a project. This person is responsible for maintaining the testing equipment and laboratory facilities. The Laboratory Manager is responsible for ensuring compliance with ISO/IEC 17025, and maintaining client confidentiality. In addition, the Laboratory Manager is responsible for identifying, assessing, evaluating and controlling risk and opportunities related to the GTX quality system, impartiality and lab integrity.

Assistant Laboratory Manager: Responsible for assisting the Laboratory Manager accomplish his/her duties. The Assistant Laboratory Manager is responsible for ensuring compliance with ISO/IEC 17025, and maintaining client confidentiality. In addition, the Laboratory Manager is responsible for identifying, assessing, evaluating, and controlling risk and opportunities related to the GTX quality system, impartiality, and lab integrity.

Quality Manager: This person is independent of the laboratory operations and reports directly to the Director of Testing Services. He/she establishes, manages, and verifies an effective quality system. This person will perform quality assurance checks on reports generated by the laboratory. This person is authorized to implement changes to the quality system and is charged with the responsibility of reporting corrective actions to management. The Quality Manager is responsible for ensuring compliance with ISO/IEC 17025, and maintaining client confidentiality. In addition, the Quality Manager is responsible for identifying, assessing, evaluating, and controlling risk and opportunities related to the GTX quality system, impartiality, and lab integrity.

Laboratory Engineer: Responsible for assisting the Director of Testing Services, Laboratory Managers, the Quality Manager, and the Quality Control Technician to ensure the technical correctness of testing, review and improve lab procedures, assist with training, help with internal QC/QA programs and reporting. The Laboratory Engineer is responsible for ensuring compliance with ISO/IEC 17025, and maintaining client confidentiality. In addition, the Laboratory Engineer is responsible for identifying and reporting risk and opportunities related to the GTX quality system, impartiality and lab integrity.

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Quality Control Technician: Responsible for assisting the Quality Manager implement and maintain the quality system prescribed by this quality manual in the laboratory. The Quality Control Technician is responsible for ensuring compliance with ISO/IEC 17025, and maintaining client confidentiality. In addition, the Quality Control Technician is responsible for identifying, assessing, evaluating, and reporting risk and opportunities related to the GTX quality system, impartiality and lab integrity.

Laboratory and Field Technicians: Person(s) responsible for receiving samples, conditioning, preparing, and testing them. Technicians prepare test data and provide draft reports to management. The Technicians are responsible for following the GTX Quality Manual and laboratory procedures and maintaining client confidentiality. In addition, the Technicians are responsible for identifying and reporting risk and opportunities related to the GTX quality system, impartiality, and lab integrity.

Finance and Accounting Controller: This person is responsible for the invoicing, collections, payroll, other payables, and financial reporting of the company.

Administrative Staff: This group performs duties related to invoicing, payroll and collections. Basic administrative duties such as phone answering, shipping and filing are also carried out by this staff.

5.6 RESOURCES, DUTIES, AND THE QUALITY SYSTEM

The resources necessary to meet our quality commitments and satisfy our clients' needs are provided by the company. The quality system outlined in this Quality Manual is designed to meet requirements in ANSI/ISO/IEC 17025:2017. Employees of GTX are responsible for adhering to procedures outlined in the Quality Manual while satisfying our clients' needs.

GTX is committed to providing the necessary resources to achieve its stated goals. This includes:

- Trained personnel to manage and perform testing as outlined by appropriate standards
- Equipment with capabilities, accuracy and precision as prescribed by appropriate standards
- Computers necessary for data acquisition, analysis, report generation, accounting and marketing
- Facilities necessary to conduct testing in appropriate atmosphere

5.6.1 Implementation, Maintenance, and Improvement of the Management System

The quality system outlined in this Quality Manual is designed to meet the requirements outlined in ANSI/ISO/IEC 17025:2017.

The GTX Quality System is designed to allow for:

- Identification and management of risks and opportunities related to the GTX quality system
- The identification of deviations from the management system
- Identification of deviations from the procedures for performing laboratory activities
- The initiation of actions to prevent or minimize such deviations

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- Reporting to laboratory management on the performance of the management system and any need for improvement
- Ensuring the effectiveness of laboratory activities.

Improvements to procedures are coordinated with proper authority to assure uniform compliance. Changes are made to existing systems only after a review of the change and how it will affect the quality system. Employees of GTX have access to the appropriate documentation needed to implement the quality system.

Open lines of communication exist between all levels of GTX employees from technician level to Chief Engineer/CEO. Periodic meetings are held and documented to discuss the quality system, problems, corrective actions, and improvement.

5.7 MANAGEMENT RESPONSIBILITIES FOR THE QUALITY SYSTEM

Laboratory management shall ensure that communication takes place regarding the effectiveness of the management system and the importance of meeting customers' and other requirements. Laboratory management shall also ensure that the integrity of the management system is maintained when changes to the management system are planned and implemented.

6 RESOURCE REQUIREMENTS

6.1 GENERAL

The GeoTesting Express, Inc. Laboratory shall be operated so that it will have the personnel, facilities, equipment, systems, and support services necessary to manage and perform its laboratory activities.

6.2 PERSONNEL AND TRAINING

Personnel of GTX have met minimum requirements of education and technical knowledge. Each technician has demonstrated an ability and willingness to work in a laboratory or field environment. Each technician undergoes a training period during which constant supervision is maintained while teaching and performing a specific test method. After demonstrating that the technician is able to perform a test with an adequate level of competence, and after passing a written examination, that technician is considered proficient enough to perform the test without constant supervision.

The Laboratory Managers and Quality Control Technician are responsible for the training program and maintenance of training records. This includes evaluation of initial competency, initial proficiency, and bi-annual renewals in addition to making available and scheduling external training such as ACI, NICET, OSHA 40-hour safety, radiation safety, etc.

Technician proficiency records (initial proficiency and renewals) are maintained electronically for each technician. These records show tests for which a particular technician has been deemed proficient, date of proficiency, trainer, and date for renewal.

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A sample Technician Training Record chart is shown in Appendix D.

GTX also maintains a technician training matrix modeled on A2LA form F117 - "Technical Staff Matrix for Accreditation - ISO-IEC 17025." (See Appendix D.) This form lists all methods performed by the GTX lab and lists all technical personnel trained to perform each test method.

Specific test method training is to be provided by a qualified technician, Quality Control Technician, Laboratory Manager, or Assistant Laboratory Manager. A technician is qualified as a test method trainer when the following has occurred:

- Complete the training for that test him/herself
- Pass the written exam
- Show continued proficiency in performing the test
- Be approved by the Quality Control Technician or Laboratory Manager as a trainer

Renewals are performed once every two years. The Laboratory Manager, Quality Control Technician or Quality Manager conducts the renewals for all technicians. The renewal requires a technician to have reviewed the current ASTM procedure and current SOP as well as conduct a demonstration of the test in the presence of the Laboratory Manager, Quality Control Technician or Quality Manager.

Every four years, a technician shall also take and pass a written examination.

Descriptions of the levels of training required for becoming a Laboratory Technician Trainee, Laboratory Technician I, II, III, IV and V are presented in Section 5.5.b.

NICET, ACI and OSHA testing and evaluation in addition to proficiency achieved as documented in training records are used to determine when a higher Technician training level has been reached.

The technician, Laboratory Manager and Quality Control Technician are responsible for keeping training proficiencies up to date and current. The Laboratory Manager and Quality Control Technician are responsible for signing off (verifying) the training records of each technician. The Laboratory Manager and Quality Control Technician are also responsible for determining when a Technician has reached a higher Technician training level. A technician may perform production work only for those tests which he/she has been determined to be proficient.

Below is an outline of the phases of indoctrination, evaluation, and training:

- Indoctrination
  - Technician meets with human resource manager to:
    - Complete necessary paperwork
    - Be given copy of company employee handbook
    - Be given copy of company health and safety plan
    - Be given a tour of the facility (includes location of exits, safety equipment, etc.)
    - Meet key personnel in the company
    - Be given any necessary items (building key, phone, computer, etc.)

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- Initial competency evaluation
  - Technician meets with member of laboratory management team to:
    - Discuss prior experience and review documentation to determine level of competency from previous employment and develop plan for training.
- General laboratory training
  - Technician reads current revision of Quality Manual
- Specific test training
  - Technician to read latest revision of the published (ASTM, AASHTO, other) test standard
  - Technician to read GTX's standard operating procedure, including job hazard analysis (JHA) specific to the test
  - Trainer to verbally explain the test
  - Technician to watch the test being performed and is encouraged to take notes and ask questions
  - Technician to perform the test in the presence of a trainer on a side-by-side sample of material
  - Technician can begin testing on client samples under supervision of trainer
  - Technician takes the written exam and must receive a grade of at least 80%; written exam is administered independently in an open-book fashion and graded by the Laboratory Manager or Quality Control Technician
- Two year renewals
  - Verify technician has read the latest revision of the published test standard
  - Verify technician has read the latest revision of GTX's standard operating procedure
  - Technician to perform the test in the presence of a trainer to verify proficiency
- Four year renewals
  - In addition to the two-year renewal requirements, technician to take written exam and receive a grade of at least 80%

6.2.1

All GTX personnel shall act impartially and work in accordance with the GTX management system as outlined in Section 4.1 and 5.2 above.

6.2.2

The GTX lab documents the competence requirements of each lab position as outlined in Sections 4.1, 5.5a and 6.2.

6.2.3

GTX ensures the competence of its staff and trains them to be able to evaluate the significance of

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deviations in tests they are trained to perform as outlined in Sections 4.1 and 5.1.

6.2.4

GTX management shall communicate to the staff their duties, responsibilities and authorities as outlined in Section 5.5.

6.2.5 Procedures and Records

Records of current procedures are maintained electronically. Obsolete versions of the procedures are also retained. These procedures include:

- Determining the competence requirements as outlined in Section 5.2.
- Selection of personnel as outlined in Section 5.2.
- Training of personnel as outlined in Section 5.2 and 6.2.
- Supervision of personnel as outlined in Sections 4.1, 5.2 and 6.2.
- Authorization of personnel as outlined in Sections 5.2 and 6.2.3.
- Monitoring of competence of personnel as outlined in Section 5.2.

6.2.6

GTX management authorizes personnel to perform specific laboratory activities, including but not limited to, the following:

- Develop, modify, verify and validate methods and any deviations (See Section 7.2.).
- Analyze results, including statements of conformity or opinions and interpretations
- Review, authorize and report results.

6.3 FACILITIES AND ENVIRONMENTAL CONDITIONS

GTX is a permanent laboratory with two locations. The MA laboratory is located at 125 Nagog Park in Acton, MA 01720 and occupies an approximately 12,000 square feet section of the building. Approximately 600 square feet is allocated to office space and 11,400 square feet allocated to the testing laboratory. The GA laboratory is located at 2358 Perimeter Park Drive in Atlanta, GA 30341 and occupies approximately 3,000 square feet, of which 500 is allocated to office space. Laboratory testing equipment is housed and used in the laboratory. Field testing equipment is housed in the laboratory and transported to the field for use. A floor plan of the laboratory showing the location of testing activities is maintained by the Laboratory Manager. The floor plan is included in Appendix E.

In most cases, samples obtained from various clients are not under the control of GTX laboratory personnel, therefore responsibility for proper shipment is the client's. Once arriving at GTX, samples are visually inspected, and the condition of samples is recorded. A determination is made regarding proper storage techniques for the samples and appropriate actions are taken.

After samples are logged in, samples are stored in the current work storage area. Samples are typically stored in the container they arrived to the laboratory in. Only those technicians who are assigned to

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perform work on a sample will take the sample from the current work storage area. Until all work for the sample is performed, the sample is stored in this area.

The geosynthetics laboratory atmosphere is controlled for temperature and somewhat controlled for relative humidity. Sample preparation, conditioning and testing is performed in this controlled area. The temperature and relative humidity are monitored and logged each day that geosynthetics testing is performed in the Geosynthetics Laboratory Atmosphere Log Book.

An environmental condition criteria table is posted in the geosynthetics laboratory which states the upper and lower limits for each geosynthetics test as per the applicable standard. It also warns that testing should be halted if the temperature in the laboratory becomes out of control. Testing can resume only when temperature is back in control and after receiving approval from a laboratory manager or director. If the relative humidity becomes out of control, it should be noted in the comment section of the laboratory test sheet, however testing may continue.

Access to and use of areas affecting the quality of testing activities is controlled. Visitors entering the laboratory meet with a supervisor and, if other than a sample drop-off is being conducted, shall sign in at the front desk with the receptionist.

6.4 EQUIPMENT

6.4.1

GTX owns all the equipment required for the correct performance of the laboratory activities it performs. These include all measuring instruments, software, measurement standards, reference materials, reference data, reagents, consumables, and auxiliary apparatus which can influence test results.

6.4.1.1

The only equipment not owned by GTX are items used for calibration and verification that are performed by others for GTX.

6.4.2

GTX does not rent or borrow equipment owned by others. If a case came up where GTX had to use equipment outside its permanent control, GTX shall ensure that equipment requirements of its quality system are met.

6.4.3

Policies and procedures are outlined in this section for proper handling, transport, storage, use and planned maintenance of equipment. These procedures ensure that equipment is functioning properly and not allowed to deteriorate or become contaminated in some way. Detailed equipment handling procedures are presented in the GTX Equipment SOP, GTX-QD014.

6.4.4

GTX verifies that all equipment conforms to test and measurement requirements before being placed into or returned to service.

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6.4.5

The equipment used by GTX for measurement shall be capable of achieving the measurement accuracy and/or measurement uncertainty required by ASTM or other test standard agency to provide a valid result.

6.4.6

GTX requires that measuring equipment shall be calibrated when:

6.4.6.1 The measurement accuracy or measurement uncertainty affects the validity of the reported results.

6.4.6.2 The calibration of the equipment is required to establish the metrological traceability of the reported result.

6.4.6.3 The calibration interval established by a test standard (i.e. ASTM, AASHTO, etc.) has been reached.

6.4.6.4 The calibration interval established by GTX has been reached.

6.4.6.5 When equipment calibration has been requested by a client before and/or after project testing, as part of the client's quality control program.

6.4.6.6 When new or repaired equipment is put into service.

6.4.6.7 Calibration or verification is not required for equipment that is out of service and has gone beyond its calibration/verification interval date. Calibration/ verification of out of service equipment is required immediately before the equipment is put back in service.

6.4.7

GTX has an established calibration program. The policies and procedures of the program are outlined below and in Section 6.4.14. The calibration program is reviewed and adjusted as necessary in order to maintain confidence in the status of calibration and test results.

6.4.8

All calibrated equipment or equipment which has a defined period of validity shall be labelled, coded, or otherwise identified to allow the user of the equipment to readily identify the status of calibration or period of validity.

6.4.9

GTX equipment shall be taken out of service if it has been subjected to overloading or mishandling, gives questionable results, has been shown to be defective or outside specified requirements' or requires calibration or verification. Equipment taken out of service is to be clearly labelled or marked, and when practicable, shall be isolated to prevent its use. Equipment labeled or marked out of service shall remain out of service until it has been verified to perform correctly. With the exception of items removed from service by the lab management because they are not currently in calibration, GTX shall initiate a corrective action to examine the effect of the defect or deviation from specified requirements and shall initiate the management of nonconforming work procedure (see 7.10).

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6.4.10

GTX shall have a procedure for all intermediate checks used to maintain confidence in the performance of the equipment.

6.4.11

GTX shall update and implement, as appropriate, all reference values and correction factors as they are generated to meet specified requirements of equipment or test methods.

6.4.12

GTX shall take practicable measures to prevent unintended adjustments of equipment from invalidating results.

6.4.13

Records shall be retained for equipment which can influence laboratory activities. The records shall include the following, where applicable:

- The identity of equipment, including software and firmware version.
- The manufacturer's name, type identification, and serial number or other unique identification.
- Evidence of verification that equipment conforms with specified requirements.
- The current location.
- Calibration dates, results of all calibrations, adjustments, acceptance criteria, and the due date of the next calibration or the calibration interval.
- Documentation of reference materials, results, acceptance criteria, relevant dates, and the period of validity
- The maintenance plan and maintenance carried out to date, where relevant to the performance of the equipment.
- Details of any damage, malfunction, modification to or repair to of the equipment.

6.4.14 Specific GTX Equipment and Equipment Verification and Calibration Policies and Procedures

An inventory list of sampling, testing, and calibration and verification equipment is maintained in the Quality folder on the GTX server. This inventory list contains the following information:

- Description
- Associated System
- Manufacturer
- Quantity
- Model #
- Serial #

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- Location
- Date Received
- Date in Service
- Date Out of Service
- Condition When Received

Laboratory personnel are responsible for verifying the following prior to performing tests:

- Equipment is in working order
- Equipment is calibrated per requirements
- The calibrated equipment to be used is accurate for the range needed for the test
- Calibration records are current

Calibration of laboratory equipment is performed internally by the use of basic calibration instruments which are traceable to NIST standards. Some pieces of equipment are sent out for external calibrations. Certificates of traceability for these instruments are located in the Laboratory Equipment Calibration Log kept in the Laboratory Manager's office. External agencies provide GTX with information regarding any out of ordinary condition of equipment including if the equipment is out of tolerance. This approach permits internal calibration and periodic checks by trained personnel. Equipment is marked with calibration labels with at least the following information: calibration agency, calibration date, and due date for next calibration. Calibration certificates and records are kept in the Laboratory Equipment Calibration Log. Calibration and/or verification procedures are located in the Laboratory Manager's office. Only personnel trained to conduct calibrations are allowed to perform the procedures. (See Section 8.)

The Table in Appendix F summarizes the frequency and requirement for test equipment calibration and verification.

With the use of GTX's LIMS, calibration/verification due dates are tracked and the specific tasks (calibrations or verifications) for equipment are assigned to technicians. In this way, the calibrations and verifications are completed by the required dates. The person responsible for making sure calibrations and verifications are performed is the Quality Control Technician. Both the MA and GA laboratory Quality Control Technician is Nancy Hubbard.

Documentation is kept on each piece of testing equipment. A "Maintenance and Calibration Record" log is maintained by the Laboratory Manager and contains the following information:

- Name of test equipment
- Manufacturer's name
- Type of equipment
- Identification (model number and serial number)
- Dates of calibration and results
- Damage, malfunctions or maintenance performed on equipment

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- As-found and as-left condition if out of ordinary

Either Internal reference materials (IRMs) or verification procedures exist for each piece of testing equipment. IRM and verification records are maintained for each piece of testing equipment.

A copy of the manufacturer's instruction manual is kept for each piece of equipment. These are located in a marked file in the testing laboratory.

The following are general control procedures for maintaining properly calibrated testing equipment:

- Newly acquired equipment without manufacturer's certification is calibrated before being placed into service.
- Prior to testing, the measurements to be made are determined and, depending upon the range, accuracy and precision required, the proper testing equipment is utilized to perform the test.
- Testing equipment and measuring devices are calibrated at regular intervals and checked at various times prior to testing. Calibrations are logged in the Laboratory Equipment Calibration Log. These calibrations are made against internationally known standards when possible. Outside agencies are contracted to perform periodic calibrations.
- Calibrations of equipment and measuring devices are performed to optimize the accuracy of the range in which testing is required. Details of calibration results are located in the Laboratory Equipment Log. These include: equipment name, type and unique identification, locations, frequency of calibration checks, the method of calibration and acceptance criteria. When a calibration or check finds a piece of equipment to be out of control, it is brought to the attention of the Laboratory Manager and a course of action is determined.
- Test equipment is marked with a label depicting identification number and calibration date.
- Records of calibration are kept in the Laboratory Equipment Calibration Log. This log is stored in the Laboratory Manager's office.
- If a piece of equipment is out of control, part of the actions taken include evaluating previous inspections to determine when the equipment became out of control and what effect it has had on previous test results. Clients are notified if previous test results were determined to be adversely affected. The piece of equipment is taken out of service and clearly labeled until repaired or corrected.
- Equipment that has been removed from service shall be verified or calibrated prior to being placed into service again.
- The laboratory atmosphere is kept at the accepted temperature and humidity for testing and is maintained at those conditions during calibrations and checking.
- Test equipment is handled and stored with care, minimizing any activity which may cause calibrations to become invalid. When not in use, equipment is stored in a clean area and typically covered or placed in a closed container.
- Calibrations are saved and stored in computer files which are uniquely named. They are also stored in the Laboratory Equipment Calibration Log.

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6.5 METROLOGICAL TRACEABILITY

6.5.1

GTX has established and maintains metrological traceability of its measurement results by means of a documented unbroken chain of calibrations, each contributing to the measurement uncertainty, linking them to an appropriate reference.

6.5.2

GTX measurement results are traceable to the International System of Units (SI) through:

- Calibration provided by a competent laboratory accredited by A2LA.
- Certified values of certified reference materials provided by a competent producer with stated metrological traceability to the SI.
- Direct realization of the SI units ensured by comparison, directly or indirectly, with national or international standards.

6.5.2.1

The standards used by GTX are primarily from ASTM International. The standards specify both units and calibration/verification methods. ASTM recognizes the use of both metric (SI) units and Imperial (IP) units. For the most part GTX uses standards from ASTM Committee D18 on Soil and Rock. Individual ASTM D18 standards specify measurement units. These units may be in SI, IP or both.

Committee D18's policies for the use of units can be found in Section 8.4 of the ASTM International D18 Standards Preparation Manual Revision 3 (2018.) Committee D18 has put a considerable effort into developing a consistent approach to units in its standards along with meeting a key objective to the users of those standards. That is, the approach is understandable, represents the users (international and domestic), and is scientifically correct.

The following is a summary of ASTM D18's guidance on the usage of units.

- For standards that have units contained in them, the unit section in the Scope must define in detail how units are being used.
- If force is mentioned in the standard and inch-pound (IP) units are included, the gravitational IP system of units shall be used. Force is represented by lbf (IP) and N (SI).
- If force and mass/density are included in the standard, only SI units are used for mass and density. The preference for the units statement is the combined (dual) units statement, either SI with IP in brackets or IP with SI in brackets (SI [IP] or IP [SI]).
- The Civil Engineering profession has used some units not covered or frowned upon by the SI standard for over a century. It is D18's policy to continue to represent this profession by using such units as unit weight (lbf/ft3) and density in g/cm^3 or Mg/m^3.
- For standards where the apparatus was developed using the IP system of units, this document has specialized guidance for the presentation of IP and SI units.

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- If the standard's Calculation section is complicated or extensive, only one set of units should be used; typically, SI if density is involved.

6.5.3

When metrological traceability to the SI units is not technically possible, the laboratory shall demonstrate metrological traceability to an appropriate reference such as:

- Certified values of certified reference materials provided by a competent producer.
- Results of reference measurement procedures, specified methods or consensus standards that are clearly described and accepted as providing measurement results fit for their intended use and ensured by suitable comparison.

6.6 EXTERNALLY PROVIDED PRODUCTS AND SERVICES

GTX shall ensure that any products or services purchased do not negatively affect lab activities and only suitable vendors and products are used. Purchasing policies are outlined below and are specified in the GTX Purchasing SOP, QD16.

6.6.1 Supplies and Services

Purchases of supplies and services that affect the quality of tests are made by using the most qualified vendor at the most reasonable price. GTX maintains a vendor list which is described below. Purchase orders with unique numbers shall be issued for each purchase. A description of the service or supply ordered shall be on the purchase order. Purchase orders can be obtained from the Laboratory Manager(s), Director of Testing Services or the GTX Administrative Assistant. Once generated, a physical copy of the purchase order is produced, and it is reviewed then signed by the person requesting the purchase. It is then maintained in a file by the Administrative Assistant.

GTX shall communicate its requirements to external providers for the products and services to be provided including:

- GTX acceptance criteria
- Evidence of subcontractor competence which can include qualifications of subcontractor's personal
- Any activities that GTX or its clients intend to perform at the subcontractor's facility.

When supplies or equipment are received in the laboratory, a check is performed to ensure it meets the end user's requirements and the specifications given on the purchase order. If acceptable, the purchase order is signed and dated as acceptable by the person who requested the purchase. For instance, when a piece of testing equipment is purchased, that piece of equipment is subject to a calibration/verification check prior to being put into use. If the piece of equipment fails the calibration/verification check, it is sent back to the manufacturer for a replacement. If it is acceptable, the person who ordered the equipment signs and dates the purchase order as acceptable.

GTX maintains a vendor list which lists the vendor, phone number, description of item(s) purchased and overall rating. The overall rating is based on prior history, pricing, availability, service, quality and ease of use. Vendors who repeatedly fail to perform up to standards are no longer used. Heavy

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consideration is given to those vendors whom GTX has fostered a long-term relationship with. When requested, a client's representative is afforded the right to verify the vendor's premises.

When purchasing software or equipment that includes software, an attachment is included which requires the supplier of the software to provide notification of any software error that is found in the future that could affect test results. Commercial software may be considered to be sufficiently validated and software suppliers are not required to provide quality assurance documentation. However, GTX shall request QA documentation at the time software is ordered.

If GTX makes any changes to the configuration or modifies commercial software, then the software shall be verified to confirm that the software is still performing correctly.

Testing for nuclear projects under NQA-1 quality systems can require verification of commercial software. This process will be handled on a case by case basis.

6.6.2 Calibration and Verification Services

When purchasing calibration services, an attachment with specific terms and conditions is included with the purchase order which allows GTX to audit the calibrating agency's facility and which requires the calibration agency to perform its activities in accordance with GTX's quality plan.

6.6.3 Subcontracted Testing Services

In the event that GTX cannot perform a portion of the work for which it is contracted, a subcontractor is used. GTX evaluates and selects subcontractors on the basis of their ability to meet requirements and the nature of their quality system. Subcontractors shall have a quality system equal to or more in-depth than that of GTX. Accreditation to ISO/IEC 17025 will be accepted as evidence of vendor qualification. Qualified subcontractors are listed on an approved vendor list which GTX maintains and makes available to the appropriate personnel.

GTX informs its clients of the subcontracted work and gains approval from the client for undertaking such activity. Reports sent to the client which have test data supplied by a subcontractor are clearly marked as such. Subcontractor results, in part or in full, are provided directly to the client as received from the subcontracted laboratory.

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7 PROCESS REQUIREMENTS

Process requirements are summarized in Figure 1 below from Annex B of ISO/IEC 17025:2017

ISO/IEC 17025:2017(E)

B.5 Figure B.1 illustrates an example of a possible schematic representation of the operational processes of a laboratory, as described in Clause 7.

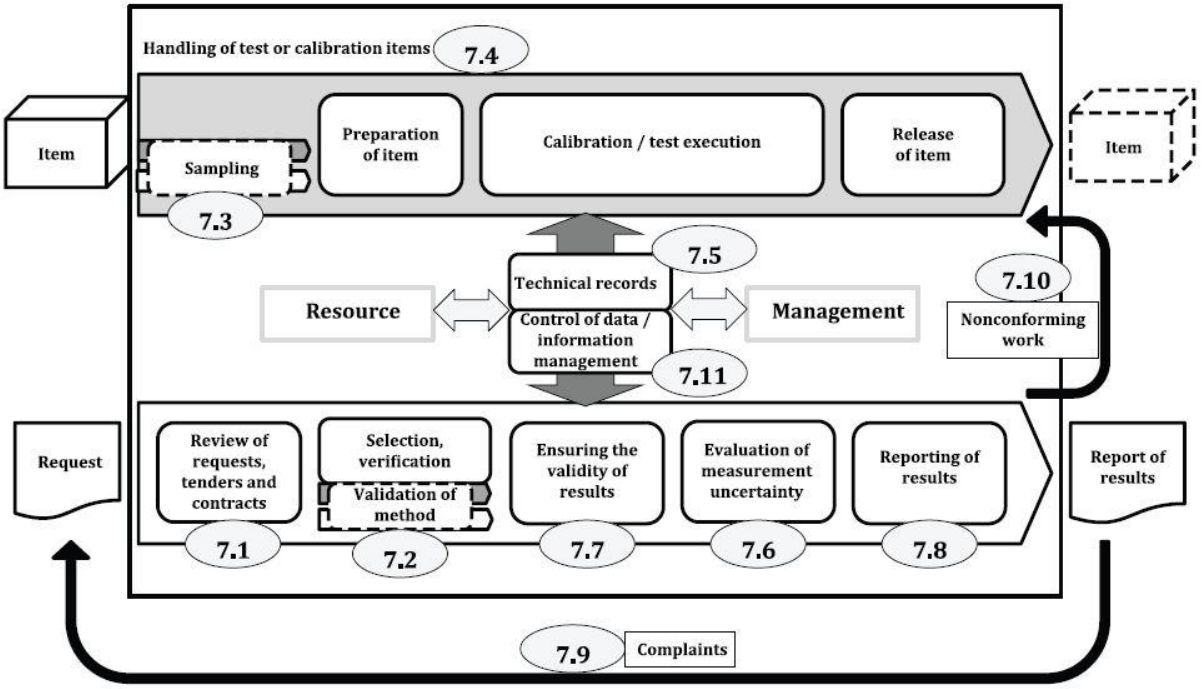

Figure B.1 — Possible schematic representation of the operational processes of a laboratory

Figure 1. ISO/IEC 17025:2017 Operational Processes of a Laboratory

7.1 REVIEW OF REQUESTS, TENDERS AND CONTRACTS

7.1.1

The GTX procedures for the review of client's requests, tenders and contracts are listed below. The procedures are designed to ensure the following:

- The requirements for reviewing a client request or contract are adequately defined, documented and understood.

- Determine if GTX has the capability and resources to meet the client's project needs.
- If a project requires subcontracted testing, GTX will ensure that the requirements of Section 6.6 are followed and GTX informs the client of the specific laboratory activities to be performed by a subcontracted lab and gains the clients approval.
- The appropriate methods or procedures are assigned for each project. GTX must also confirm that the lab is capable of meeting the client's project specific requirements.

7.1.2

GTX shall inform clients when the test method requested is considered to be inappropriate or out of date.

7.1.3

ISO/IEC 17025:2017 requires the following: "When the customer requests a statement of conformity to a specification or standard for the test or calibration (e.g. pass/fail, in-tolerance/out-of-tolerance) the specification or standard, and the decision rule shall be clearly defined. Unless inherent in the requested specification or standard, the decision rule selected shall be communicated to, and agreed with, the customer." However, GTX does not make engineering judgements for its clients. The client is responsible for making pass/fail or in-tolerance/out-of-tolerance decisions based on the client's project specifications and the test data provided by GTX.

7.1.4

Any differences between the test assignment, request or tender and the contract shall be resolved before laboratory activities commence. Each contract shall be acceptable both to the laboratory and the client. Deviations requested by the client shall not impact the integrity of GTX or the validity of the results.

7.1.5

The client shall be informed of any deviation from the contract.

7.1.6

If a contract is amended after work has commenced, the contract review shall be repeated and any amendments shall be communicated to all affected personnel.

7.1.7

The laboratory shall cooperate with clients or their representatives in clarifying the client's request and in monitoring GTX's performance in relation to the work performed.

7.1.8

GTX saves all contract, agreements and assignment reviews in the project files (either electronically or hardcopy), in the GTX database or the Manager's lab notebooks. Records of significant changes are also retained. Emails and written records of pertinent discussions with the client about project assignments, test requirements and test results are retained for a minimum period of seven years.

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7.1.9 GTX Specific Policies on Review of Client Contracts, Agreements and Test Assignments

7.1.9.1 GTX Quotes and Terms and Conditions

Much of GTX's lab work is performed under a GTX Price Quote for Testing Services and the GeoTesting Express, Inc. Standard Terms and Conditions for Engineering, Testing and Consulting Services. In addition, GTX may also perform work under a client's contract or purchase order. (See below)

7.1.9.2 Contracts

A contractual agreement should exist between GTX and the client for laboratory and/or field services supplied by GTX before GTX performs any of these services. This contractual agreement can be in the form of a Purchase Order (PO), contract, Work Order, Task Order, Chain of Custody, or verbal okay. GTX has a procedure in place for reviewing contracts which adhere to the following guidelines:

- Requested services are defined
- Verbal orders are followed with documented requests
- GTX can perform requested services
- GTX can perform the requested services within schedule limits
- GTX can perform the requested services under proposed pricing structure

Review of these contractual agreements is the responsibility of the Chief Engineer, Director of Testing Services and/or Laboratory Manager. The Director of Testing Services coordinates this activity.

Changes which may occur in the scope of work and/or time schedules due to the client's needs are reviewed in the same way as initial contracts. The same key elements are reviewed with respect to the proposed changes. Any condition encountered during execution of the requirements of the contract which changes the scope of work established by the original contract is documented. A written amendment to the contract is agreed upon by both GTX and the client with price and schedule adjustments.

Conditions which change the scope of work encountered during execution are brought to the attention of the Laboratory Manager who in turn initiates a discussion with the client. After the client and GTX mutually agree upon change orders, these changes in scope of work are reviewed with the staff performing this work.

Contracts, amendments and change orders are recorded and signed by responsible representatives of both GTX and the client. These records are filed in the yellow section of a specific project file.

7.1.9.3 Client Service

GTX's level of service can be measured by the results of its client satisfaction survey. The survey is sent to each client upon completion of a project. The survey asks the client to rate GTX on a number of different items on an escalating scale (1-5). GTX's goal is to receive a grade of 4 or higher (1 = failed to meet expectations and 5 = exceeded expectations). GTX aims to continuously improve its

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quality system in order to better serve our clients. A return rate of 5% is required to validate the survey. It is our policy to respond to all complaints received in the customer survey. Data obtained in the customer survey is discussed in quarterly management review meetings.

7.2 SELECTION, VERIFICATION AND VALIDATION OF METHODS

7.2.1 Selection and verification of methods

The GTX procedures for the selection, verification and validation of methods are listed below. The procedures are designed to ensure the following:

7.2.1.1

GTX shall use appropriate methods and procedures for all laboratory activities and, where appropriate, for evaluation of the measurement uncertainty as well as statistical techniques for analysis of data.

7.2.1.2

All methods, procedures and supporting documentation used by GTX, such as GTX Standard Operating Procedures, test standards, manuals and reference data relevant to GTX activities, are kept up to date and are readily available to GTX personnel (see 8.3).

7.2.1.3

GTX uses the latest valid version of a method unless it is not appropriate or possible to do so. This is done primarily through the use of the ASTM International Standards Tracker Alert system. Tracker Alert sends an email notification about newly published or revised ASTM standards. When necessary, the application of the method shall be supplemented with additional details to ensure consistent application. Supplemental details are generally included in the GTX Standard Operating Procedure (SOP.)

7.2.1.4

When the client does not specify the method to be used, GTX shall select an appropriate method and inform the client of the method chosen. Methods published either in international, regional or national standards, or by reputable technical organizations, or in relevant scientific texts or journals, or as specified by the manufacturer of the equipment are recommended. Laboratory-developed or modified methods can also be used.

7.2.1.5

GTX shall verify that it can properly perform methods before introducing them by ensuring that it can achieve the required performance. Records of the verification shall be retained. If the method is revised by the issuing body, verification shall be repeated to the extent necessary.

7.2.1.6

When method development is required, this shall be a planned activity and shall be assigned to competent personnel equipped with adequate resources. As method development proceeds,

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periodic review shall be carried out to confirm that the needs of the client are still being fulfilled. Any modifications to the development plan shall be approved and authorized.

GTX will occasionally develop in-house procedures for testing and calibration. Development of these procedures is a planned activity and is only conducted by qualified personnel with appropriate equipment. Laboratory management is involved in the process during development of procedures and maintains close contact with personnel conducting the development.

7.2.1.7

Planned deviations from methods for all laboratory activities shall occur only if the deviation has been documented, technically justified, authorized, and accepted by the client.

7.2.2 Validation of methods

7.2.2.1

GTX shall validate non-standard methods, laboratory-developed methods and standard methods used outside their intended scope or otherwise modified. The validation shall be as extensive as is necessary to meet the needs of the given application or field of application.

7.2.2.2

When changes are made to a validated method, the influence of such changes shall be determined and where they are found to affect the original validation, a new method validation shall be performed.

7.2.2.3

The performance characteristics of validated methods as assessed for the intended use, shall be relevant to the clients' needs and consistent with specified requirements.

7.2.2.4

The laboratory shall retain the following records of validation:

- The validation procedure used.
- Specification of the requirements.
- Determination of the performance characteristics of the methods.
- Results obtained.
- A statement on the validity of the method, detailing its fitness for the intended use.

7.2.3 GTX Test and Calibration Methods and Method Validation Procedures

7.2.3.1 General

GTX uses appropriate methods and procedures for all testing and calibrations performed. Sampling, handling, transporting, storing and preparing test samples are conducted in accordance with established procedures.

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If a condition exists which will hinder GTX from performing tests in accordance to published standards, the client is notified. If the condition of the samples is such that damage has occurred during shipment the client is notified. In addition, the client is notified if a discrepancy in sample identification exists. Unsuitable conditions or discrepancies in customer supplied samples are noted and recorded in the project file. Any discrepancies will be resolved to the satisfaction of the client with a mutually acceptable course of action. Quality control of test procedures is continuously monitored by technicians performing tests as well as supervisor(s) and the Quality Control Technician. These inspections occur before, during and after testing. Each technician documents procedural deviations in the comment section of the laboratory work sheet or appropriate LIMS entry field.

GTX performs tests according to test standards found in ASTM, AASHTO, EPA, the USA COE and other published test standards. Deviations from stated test methods are noted on the test report. The test standards are located on the GTX server. Technicians have access to these standards. To assist technicians, GTX Standard Operating Procedures are also accessible.

Samples are conditioned according to the appropriate test standard. These conditions include environmental considerations such as temperature and humidity. Client's requests supersede this requirement, and shorter conditioning times may be employed.

GTX maintains a file containing equipment instructions which is available to technicians as appropriate.

7.2.3.2 Selection of Methods

GTX utilizes test methods which meet the client's request and are appropriate for the test. Except where otherwise requested by the client, GTX uses the latest revision of a test standard. If the client does not specify a method, GTX will suggest to the client an appropriate published method or, as a last alternative, a laboratory-developed method. In either case, the client is notified and agrees to the suggested method prior to commencing testing. In addition, if the client specifies a test that is out of date or otherwise inappropriate, GTX will notify that client and provide viable alternatives.

7.2.3.3 Laboratory-developed Methods

GTX will occasionally develop in-house procedures for testing and calibration. Development of these procedures is a planned activity and is only conducted by qualified personnel with appropriate equipment. Laboratory management is involved in the process during development of procedures and maintains close contact with personnel conducting the development.

7.2.3.4 Non-standard Methods

When conducting tests not covered by standard methods, the client must agree on the standard.

7.2.3.5 Validation of Methods

When conducting tests not covered by standard methods, GTX will validate the methods to confirm that they are fit for the intended use. The results, procedure used and a statement as to the soundness of the test for the intended use are obtained are recorded.

GTX conducts its own verification and in some cases, the calibration, of a number of pieces of test equipment. In addition, it contracts an outside agency to conduct verification/calibration on pieces

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of equipment. Several pieces of reference equipment (weights, gauge blocks, pressure gauge, etc.) are sent out to external NIST traceable, A2LA accredited labs for calibration. This calibrated equipment is then used to internally verify equipment such as dies, scales and gauges according to GTX's calibration schedule.

7.2.3.6 Estimation of Uncertainty of Measurement

GTX has adopted A2LA's "G103 – A2LA Guide for Estimation of Uncertainty of Dimensional Calibration and Testing Results" as its own in relation to estimating uncertainty in measurements made on different types of equipment. This procedure can be found on the GTX server.

Uncertainty budgets shall be developed for any type of measuring device calibrated by GTX for in-house use. Established uncertainty budgets shall be updated when there are any changes in the factors effecting the estimated uncertainty, such as recalibrated reference measuring devices. Uncertainty budgets for new models of measuring devices shall be determined before the device is put into service.

7.2.3.7 Control of Data

Computers used to collect data for testing are checked prior to being placed into service for testing. This is performed by the Laboratory Manager, Quality Manager, Quality Control Technician or the IT Manager. Access to computers used for data acquisition is limited (by password protection) to those authorized to do so. Software used to collect data is documented. Software used for testing is checked for accuracy prior to being used in the laboratory and then periodically thereafter. Records of computer and software checks are maintained by the Laboratory Manager. Suppliers of software to GTX are required to provide notification of software errors that could affect test results.

If software that has been utilized for testing is determined to have an error that could affect test results, part of the actions taken include evaluating previous tests performed to determine the extent to which test results have been affected. Clients are notified if previous test results were determined to be adversely affected. The software is taken out of service until an appropriate solution is determined. These actions are documented in a corrective action report.

When data is collected, it is saved to a file on the computer's hard drive with a unique name. At the completion of testing, this file is copied from the hard drive to the project's directory on the GTX server. The GTX server is backed up on a weekly basis.

7.3 SAMPLING

In general, GTX does not sample materials provided by a client for testing. Samples are typically brought to the laboratory for testing by our clients (either hand-delivered or shipped via courier). Samples are conditioned as per the test standard or at the client's request. Test specimens are taken from samples per the test standard requirements or the client's project sampling instructions. See the GTX Geosynthetic Test Standard Operating Procedures book for instructions on sampling geosynthetics.

If a sampling plan is required for a project, then GTX shall follow the ISO/IEC 17025:2017 requirements for sampling as outlined below in 7.3.1 – 7.3.3

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7.3.1

The laboratory shall have a sampling plan and method when it carries out sampling of substances, materials or products for subsequent testing or calibration. The sampling method shall address the factors to be controlled to ensure the validity of subsequent testing or calibration results. The sampling plan and method shall be available at the site where sampling is undertaken. Sampling plans shall, whenever reasonable, be based on appropriate statistical methods

7.3.2

If required, the GTX sampling method shall describe:

- The selection of samples or sites
- The sampling plan
- Preparation and treatment of sample(s) to produce the number of subsamples for subsequent testing.

NOTE: When received into the laboratory, further handling can be required as specified in 7.4

7.3.3

GTX shall retain records of sampling data. These records shall include, where relevant:

- The reference to the sampling method used.
- Date and time of sampling.
- Data to identify and describe the sample (e.g. number, amount, name).
- Identification of the personnel performing sampling.
- Identification of the equipment used.
- Environmental or transport conditions.
- Diagrams or other equivalent means to identify the sampling location when appropriate.
- Deviations, additions to or exclusions from the sampling method and sampling plan.

7.4 HANDLING OF TEST OR CALIBRATION ITEMS

7.4.1 The GTX procedures for the transportation, receipt, handling, protection, storage, retention, and disposal or return of test items are outlined throughout Section 7.4. Details are presented in the GTX Sample Handling SOP, QD019. This includes provisions necessary to protect the integrity of the test item, and to protect the interests of GTX and the client. Precautions are taken to avoid deterioration, contamination, loss or damage to the item during handling, transporting, storing/waiting, and preparation for, testing. If client handling instructions are provided with the item, the instructions shall be followed as long as they do not conflict with standards that define proper handling of samples. All client special handling instructions will be discussed with the client as needed.

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7.4.2

GTX has a system for the unambiguous identification of test samples. The sample identification is retained while the sample is under the responsibility of GTX. The GTX system ensures that samples will not be confused physically or when referred to in records or other documents. Where appropriate, the GTX system can allow for the sub-division of a sample or groups of samples and the transfer of samples between GTX labs as well as subcontracted labs.

7.4.3

Upon receipt of the test or calibration item, deviations from specified conditions shall be recorded. When there is doubt about the suitability of an item for test or calibration, or when an item does not conform to the description provided, GTX shall consult with the client for further instructions before proceeding and shall record the results of this consultation. When the client requires the item to be tested and acknowledges that it has requested a deviation from specified conditions, GTX shall include a disclaimer in the report indicating which results may be affected by the deviation.

7.4.4

When items have to be stored or conditioned under specified environmental conditions, these conditions shall be maintained, monitored and recorded.

7.4.5 GTX Procedures for Sample Receiving

7.4.5.1 Incoming Sample Inspection

After entering the laboratory and before testing, samples shipped (commercial courier, delivered by client, or picked up by a GTX employee) to GTX are visually inspected. This inspection consists of the following:

- Physical condition of the samples
  - Scratches, creases, holes or any other defects for geosynthetics
  - Broken sample containers (such as glass, etc.), condition of undisturbed materials
- Identification numbers are checked for accuracy
- The shipment is checked for completeness (all samples are included)
- Identification of client name, project name, etc. and other pertinent information unique to a project

Deficiencies or problems found during the inspection are documented in GTX's LIMS in the "Comments" field under the specific sample. The client is notified if any deficiencies or problems are found during this inspection.

7.4.5.2 Project Identification

When samples enter the laboratory, a determination is made by the person accepting them: the samples are either part of an existing project or they are part of a new project. If the samples are part of a new project, the Sample Receiving Technician or Administrative Assistant proceeds to create a project folder and assigns a GTX Project Number to that project. The Project Number is

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simply a number incremented by 1 for each new project. This is performed utilizing GTX's Laboratory Information Management System (LIMS). Information such as client name, address, phone and fax numbers, contact, project name, and project location are logged in with the project. A white project information sticker is produced and placed on the cover of the project folder for quick reference.

Located on the project folder is a checklist with the following information:

- Contract, PO, Quote, COC/Test Assignment or Order Confirmation
- Lab Data Sheets (if necessary)
- Archive E-mail Files
- Archive Electronic Files
- Scan Final Report
- Add to Satisfaction Survey List
- Sample Disposition

This checklist is a guide that assists in performing each task as well as a guide to confirm that the project file is complete and samples have been handled appropriately.

Physical project folders are color coded to indicate which type of project it is and consequently, how to handle the samples for that project.

Figure 2. Sample/Folder Color Key

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7.4.5.3 Sample Log-In

After receiving a new project folder or determining that samples received are part of an existing project, the samples are logged in. In the LIMS, the function "Samples" enables logging-in and test assignment. The following is a step-by-step log in procedure:

1) Conduct a search for the GTX Project Number the samples correspond to
2) In the "sample #" field, enter the sample number or numbers if more than one (only one sample number is allowed per line.) Also enter Boring and Depth if either of those are provided. Enter the date received and date results are due in the appropriate fields
3) With a mouse, double click the sample #
4) This new field allows entering each test required for this specific sample, assigning which technician will perform the test, entering any specific special instructions for each test, and the price of the test
5) When finished, click "close"
6) Finally, generate a sample sticker by clicking the box next to the sample name with the mouse to create an "x", then press "Print Labels"
7) Labels will print (see example below)

Samples are color coded with stickers to indicate special handling, storage or disposal procedures. The color scheme used is the same as for physical project folders (shown above).

7.4.5.4 Testing

A technician is assigned to perform each test required. GTX uses the same sample names which our clients use. Each sample identification sticker contains information related to that sample and project. This sticker is placed on the corresponding sample container. An example of GTX's sample identification sticker is shown below.

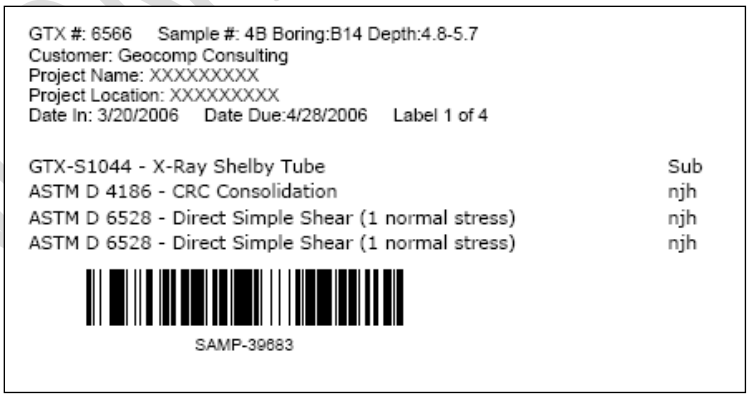

Figure 3. Sample Label

Specimens taken from samples are placed into sealed containers such as zip-loc bags, buckets, jars, etc. This container is then labeled with a sticker or a marker.

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Archive and Disposal

After testing is complete for a particular sample, the sample and any specimens originating from the sample are placed into one of the holding bins for completed work. These holding bins are set up to hold samples for sixty to ninety days. If there is a request for further testing or re-testing during this period, GTX can honor the request because the sample is in the holding bin. At the end of the holding period, the samples are either:

- Discarded
- Returned to client (if requested)
- Returned to client (if hazardous) for disposal on site
- Treated as outlined in USDA Foreign Soil Permit and discarded
- Held in long-term storage

7.5 TECHNICAL RECORDS

7.5.1

GTX maintains technical records for each laboratory project including results, report and other relevant project information. Where possible, sufficient information is maintained to facilitate, if possible, identification of factors affecting the measurement result. If sufficient sample remains, the test records will allow for the repetition of the laboratory test under conditions as close as possible to the original. The technical records shall include the date and the identity of personnel responsible for performing the test, and for checking data and results. For each test, original observations, data and calculations are recorded at the time they are made.

7.5.2

When records are revised or amended, both the original and amended data and files shall be kept. Revision records shall include the date of alteration, an indication of the altered aspects and the personnel responsible for the alterations. GTX shall ensure that revisions to technical records can be tracked to previous versions or to original observations.

7.5.3 GTX Technical Records Management

7.5.3.1

GTX has established and maintained procedures for identifying, collecting, indexing, accessing, filing, storage, maintenance, and disposal of technical and other quality records.

GTX maintains technical records chronologically and via project numbers (for project files). Physical project files are maintained in secure file cabinets in the laboratory for approximately three-four years then archived in a secure storage location until the project is approximately seven years old. Once a project has been closed for seven years or longer, the physical project folder is discarded.

7.5.3.2

The following types of technical records are maintained:

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- Project Files
  - Project verification
  - Test assignment forms
  - Data sheets or electronic test files with raw data
  - Reports
  - Calculations
  - Correspondence
- Equipment
  - Maintenance and calibration
  - Internal Reference Materials (IRM's)

7.5.3.3

GTX test reports have unique dates and number identification to segregate them from other reports. Copies of reports sent to clients are stored in the project file. A physical project file consists of at least two parts: project information (contract, PO, quote, communications, chain of custodies, etc.) in a yellow folder and laboratory test sheets in a blue folder. Electronic copies of original reports are maintained in project folder on the GTX server. Any hard copy test reports are kept in the main part of the physical folder.

7.6 EVALUATION OF MEASUREMENT UNCERTAINTY

7.6.1

GTX has adopted A2LA's "A2LA P103 - Policy on Estimating Measurement Uncertainty for Testing Laboratories" and A2LA P103d - Annex - Policy on Measurement Uncertainty for Construction Materials and Geotechnical Testing Laboratories as its own in relation to estimating uncertainty in measurements made on different types of equipment. These policies can be found on the GTX server.

7.6.2

Uncertainty budgets shall be developed for any type of measuring device calibrated by GTX for in-house use. Established uncertainty budgets shall be updated when there are any changes in the factors effecting the estimated uncertainty, such as recalibrated reference measuring devices. Uncertainty budgets for new models of measuring devices shall be determined before the device is put into service.

7.6.3

GTX will evaluate measurement uncertainty for test methods and equipment calibrations it performs as required by A2LA guidance document "G103d - Annex-Policy on Estimating Measurement Uncertainty for Construction Materials & Geotechnical Testing Labs." This A2LA guidance document can also be found on the GTX server.

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7.7 ASSURING THE QUALITY OF RESULTS

7.7.1

GTX monitors the validity of results in several ways. Where possible, the resulting data is recorded in such a way that trends are detectable and, where practicable, statistical techniques shall be applied to review the results. This monitoring data is planned and reviewed and shall include, where appropriate, but not be limited to:

7.7.1 a) GTX use of reference materials or quality control materials – IRM's.

Use of Internal Reference Materials (IRM's) helps GTX assure the quality of its work. The extent to which the factors contribute to the total uncertainty of measurement differs considerably between (type of) tests and between (types of) calibrations. GTX takes account of these factors by way of internal reference materials (IRMs) and verification checks to verify that equipment is functioning properly prior to testing. An IRM or gauge standard (e.g. calibrated weight, gauge block) is assigned to each piece of equipment. Written procedures for performing IRM or verification checks exist and are maintained electronically.

IRMs are traceable and have known values. Verification checks the critical dimensions and functionality of equipment. The frequency at which these reference checks and verifications are performed varies depending upon the piece of equipment, volume of testing performed with that equipment and likelihood of that piece of equipment to yield results which stray from accepted ranges. At a minimum, GTX performs an IRM or verification check on each piece of equipment once every three months. Typically, three IRMs are conducted each year in addition to a yearly calibration. For scales, ovens, liquid limit bases and liquid limit grooving tools, the IRM/verification check is conducted daily. An IRM schedule listing equipment and frequencies is maintained electronically. If a piece of equipment has been taken out of service, an IRM is not required to be performed until it is brought back in service. Reference and verification checks are recorded in the Laboratory Equipment Calibration Log. Reference checks can be performed by laboratory technicians or management. The Laboratory Manager maintains a list of testing equipment giving details of IRMs, verifications and calibrations for test equipment in the laboratory.

If, after performing a reference check or verification, a piece of equipment is found to be yielding unacceptable results, the Laboratory Manager is consulted and a calibration check is performed. If this does not resolve the problem, the Quality Manager or Director of Testing Services is consulted and a new course of action is determined. Actions taken to resolve the problem include: checking to determine if any previous test results may be affected and documenting corrective actions.

7.7.1 b)

ISO 17025:2017 7.711 b includes the use of alternative instrumentation to assure quality results. However, the methods performed by GTX are based on ASTM, AASHTO and other test standards that prescribe the required equipment and generally do not allow for the use of alternative test apparatus.

7.7.1 c)

Functional check(s) of measuring and testing equipment (See Section 7.7.1.a on IRM's and Section

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6.4.14 on calibration and validation).

7.7.1 d)

Use of check or working standards with control charts, where applicable.

7.7.1 e)

Intermediate checks on measuring equipment (See Section 7.7.1.a on IRM's.)

7.7.1 f)

ISO 17025:2017 7.71 f includes the use of replicate tests using the same or different methods. However, the test standards used by GTX do not require replicate testing. GTX does perform replicate tests from time to time at the request of our clients.

7.7.1 g)

ISO 17025:2017 7.7.1 g includes the retesting of retained items. If there is a question about a test result or a retest is requested by the client, GTX will retest a retained sample, when possible. However, in many cases retesting is not possible because of the lack of available sample, the condition of the sample or natural soil and rock variability that prevents repeatable results. This is particularly true of undisturbed soil samples (Shelby tubes) and rock core.

7.7.1 h)

ISO 17025:2017 7.7.1 h includes the correlation of results for different characteristics of an item. Correlation of different geotechnical test results are frequently used in civil engineering (examples: grain size and hydraulic conductivity, moisture content and organic content, unit weight and density, etc.). However, because of the natural variability of soil and rock, it is not practical to statistically analyze the correlations. GTX does take these correlations into account when reviewing test results but does not systematically record them.

7.7.1 i)

ISO 17025:2017 7.7.1 i includes the review of reported results as a measure of result quality. However geotechnical results are rarely comparable either within or between projects.

7.7.1 j)

ISO 17025:2017 7.7.1 j includes the intra-laboratory comparisons.

7.7.1 k)

Testing of blind sample(s): GTX tests blind samples as part of the AASHTO re:source proficiency testing program. Since each proficiency sample is unique, results can only be used to compare results between participating labs for a single sample. Statistical analysis of all results received is provided by AASHTO re:source for each proficiency sample provided.

7.7.2

GTX shall monitor its performance by comparison with results of other laboratories, where available and appropriate. This monitoring shall be planned and reviewed and shall include, but not be limited to, either or both of the following:

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7.7.2 a) Participation in proficiency testing

GTX Proficiency Testing Program: GTX participates in several proficiency testing programs. These programs help control and maintain that laboratory personnel are performing tests properly. GTX participates in the following proficiency testing programs:

- ASTM/ISR: American Society for Testing and Materials/Institute for Standards Research (soils)
- GAI-LAP: Geosynthetic Accreditation Institute - Laboratory Accreditation Program (geosynthetics)
- AASHTO re:source: AASHTO: (American Association of State Highway and Transportation Officials) (soils & aggregate)
- CCRL: Cement and Concrete Reference Laboratory (AASHTO) (concrete)

These programs give GTX the means to check quality of work whenever a question is raised. The procedure that GTX follows is outlined in the GTX procedure "Proficiency Test Program." If results are outside the control limits (usually 2 standard deviations from the average) the following procedures are followed:

- Determine if the agency conducting the program corrected reported results
- Determine if the test result submitted was properly taken from laboratory data
- Determine if the calculations of the results were correct
- Determine if the equipment used to perform the test is in control
- Determine if the procedures used to perform the test are in accordance with test standards
- Take corrective action to repair or replace defective equipment or inform the technician of proper technique to follow
- Document corrective actions in accordance with section 11.0 Corrective Actions

GTX retains results of participation in proficiency programs including: data sheets, reports, results, and documentation describing steps taken to determine the cause of poor test results and any corrective actions taken.

AASHTO re:source proficiency results for soil testing shall be submitted in accordance with A2LA R103. Soil proficiency results and any corrective actions, if required, shall be submitted to A2LA along with the A2LA Proficiency Testing Data Submissions Form (F104) within 30 days of receiving the results from AASHTO re:source.

7.7.2 b) participation in interlaboratory comparisons other than proficiency testing.

7.7.3

Data from monitoring activities shall be analyzed and used to control, and if applicable, improve the laboratory's activities. If the results of the analysis of data from monitoring activities are found to be

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outside pre-defined criteria, appropriate action shall be taken to prevent incorrect results from being reported.

7.7.4 On-Site Inspection Programs

Assessments performed by external agencies is an additional way to demonstrate the quality of results and, in some cases, can be used to demonstrate conformance with ISO/IEC 17025:2017 and metrological traceability.

GTX participates in various on-site inspection programs, namely: AASHTO re:source, AASHTO-CCRL, A2LA, GAI and US Army Corps of Engineers. Three aspects of the laboratory are checked during these inspections: equipment, procedures, and the quality system. GTX may also undergo project-specific quality audits conducted by its clients. The scope of these audits is project-specific.

If deficiencies are found the following steps are taken:

- Equipment is replaced or repaired
- Correct procedures are discussed and reviewed with the technician; the technician is observed performing the test properly
- The Laboratory Manager reviews deficiencies in the Quality Manual with responsible individuals; corrections are made

Corrective actions are documented and maintained by the Laboratory Manager on the GTX server. Results of on-site inspections are maintained by the Laboratory Manager on the GTX server.

GTX currently holds the accreditations and validations given in Table 1 below. Accreditation certificates and validation letters can be found in Appendix G and on the GTX server at: \\HAL3-GTX.geocomp.com\GTX\Qualifications\Lab Accreditation. Each accreditation or validation document lists the scope of tests covered by the last assessment. The documents also list the dates over which the accreditation or validation are valid.

Agency	Type	Hold Certificate	
		GTX-MA	GTX-GA
AASHTO re:source	Accreditation	YES	YES
CCRL-AASHTO	Accreditation	YES	NO
A2LA	Accreditation	YES	NO
GAI-LAP	Accreditation	YES	NO
USACOE	Validation	YES	YES

Table 1. GeoTesting Express Accreditations and Validations

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7.8 TEST DATA AND REPORTING OF RESULTS

Test reports are created from test data obtained from technicians. Technicians use laboratory work sheets or the LIMS to document and check their work. Lab work sheets are initialed by technicians prior to submitting for review. Laboratory work sheets and the LIMS are designed to minimize errors in performing and reporting the test results. Comment sections of the laboratory work sheets and LIMS are used by technicians to record deviations from applicable test standards, problems arising during the performance of the test, etc. Once checked, the Laboratory Manager initials the laboratory work sheet.

Test report sheets and laboratory work sheets are placed into the project file for the appropriate project. A project file is a collection of color coded folders which represent separate components of the file. The folder which contains all of the sub folders is green. This green folder has a sticker on the outside with pertinent project information such as:

- GTX project #
- Client name, address, contacts, phone and fax numbers, E-mail addresses
- Project name and location
- Special reporting instructions
- Disposal method

The blue folder contains all laboratory work sheets and calculations (hand and computer generated). The yellow folder contains all background project information, pricing, correspondence, records of communication, etc. The orange folder contains all computer generated test report sheets and reports as needed. Most test report sheets and reports are now stored electronically on the GTX server.

Active project files are stored in the lab file cabinets. Technicians have access to these files for information they may need to perform tests.

Each project file has the unique GTX project number located at the top to distinguish it from other files. In addition, each file has a white label on the front to relay pertinent project information.

Most test reports are e-mailed to the client upon passing the internal quality check. (See Section 7.10.4, “GTX Procedures for Identifying and Correcting Nonconforming Work” for quality checking procedure.) All test reports are generally e-mailed to the client. Final project reports are posted to the GTX Client Portal for down loading. Alternatively, GTX can e-mail or mail a final report at the client’s request.

Documents, data, calculations and work papers prepared or furnished by GTX are instruments of service and remain GTX’s property. Reports, data and other work product delivered to or on behalf of the client are for the client’s use only for the limited purposes disclosed to GTX. GTX does not disclose results contained in any report produced for the client or provided by the client without the consent of the client. GTX treats test results, data and reports prepared by GTX as proprietary and confidential. These are only disclosed to a third party with written authorization from the client. If GTX wishes to use test data for purposes other than reporting to a client, it sanitizes the data by removing client names, project names, etc.

Appendix H contains example GTX test reports. A complete set of report formats is located in the Laboratory Manager’s office. Sample report formats are sanitized to maintain customer confidentiality.

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7.8.1 General Report Requirements

7.8.1.1

The Laboratory Manager, Assistant Laboratory Manager or Director of Testing Services generally checks and approves most test reports before being transmitted to the client. If it is impossible for the Laboratory Manager, Quality Technician, Assistant Laboratory Manager or Director of Testing Services to check and approve the report, the Chief Engineer will approve the report. The test report denotes who performed the test and who checked the test/report. Reports have a comment section on them which communicates to the client any deviation from the test standard, problems, observations, etc. Reports are dated and signed by any of the following: Laboratory Manager, Quality Technician, Assistant Laboratory Manager, Director of Testing Services or the Chief Engineer.

7.8.1.2

With approval from the client, the results may be reported in a simplified way. Any information listed in 7.8.2 to 7.8.7 that is not reported to the client shall be readily available.

If corrections are required to reports, they are made and the report is checked again. In the event an incorrect report goes to a client, a new and corrected report is supplied to the client and a corrective action report is completed. Both the corrected report and the original report are considered to be technical records and shall be retained in the project file. (See 7.8.8, Amendments to Reports)

7.8.2 Reports – Common Requirements

7.8.2.1

All test results produced by GTX are submitted to the client in the form of a test report. A test report consists of one or several test report sheets, a written letter and a transmittal page. At a minimum, the test report should satisfy the conditions and instructions outlined in test methods and contract with the client. Per the requirements of ISO/IEC 17025 each report shall include at least the following information, unless the laboratory has valid reasons for not doing so, thereby minimizing any possibility of misunderstanding or misuse:

- Title of "Test Report"
- Name and address of laboratory
- If testing not performed at the GTX laboratory, the report should include the location of performance of the laboratory activities, including when performed at a client facility or in associated temporary or mobile facilities.
- Unique identification of the report (GTX project number)
- Name and address of client, and name of contact at client firm
- Identification and description of samples tested
- Characterization of condition of test sample
- Date test samples were received in laboratory and date tests were performed

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- Identification of test methods used (including any additions to, deviations, or exclusions from the methods)
- Reference to sampling procedures
- Test results with, where appropriate, the units of measurement and supported by tables, graphs, sketches, etc. which support test results
- Signature of a Laboratory Manager or other authorized member of the lab management team who accepts responsibility for the content of the report and date report issued
- Statement declaring test results relate only to the samples tested
- Clear identification when results are from external providers.
- Statement that the report shall not be reproduced except in full, without the written approval of the laboratory

It should be noted that GTX may alter the format of reports but not content to satisfy specific requests of clients. At our clients' request, GTX generally issues reports using English/Imperial units, as is the standard practice in the United States. Reports in SI units can be generated when requested or required.

7.8.2.2

GTX shall be responsible for all the information provided in the report, except when information is provided by the client.

7.8.2.2.1

Where GTX has not been responsible for the sampling stage (e.g. the sample has been provided by the client), it shall state in the report that the results apply to the sample as received.

7.8.2.2.1

In general, the correctness of sample identification provided by the client (including boring number, exploration number, sample number, sample depth, sample description, soil or rock type, etc.) is the responsibility of the client. It is GTX's responsibility to correctly report sample identification provided by others.

7.8.2.2.2

All other information provided by a client shall be clearly identified.

7.8.2.2.3

In addition, a disclaimer shall be put on the report when the information is supplied by the client and can affect the validity of results.

7.8.3 Test Reports – Specific Requirements

7.8.3.1

In addition to the requirements listed in 7.8.2, test reports shall, where necessary for the interpretation of the test results, include the following:

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- Information on specific test conditions, such as environmental conditions.
- Where relevant, a statement of conformity with requirements or specifications
- Where applicable, the measurement uncertainty presented in the same unit as that of the measurand or in a term relative to the measurand (e.g. percent) when it is relevant to the validity or application of the test results, or a client's instruction so requires, or the measurement uncertainty affects conformity to a specification limit.
- Where appropriate, opinions and interpretations.
- Additional information which may be required by specific methods, authorities, clients or groups of customers.

7.8.3.2

Where GTX is responsible for the sampling activity, test reports shall meet the requirements listed in 7.8.5 where necessary for the interpretation of test results.

7.8.4 Calibration Certificates – Specific Requirements

7.8.4.1

In addition to the requirements listed in 7.8.2, calibration certificates shall include the following:

- The measurement uncertainty of the measurement result presented in the same unit as that of the measurand or in a term relative to the measurand (e.g. percent).
- The conditions (e.g. environmental) under which the calibrations were made that have an influence on the measurement results.
- A statement identifying how the measurements are metrologically traceable
- The results before and after any adjustment or repair, if available.
- Where relevant, a statement of conformity with requirements or specifications
- Where appropriate, opinions and interpretations

7.8.4.2

It is extremely rare that a GTX calibration procedure would require sampling. However, if sampling is required for a calibration, ISO 17025 requires that the calibration certificates shall meet the requirements listed in "Reporting Sampling", 7.8.5, if necessary for the interpretation of test results.

7.8.4.3

GTX is required to conform to ASTM D3740, "Standard Practice for Minimum Requirements for Agencies Engaged in Testing and/or Inspection of Soil and Rock as Used in Engineering Design and Construction." This standard specifies calibration intervals for geotechnical laboratories. Therefore, GTX lists a calibration date and the next calibration due date for each piece of calibrated equipment. These dates are included on each of our calibration certificates and calibration labels. This runs counter to ISO/IEC 17025 7.8.4.3 which states that calibration certificates or calibration labels shall not contain any recommendation on the calibration interval except where this has been agreed with

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the customer. GTX will comply with the ISO requirement stated in 7.8.4.3 and issue a calibration certificate without a calibration interval if specifically requested to by a client or agency. GTX equipment labels, however, will continue to list a due date for the next calibration.

7.8.5 Reporting Sampling

Where GTX is responsible for the sampling activity, in addition to the requirements listed in 7.8.2, reports shall include the following, where necessary for the interpretation of results:

- The date of sampling.
- Unique identification of the item or material sampled.
- The location of sampling, including any diagrams, sketches or photographs.
- A reference to the sampling plan and sampling method.
- Details of any environmental conditions during sampling that affect the interpretation of the test results.
- Information required to evaluate measurement uncertainty for subsequent testing or calibration.

7.8.6 Reporting Statements of Conformity

7.8.6.1

As a rule, GTX does not make statements of conformity to a specification. That is for our clients to determine based on our test results.

7.8.6.2

If a statement of conformity to a specification or standard is provided, GTX shall document the decision rule employed, taking into account the level of risk (such as false accept and false reject and statistical assumptions) associated with the decision rule employed and apply the decision rule. If GTX is required by a client to report a statement of conformity it shall identify the following:

- To which results the statement of conformity applies.
- Which specifications, standard or parts thereof are met or not met.
- The decision rule applied (unless it is inherent in the requested specification or standard).

7.8.7 Reporting Opinions and Interpretations

GTX reports provide the results of tests. Opinions and interpretation of the GTX test results are generally left to the professional judgement of our clients. Our client's opinions and interpretation are not included in GTX test reports unless the client specifically requests that they be included. If a client's opinions and interpretation are included in a GTX report they would be clearly noted as coming from the client. If GTX is requested to state its opinions and interpretation in a report than that report shall conform to Sections 7.8.7.1 to 7.8.7.3 below.

7.8.7.1

When opinions and interpretations are expressed, GTX shall ensure that only personnel authorized

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for the expression of opinions and interpretations releases the respective statement. Authorized personnel include the Laboratory Manager, Quality Technician, Laboratory Engineer and the Director of Testing Services. GTX shall document the basis upon which the opinions and interpretations have been made.

7.8.7.2

The opinions and interpretations expressed in reports shall be based on the results obtained from the tested item and shall be clearly identified as such.

7.8.7.3

When opinions and interpretations are directly communicated by dialogue with the client, a record of the dialogue shall be retained.

7.8.8 Amendments to Reports

7.8.8.1

When an issued report needs to be changed, amended or re-issued any change of information shall be clearly identified and, where appropriate, the reason for the change included in the report.

7.8.8.2

Amendments to a report after issue shall be made only in the form of a further document, or data transfer, which includes the statement: "Amendment to Report", or an equivalent form of wording. Such amendments shall meet all the requirements of this document.

7.8.8.3

When it is necessary to issue a complete new report, this shall be uniquely identified and shall contain a reference to the original that it replaces.

7.9 COMPLAINTS

7.9.1

A complaint is defined by ISO 17025:2017 as an expression of dissatisfaction by any person or organization to a laboratory, relating to the activities or results of that laboratory, and where a response is expected.

GTX has a documented process to receive, evaluate and make decisions on complaints. If a complaint is received by the laboratory, the Quality Manager or Director of Testing Services completes a Complaint Form. Resolution of the complaint is recorded on the Complaint Form and maintained in the corrective action file.

7.9.2

The GTX Complaint Form and the corrective response process (Section 8.7) describes the handling process for complaints receive by GTX. The GTX complaint documentation is available to any client or accreditation agency on request. Upon receipt of a complaint, GTX shall confirm whether the complaint relates to laboratory activities that it is responsible for and, if so, shall address it. GTX shall be responsible for all decisions at all levels of the handling process for complaints.

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7.9.3

The GTX Complaint Form and the corrective action procedure includes the following:

- Description of the process for receiving, validating, investigating the complaint, and deciding what actions are to be taken in response to it.
- Tracking and recording complaints, including actions undertaken to resolve them.
- Ensuring that appropriate action is taken.

7.9.4

The GTX manager receiving the complaint shall be responsible for gathering and verifying all necessary information to validate the complaint. If a GTX technician directly receives a communication in any form that maybe considered a complaint, the technician shall discuss the communication with a Lab Manager. The Lab Manager will begin the complaint process based on the discussion with the lab technician.

7.9.5

Whenever possible, GTX shall acknowledge receipt of the complaint, and provide the complainant with progress reports and the outcome.

7.9.6

The outcomes to be communicated to the complainant shall be made by, or reviewed and approved by, individual(s) not involved in the original laboratory activities in question.

7.9.7

Whenever possible, GTX shall give formal notice of the end of the complaint handling to the complainant.

7.10 MANAGEMENT OF NONCONFORMING WORK

7.10.1

The following is GTX's procedure for non-conforming work. It shall be implemented when any aspect of its laboratory activities or results of this work do not conform to its own procedures or the agreed requirements of the client. The procedure shall ensure that:

- The responsibilities and authorities for the management of nonconforming work are defined.
- Actions (including halting or repeating of work and withholding of reports, as necessary) are based upon the risk levels established by GTX.
- An evaluation is made of the significance of the nonconforming work, including an impact analysis on previous results.
- A decision is taken on the acceptability of the nonconforming work.
- Where necessary, the client is notified and work is recalled.
- The responsibility for authorizing the resumption of work is defined.

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7.10.2

GTX shall retain records of nonconforming work and actions as specified in 7.10.1.

7.10.3

Where the evaluation indicates that the nonconforming work could recur or that there is doubt about the conformity of the laboratory's operations with its own management system, the laboratory shall implement corrective action.

7.10.4 GTX Procedures for Identifying and Correcting Nonconforming Work

Test data used to create a report is checked by the Laboratory Manager, Quality Technician, Director of Testing Services, Quality Manager, Chief Operating Officer, or Chief Engineer. Final reports are checked, signed and dated by the Laboratory Manager, Quality Manager, Director of Testing Services, Chief Operating Officer or Chief Engineer before being delivered to the client. Laboratory work sheets and test reports have a comments section where any out of ordinary, pertinent information is included concerning the test.

If an error is detected in the test data, the Laboratory Manager performs a preliminary investigation. If the preliminary investigation finds and resolves the problem, re-testing is performed. If, upon the preliminary investigation, there is no acceptable reason found for the error, the Quality Manager is consulted and a more in depth investigation is performed. Findings and resolutions are documented.

Nonconforming reports identified by the client are corrected, checked by the Laboratory Manager, Quality Technician, Director of Testing Services, Quality Manager, Chief Operating Officer or Chief Engineer and shipped to the client. Both the re-submitted report and the nonconforming report are kept in the project file. If re-testing is required to remedy the situation, it is done at no cost to the client. Again, nonconforming data and/or reports require documentation in the form of a corrective action report. Customer complaints require documentation by way of the corrective action report.

After nonconforming testing and/or calibrations are corrected, it is the responsibility of the Laboratory Manager, Quality Technician, Director of Testing Services, Quality Manager, Chief Operating Officer, or Chief Engineer to authorize the resumption of work.

7.11 CONTROL OF DATA – INFORMATION MANAGEMENT

ISO/IEC 17025 requires the following for the control of data and laboratory information management:

- GTX shall have access to the data and information needed to perform laboratory activities.
- The laboratory information management system (LIMS) used for the collection, processing, recording, reporting, storage or retrieval of data shall be validated for functionality, including the proper functioning of interfaces within the laboratory information management system(s) by the laboratory before introduction. Whenever there are any changes, including laboratory software configuration or modifications to commercial off-the-shelf software, they shall be authorized, documented and validated before implementation. (See Section 7.2.3.7.)
- The laboratory information management system shall:
    - Be protected from unauthorized access.

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- Be safeguarded against tampering and loss.
- Be operated in an environment that complies with supplier or laboratory specifications or, in the case of non-computerized systems, provides conditions which safeguard the accuracy of manual recording and transcription.
- Be maintained in a manner that ensures the integrity of the data and information.
- Include recording system failures and the appropriate immediate and corrective actions.

  See Section 7.2.3.7 for details.

- GTX manages and maintains its LIMS and laboratory data control system with help from the company's Information Technology Department (IT.) IT shall comply with all applicable requirements of the GTX Quality Manual. GTX does not use an external provider to manage or maintain the control of data.
- All GTX SOP's, manuals and reference data relevant to the laboratory information management systems are made readily available to personnel. (See Section 8.3, "Control of Management System Documents")
- Calculations and data transfers shall be checked in an appropriate and systematic manner.

8 MANAGEMENT REQUIREMENTS

GTX's management is committed to good professional practice and to providing quality testing services to its clients. In addition, GTX's management is committed to continually improving the effectiveness of the management system. Testing services are conducted in accordance with stated methods and clients' instructions. GTX's standard of service is to score a 4 or higher on both its customer survey and proficiency tests. These results are tracked and improvement is expected over time. All personnel of GTX are required to familiarize themselves with the GTX quality system and appropriate documentation. This quality system includes the following elements:

- GTX has in place a quality plan which is described in the Quality Manual. This Manual is kept up to date by the Quality Manager.
- Prior to embarking on a new project, a review is required to determine if all the necessary resources for the execution of the project are available in-house and are capable of completing all tasks as required by the client. This includes equipment and technicians. Both must have passed quality system reviews to determine fitness (calibrations, accuracy for equipment and training for technicians).
- GTX performs an internal quality audit once per year. As part of this audit, quality documents are updated and reviewed by the Quality Manager, and Laboratory Managers.
- Management and staff of GTX maintain current memberships in outside organizations such as ASTM, ASCE, NAGS, etc. which enable them to meet or exceed current practices in the geosynthetics and soils industry.

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- Technicians sign their work off in the appropriate location on laboratory work sheets; whoever performs the peer review also signs off on the laboratory work sheet. Final reports are signed by the Laboratory Manager, Chief Engineer, Laboratory Director, Lab Manager, Quality Manager, Assistant Laboratory Manager or Quality Manager as part of the final verification.
- GTX performs tests in accordance with published standards and with assistance from their own standard operating procedures. These procedures are documented and available to all persons within GTX and help reduce ambiguity where confusion may otherwise exist. These documents are available to clients upon request.
- GTX tracks each individual project entering the laboratory with its own unique sequential project identification number.

GTX's quality system objectives are as follows:

- Provide quality test results by conducting testing in accordance with published standards, accepted industry practice and clients' instructions within the time required by the client.
- Continually improve quality by self-evaluation, process review and documentation.
- Constantly meet or exceed our clients' expectations.

GTX's quality goals are summarized in Table 2 below.

Indicator	Measurement	Level of Service (Goal)
Service to clients	Survey response	≥4
	Client complaints per year	0
Proficiency tests	Rating	≥3
Safety	Incidents per year	0
Production	Yearly targets	100%
Turnaround time	On or ahead of schedule per week	95%
Process control	IRM rating	±2 standard deviations

Table 2. GTX Quality Goals

8.1 ISO/IEC 17025:2017 MANAGEMENT OPTIONS

8.1.1 General

ISO/IEC 17025:2017 requires that laboratories establish a management system that follows one of two options, A or B. Option A follows the management of requirements of ISO/IEC 17025:2005. Option B is

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new and requires a management system that is in accordance with the requirements of ISO 9001. In addition, the management system shall meet the requirements of clauses 4 to 7 of ISO/IEC 17025:2017.

GeoTesting Express has decided to follow the requirements of Option A and continue to follow the management system outlined in ISO/IEC 17025:2005 with the addition of the new section 8.5 that addresses risks and opportunities.

8.1.2 Option A (17025:2005)

As a minimum, the GTX management system of the laboratory shall address the following:

— management system documentation (see 8.2).

— control of management system documents (see 8.3).

— control of records (see 8.4).

— actions to address risks and opportunities (see 8.5).

— improvement (see 8.6).

— corrective actions (see 8.7).

— internal audits (see 8.8).

— management reviews (see 8.9).

8.1.3 Option B (9001:2015)

GTX is not following the Option B Management system. The Option B Management System is defined as:

A laboratory that has established and maintains a management system, in accordance with the requirements of ISO 9001, and that is capable of supporting and demonstrating the consistent fulfilment of the requirements of clauses 4 to 7 of ISO/IEC 17025 also fulfils at least the intent of the management system section requirements (8.2 - 8.9)

8.2 MANAGEMENT SYSTEM DOCUMENTATION (OPTION A)

8.2.1

The GTX Quality Manual and quality procedures document the GTX management system. These procedures are followed by employees of GTX. GTX employs a management team appropriate to the volume and nature of laboratory testing conducted. The Quality Manager is responsible for implementing the quality system. The Quality Manual is electronically maintained on GTX's server as a read-only protected file. It is available to each employee for viewing. The Quality Manager is responsible for distribution and maintenance of controlled documents related to the quality system.

8.2.2

The quality system outlined in this Quality Manual is designed to meet requirements in ANSI/ISO/IEC 17025:2017. Procedures used to maintain compliance with the GTX quality system and ANSI/ISO/IEC 17025:2017 are documented below in Sections 8.3 – 8.9. Employees of GTX are responsible for adhering to procedures outlined in the Quality Manual while satisfying our clients' needs.

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Improvements to procedures are coordinated with proper authority to assure uniform compliance. Changes are made to existing systems only after a review on the effect of such changes is performed. Employees of GTX have access to the appropriate documentation needed to implement the quality system.

8.3 CONTROL OF MANAGEMENT SYSTEM DOCUMENTS (OPTION A)

8.3.1

Documents relating to the GTX quality plan are controlled. These include the Quality Manual; test procedures; documents required by GTX to ensure effective planning, operation or and control of its processes; and records required by ISO 17025. Records are maintained indicating controlled documents and those who possess them. Controlled Documents are located where appropriate. Test standards are available in the laboratory. GTX Standard Operating Procedures are located in the Laboratory Manager's office and on the GTX server. GTX's Quality Manual is maintained electronically on GTX's server. SOPs and the Quality Manual are available to employees of GTX.

8.3.2

Controlled documents are reviewed and approved by the Quality Manager and/or Director of Testing Services. Controlled documents (such as the Quality Manual, SOPs, standards and report blanks) are numbered by a unique identification system. Persons receiving a copy of a controlled document are assigned a unique numbered copy of that document. A master list of control documents is maintained which contains the following:

- Unique number or name of controlled document
- Current revision # in use
- Date of issue of current revision

This system allows for tracking current as well as obsolete copies. Master copies of current controlled documents are kept with the Laboratory Manager, or the Quality Manager. The master list and each controlled document's list are maintained by and kept with the Quality Manager.

It is possible to circulate an uncontrolled copy of a controlled document. The cover of the document must clearly state "uncontrolled document" or "UC".

Invalid and/or obsolete controlled documents are removed from those who possess them. This is possible by utilizing the controlled document lists to locate current copies. Uncontrolled copies do not need to be retrieved and/or replaced. Obsolete documents are marked with an "Obsolete" stamp. One copy of obsolete documents is retained for historical preservation and the remainder destroyed.

8.3.3

The GTX Quality System uses a variety of supporting procedures and reference documents. The documents have been organized into a hierarchical structure that is outlined in the GTX Document Control List. The hierarchical structure is organized as follows:

1. Quality Manual

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2. Test Standards/Methods
3. Test Standard Operating Procedures
4. GTX Operating Procedures
5. Form - Test Report Blanks
6. Form - Lab Data Sheets
7. Test Exams
8. Calibration/Verification Standard Operating Procedures
9. Internal Reference Material Standard Operating Procedures
10. A2LA Normative Documents
11. ISO Normative Documents
12. Other Normative Documents

8.3.4

All GTX quality documentation, processes, systems, records are included in, referenced from, or linked to the management system.

8.3.5

All GTX personnel involved in laboratory activities have access to the parts of the management system documentation and related information that are applicable to their responsibilities. Management system documents are maintained either on the GTX lab server or hardcopies are available in the lab manager's office.

8.3.6

For the tests it performs, GTX reviews each revision of a test standard as the revisions are published. This review occurs within one month of receiving notification of the latest published standards. (e.g. ASTM revises every 5 to 8 years, AASHTO issues revised standards once every two years). The review consists of:

- Determining which standards have been updated (GTX Administrative Assistant)
- Revising our master controlled document list to reflect the updated test standard (GTX Administrative Assistant)
- Determining if the test standard revision requires a revision to GTX's standard operating procedures (Lab Manager, Assistant Lab Manager, Quality Manager, Quality Technician)
- Revising GTX's standard operating procedures (if necessary) (Lab Manager, Assistant Lab Manager, Quality Manager, or Quality Technician)
- Revising our master controlled document list (if necessary) to reflect the updated standard operating procedure (Administrative Assistant, Lab Manager, Assistant Lab Manager, Quality Manager, or Quality Technician)

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- Determining if the test standard revision requires a revision to GTX's lab test data sheet
- Revising GTX's lab test data sheet (if necessary) (Lab Manager, Assistant Lab Manager, Quality Manager, or Quality Technician)
- Revising our master controlled document list (if necessary) to reflect the updated lab test data sheet (Administrative Assistant, Lab Manager, Assistant Lab Manager, Quality Manager, or Quality Technician)
- Determining if the test standard revision requires a revision to GTX's standard report format (i.e. verifying if all elements given in the "Report" section of the test standard are recorded by GTX) (Lab Manager, Assistant Lab Manager, Quality Manager, or Quality Technician)
- Revising GTX's standard report format (if necessary) (Lab Manager, Assistant Lab Manager, Quality Manager, or Quality Technician)
- Revising our master controlled document list (if necessary) to reflect the updated standard report format (Administrative Assistant, Lab Manager, Assistant Lab Manager, Quality Manager, or Quality Technician)

Changes to controlled documents are reviewed and approved as stated in section 8.3.2. Each year, documents are re-issued if there have been revisions or changes made, however procedural changes are issued and used immediately following the revision. When changes are made to controlled documents, the Quality Manager or Director of Testing Services is responsible for updating current copies of the manual

Master copies of documents are "read only" protected and can only be modified by the Quality Manager or Director of Testing Services.

8.4 CONTROL OF RECORDS (OPTION A)

GTX has established and maintained procedures for identifying, collecting, indexing, accessing, filing, storage, maintenance and disposal of quality and technical records.

8.4.1

The following types of quality records are maintained:

- Technical Records (see Section 7.5.3)
  - Project Files
  - Equipment Records
- Quality Assurance
  - Management review
  - Internal and external audits
  - Corrective action file
  - Client survey

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- Customer complaint
- Preventative action

- Personnel
  - Training log
  - Resumes

8.4.2

Physical project files (hardcopies) are maintained until the project is approximately seven years old. Electronic project files are maintained indefinitely. Other quality documents are maintained indefinitely in electronic form.

8.4.3

Access to project records are consistent with client confidentiality commitments. Project records shall not be released without the consent of GTX's client.

8.5 ACTIONS TO ADDRESS RISKS AND OPPORTUNITIES (OPTION A)

8.5.1

Options to address risks can include identifying and avoiding threats, taking risk in order to pursue an opportunity, eliminating the risk source, changing the likelihood or consequences, sharing the risk, or retaining risk by informed decision.

Opportunities can lead to expanding the scope of the laboratory activities, addressing new customers, using new technology and other possibilities to address customer needs.

The GTX management system includes a varieties of ways to identify and address risks and opportunities under Option A. GTX considers the risks and opportunities associated with the laboratory activities in order to:

- Give assurance that the management system can achieve its intended results
- Enhance opportunities to achieve the purpose and objectives of GTX
- Prevent, or reduce, undesired impacts and potential failures in the laboratory activities
- Achieve improvement.

8.5.2

As outlined below in Section 8.5.4, the GTX management system includes multiple ways to address risks and opportunities. In addition, the management system is designed to integrate and implement risk and opportunity actions into its management system and to evaluate the effectiveness of these actions.

8.5.3

Proposed actions taken to address risks and opportunities shall be reviewed and evaluated to make sure that the actions are proportional to the potential impact on the validity of laboratory results.

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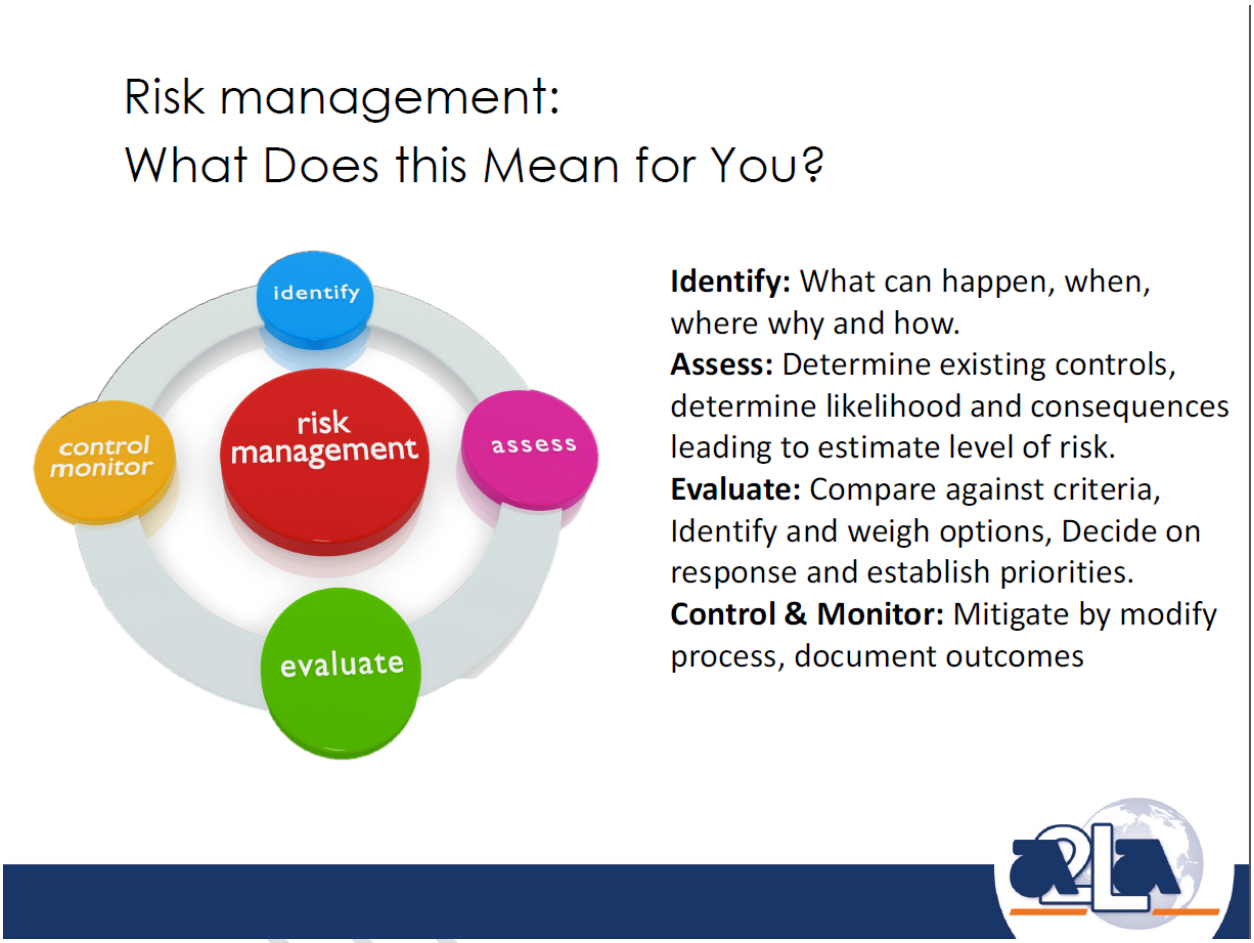

Figure 4. Steps for Risk Management

8.5.4 GTX Procedures related to Risk Management

GTX Actions Used to Identify, Access and Evaluate, Control and Monitor Risk and Opportunity:

- Reviews of risk to GTX's Impartiality
- Review of Client project inquires, quotes, project planning, and contracts
- Project test assignment reviews
- Weekly Staff meetings: in particular, the agenda items that cover Continual Improvement, Preventative Actions, Risk Management
- Bi Weekly Management meetings
- Continual Improvement, Preventative Action, Risk Management Action procedures
- Document Control reviews

- ASTM Tracker Alert (identifies revised test standards)
- Client communication and follow-up discussions between managers and staff
- Vendor List process (vendors reviewed and rated)
- Client complaints
- Corrective Actions and Root Cause Analysis
- Internal Audits
- Management Quality Review
- Results from Internal Reference Materials (IRM's)
- Results from Equipment Calibration and Verification
- Proficiency Testing

8.5.5 GTX Risk Management Assessment and Evaluation

When a new risk or opportunity is identified, it can be assessed, evaluated and recorded by following any of the established processes listed above or it can be reviewed using the GTX Risk Management Assessment form.

8.6 IMPROVEMENT (OPTION A)

8.6.1

GTX strives for continuous improvement in its procedures. The effectiveness of GTX's efforts can be measured by the following means over time:

- Client survey results
- Internal reference material (IRM) testing
- Corrective action files

All of the above are integral parts of our quality system and are reviewed by management at regular intervals.

8.6.2

GTX seeks feedback, both positive and negative, from its clients. The feedback is reviewed and used to improve all phases of the lab's performance. GTX's level of service can be measured by the results of its client satisfaction survey. The survey is sent to each client upon completion of a project. The survey asks the client to rate GTX on a number of different items on an escalating scale (1-5). GTX's goal is to receive a grade of 4 or higher (1 = failed to meet expectations and 5 = exceeded expectations). GTX aims to continuously improve its quality system in order to better serve our clients. A return rate of 10% is required to validate the survey. It is our policy to respond to all complaints received in the customer survey. Data obtained in the customer survey is discussed in quarterly management review meetings.

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8.7 CORRECTIVE ACTION (OPTION A)

Investigations are performed when non-conformities are found in the quality system or from the analysis of materials, test reports or customer complaints. The Laboratory Manager performs a preliminary investigation of all complaints. The investigation shall include a documented Root Cause Analysis of the problem in accordance with the requirements of ISO 17025 section 8.7.1b. If the preliminary investigation finds the problem, GTX will deal with the consequences and correct the problem and resolve any related issues associated with the non-conformity. Once the problem has been corrected, the process is documented in a Corrective Action Report. If, upon the preliminary investigation, there is no acceptable reason found for the error, the Quality Manager and/or Director of Testing Services is consulted and a more in depth investigation is performed. Once a suitable resolution is determined, the findings, Root Cause Analysis, resolutions, and consequences are documented in a Corrective Action Report. The Corrective Action Report is to include a review to determine if similar nonconformities exist, or could potentially occur. In addition, the corrective action should be evaluated to determine if the non-conformity or the corrective action require changes to the management system and, if necessary, update risks and opportunities determined during planning.

A corrective action file is maintained on the GTX server to document non-conformities. Corrective Action Reports are filled out by the Laboratory Manager or Quality Manager. Corrective Action Reports are reviewed by the Management at the end of each year. The reports are reviewed to evaluate the effectiveness of any corrective action take. Appropriate actions are taken to eliminate the possibility of the same problem occurring again. The laboratory staff is part of this process.

A corrective action summary list is constructed and maintained by the Laboratory Manager. This list helps aid in tracking customer complaints, audit deficiencies, etc. A yearly review is conducted with the aid of information contained in this list.

8.8 INTERNAL AUDITS (OPTION A)

Procedures for GTX Internal Audits are documented in Section 8.8. Internal quality audits are performed annually between February and June. The audit shall cover the previous calendar year. The internal audit shall verify if quality activities and related results comply with the GTX quality management system and the requirements set forth in ISO/IEC Guide 17025:2017. The Quality Technician and/or Director of Testing Services perform these audits.

The Quality Audit includes an examination of the following areas of GTX's Quality System:

- Quality Manual
- Corrective Action Summary List during the past year
- Review of previous year's corrective actions.
- Review effectiveness of past corrective actions to verify that issues remain resolved.
- External Audits performed during the past year
- Customer complaints from the past year
- Equipment problems

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- Organizational structure of the laboratory
- Results of proficiency tests during the past year
- Laboratory & field testing
- Training
- Review of controlled documents including Normative References and the Document Master Control List
- Results of customer surveys
- Any other quality issue

The audit shall also take into consideration the importance of the laboratory activities, changes affecting the laboratory, and the results of previous audits.

In addition, the audit shall consider the risks and opportunities associated with the laboratory activities that were identified in the prior year and evaluate the effectiveness of any related actions implemented.

Results of quality audits are recorded and reviewed with GTX management. Corrective actions, if necessary, are implemented without undue delay. Corrective actions are followed-up to verify implementation and effectiveness. The actions taken are documented. Results of quality audits are maintained by the Laboratory Manager in the Laboratory Manager's office and on the GTX server.

If deficiencies are determined by customer complaints, peer review and/or external audits, an increase in internal quality audits will occur. These increased internal quality audits will be in place until deficiencies are rectified.

The Quality Manager is responsible to schedule reminders for the annual Management Review on the GTX Calendar.

8.9 MANAGEMENT REVIEWS (OPTION A)

8.9.1

Procedures for GTX Management Reviews are documented in Section 8.9. The annual management review of the quality system is performed each year after the completion of the internal quality audit. The laboratory management review is to ensure the continuing suitability, adequacy and effectiveness of the GTX quality system in relation to the GTX quality document and the requirements of ISO/IEC 17025:2017.

The management review shall cover the previous calendar year. The management review shall be completed by August 31 at the latest. The management review is the responsibility of the Director of Testing Services with assistance from the Quality Technician and Laboratory Managers. The Director of Testing Services is responsible to schedule reminders for the annual Management Review on the GTX Calendar.

8.9.2

Topics covered during the review of the previous year include:

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- Review of previous year:
  - Changes in internal and external issues that are relevant to the laboratory, including any quality system changes
  - Fulfilment of objectives
  - Suitability of policies and procedures
  - Status of actions from previous management reviews
  - Outcome of recent internal audits including resolution of any related deficiencies
  - Corrective action reviews
  - Review effectiveness of past corrective actions to verify that issues remain resolved.
  - Assessments by external bodies including resolution of any related deficiencies
  - Changes in the volume and type of the work or in the range of laboratory activities
  - Customer and personnel feedback;
  - Complaints
  - Effectiveness of any implemented improvements
  - Adequacy of resources
  - Results of risk identification
  - Outcomes of the assurance of the validity of results including proficiency test results
  - Other relevant factors, such as monitoring activities and training.
- Review of Actions to Address Risks and Opportunities:
  - Review risks and opportunities identified, accessed, evaluated, and controlled or monitored.
  - Review risks to the labs impartiality that were identified since the last Management Review and determine if they were adequately addressed.
  - Review audit deficiencies to determine if required actions are underway, completed or not begun.
  - Review QM Section 5.5 to determine if any position responsibilities need updating, revision or improvement.
  - Review QM Section 5.5.c.1 to determine if any management responsibilities need updating, revision or improvement.
  - Based on the findings of this Management Review were there additional risks to the labs impartiality identified?
  - Based on the findings of this Management Review were there additional risks or opportunities identified?
- Planning for coming year:

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- Preventative action
- Recommendations for improvement
- Resource (personnel and equipment) needs
- Reports from management and supervisory personnel (addressing trends, equipment wish lists, suggestions for improvement, etc.)
- Changes in volume and type of work
- Action items stemming from review with due dates

8.9.3

The outputs from the management review shall record all decisions and actions related to at least:

- The effectiveness of the management system and its processes
- Improvement of the laboratory activities related to the fulfilment of the requirements of this document
- Provision of required resources
- Any need for change.

8.9.4

A copy of the minutes from the management review is provided to the Chief Engineer. Records of management review are maintained and kept in the Director of Testing Services' office.

9 SUPPLEMENTAL INFORMATION

9.1 ADVERTISING POLICY

GTX has adopted A2LA's "R105 – Requirements When Making Reference to A2LA Accredited Status" as its own policy in relation to advertising the A2LA logo. This is given in GTX's Standard Operating Procedure GTX-AD004 and can be found in the Lab Manager's office and on the GTX server.

Whenever the A2LA logo is used in GTX advertising materials it will be accompanied by the GTX A2LA accreditation certificate number 2965.01.

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10 SUMMARY OF CHANGES FROM VERSION 14.0

- Title page: Revised document revision number, date of issue, controlled document number, Joe Tomei's title, and copyright date.
- All headers: Revised document revision number.
- All footers: Revised document revision number, date of issue, and controlled document number.
- Updated Table of Contents.
- Corrected typos and edited short sections of text to improve clarity.
- Section 2 Normative References:
  - Updated ANSI/ASQC Q9001 version to 2015
  - Added A2LA R103a – Annex – Proficiency Testing for ISO-IEC 17025 Laboratories
  - Removed A2LA R104 – General Requirements: Accreditation of Field Testing and Field Calibration Laboratories
  - Added ASTM D653
- Moved updated Figures 1 and 2, Organizational Charts, from Sections 5.1 and 5.2 to new Appendix A.
- Added Appendix B, GeoTesting Express, Inc. Laboratory Activities.
- Rewrote GTX Job Descriptions and moved them from Section 5.5.b.3 to new Appendix C.
- Moved Figure 2, GTX Training Record, from Section 6.2 to new Appendix D and revised text in Section 6.2 as appropriate.
- Added Technician Training Matrix to Appendix D.
- Added Appendix E, Laboratory Floor Plan, and added reference to Appendix E in Section 6.3.
- Moved Table 1, Laboratory Equipment Calibration and Verification Requirements, from Section 6.4.14 to new Appendix F.
- Added Appendix G, GTX Accreditation and Validation Certificates.
- Removed Figures 6-11, Example Test Reports, from Section 7.8 and placed example reports in a new Appendix H containing examples of all soil and rock test reports.
- Renumbered remaining figures and tables.
- Quality Manual 14.0 showed New ISO/IEC 17025:2017 requirements are in RED. However, the A2LA assessment of 2020 pointed out that a number of new requirements were missed in Quality Manual 14.0. Quality Manual 14.1 corrected these omissions in the following sections:
  - 5.3, 6.4.13, 7.1.1, 7.13, 7.2.1.5, 7.7.2 a, 7.7.2 b, 7.8.1.2, 7.8.7.3, 7.10.1, 7.10.2, 7.11, 8.1.1, 8.7, and 8.8
- Rewrote Sections 5.1 – 5.7.

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- Added the following to Section 6.2: “The Laboratory Manager and Quality Control Technician are also responsible for determining when a Technician has reached a higher Technician training level.”
- Added reference to GTX Purchasing SOP, QD16 to Section 6.6.
- Removed Section 7.1.9.3 on Selection of Test Methods. Section content was already covered word for word in Section 7.2.3.2 Selection of Methods.
- Renumbered Section 7.1.9.4.
- Added reference to GTX Sample Handling SOP, QD019 to Section 7.4.1.
- Added new category, Rush Sample, indicated by a green marker, to Figure 4, “Sample/Folder Color Key” in Section 7.4.5.2.
- Corrected bulleting on list of technical records in Section 7.5.3.2.
- Corrected CCRL reference in Section 7.7.2.a.
- Revised last sentence of Section 7.8.1.2.
- Added ISO 17025:2017 definition of complaint to Section 7.9.1.
- Section 7.10.1: Added new 1st sentence: “The following is GTX's procedure for non-conforming work.” Added to document 7.10 as GTX’s procedure.
- Rewrote 1st sentence of 2nd bullet of Section 8.
- Added new 1st sentence to Section 8.2.1 stating that the GTX management system is documented in the Quality Manual and other GTX quality documents. Moved old first sentence from QM 14.1 to 3rd sentence.
- Added new 2nd sentence to Section 8.2.2 indicating that procedures for the management system are listed in Sections 8.3-8.9.
- Added new 1st bullet to Section 8.5.4 adding reviews of risk to impartiality.
- Added new 1st sentence to Section 8.8 indicating that procedures for internal audits are documented in Section 8.8.
- Deleted “effectiveness” from 3rd bullet of Section 8.8.
- Added new 4th bullet to Section 8.8, Review effectiveness of past corrective actions to verify that issues remain resolved.
- Added new 1st sentence to Section 8.9 indicating that procedures for management review are documented in Section 8.9.
- Added new 7th sub-bullet to 1st main bullet of Section 8.9.2, Review effectiveness of past corrective actions to verify that issues remain resolved.
- Added 2nd main bullet and seven new 7th sub-bullets to Section 8.9.2 covering Review of Actions to Address Risks and Opportunities.
- Added Summary of Changes section at end of document.

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USACE CERTIFICATE OF LABORATORY VALIDATION

ERDC
ENGINEER RESEARCH & DEVELOPMENT CENTER

GeoTesting Express, LLC

125 Nagog Park
Acton, MA, United States
Joe Tomei
(978) 893-1241

has demonstrated, by abbreviated audit of its AASHTO accreditation, or by inspection of required records, equipment, procedures, facilities, and/or final reports, its proficiency to perform testing of construction materials, as established by the quality standards of AASHTO R 18 guidance and the requirements of the applicable ASTM standards.

THIS USACE CERTIFICATE OF LABORATORY VALIDATION IS ACCURATE AS OF ITS DATE AND TIME OF GENERATION:
16 JUL 2024 AT 07:27 HOURS

ALL METHODS LISTED ON THIS CERTIFICATE OF VALIDATION WILL EXPIRE ON 04/10/2025

PLEASE CONFIRM THE CURRENT VALIDATION STATUS OF THIS LABORATORY USING THE SEARCH FEATURE ON OUR PUBLIC WEBSITE: https://mtc.erdc.dren.mil

Chad A. Gartrell, PE, Director
USACE Materials Testing Center
Vicksburg, Mississippi, USA

AGGREGATE

Aggregate - C 29 - Unit Weight and Voids in Aggregate
Aggregate - C 40 - Organic Impurities
Aggregate - D 75 - Sampling
Aggregate - C 117 - Material Finer than 75 µm (No. 200) Sieve
Aggregate - C 123 - Lightweight Particles
Aggregate - C 127 - Specific Gravity & Absorption in Coarse Aggregate
Aggregate - C 128 - Specific Gravity & Absorption in Fine Aggregate
Aggregate - C 131 - Los Angeles Abrasion Resistance on Small-Size Coarse Aggregate
Aggregate - C 136 - Sieve Analysis of Aggregates
Aggregate - C 142 - Clay Lumps
Aggregate - E 329 - Standard Specification for Agencies Engaged in Construction Inspection, Testing, or Special Inspection
Aggregate - C 535 - Los Angeles Abrasion Resistance on Large Size Coarse Aggregate
Aggregate - C 566 - Total Moisture Content
Aggregate - C 702 - Reducing Samples to Testing Size
Aggregate - C 1077 - Concrete and Concrete Aggregate Testing Standards (Quality Standards)
Aggregate - C 1252 - Uncompacted Void Content of Fine Aggregate (as influenced by particle shape, surface texture, and grading)
Aggregate - D 2419 - Sand Equivalent Value
Aggregate - D 4791 - Flat and Elongated Particles in Course Aggregate
Aggregate - D 5821 - Percentage of Fractured Particles in Coarse Aggregate

CONCRETE

Concrete - C 31 - Making and Curing Test Specimens in the Field
Concrete - C 39 - Compressive Strength of Cylindrical Specimens
Concrete - C 138 - Unit Weight and Air Content by Gravimetric
Concrete - C 143 - Slump
Concrete - C 172 - Sampling
Concrete - C 231 - Air Content by Pressure ***required if C173 not performed***
Concrete - E 329 - Standard Specification for Agencies Engaged in Construction Inspection, Testing, or Special Inspection

Concrete - C 511 - Moist Cabinets, Moist Rooms, Water Storage Tanks
Concrete - C 617 - Capping Cylindrical Specimens
Concrete - C 1064 - Temperature of Concrete
Concrete - C 1077 - Concrete and Concrete Aggregate Testing Standards (Quality Standards)
Concrete - C 1231 - Unbonded Caps
Concrete - D 4832 - Preparation and Testing of Controlled Low Strength Material (CLSM) Test Cylinders
Concrete - D 5971 - Sampling Freshly Mixed Controlled Low-Strength Material

GEOTEXTILES

Geotextiles - D 374 - ASTM Geosynthetic Testing Standard
Geotextiles - D 413 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 751 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 792 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 882 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 1004 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 1204 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 1238 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 1505 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 1593 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 1603 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 1777 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 3015 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 3776 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 3786 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 3787 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 4218 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 4491 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 4533 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 4595 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 4632 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 4716 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 4751 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 4833 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 4884 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 4885 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 5035 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 5199 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 5261 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 5321 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 5596 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 5884 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 5887 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 5890 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 5891 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 5993 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 5994 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 6140 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 6214 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 6241 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 6243 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 6364 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 6392 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 6496 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 6636 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 6637 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 6693 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 6766 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 6768 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 7003 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 7004 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 7005 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 7179 - ASTM Geosynthetic Materials Testing Standard
Geotextiles - D 7737 - ASTM Geosynthetic Materials Testing Standard

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Appendix D GLNPO Requirements for Submission of Field Data

GREAT LAKES LEGACY ACT

DATA REPORTING STANDARD

REVISION 1

August 2023

Prepared for:

U.S. Environmental Protection Agency
Great Lakes National Program Office
77 West Jackson Boulevard
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Approved by:

_____	August 30, 2023
Mark Loomis, GLLA QA Manager	Date

Preface

The 2023 *Great Lakes Legacy Act Data Reporting Standard* (GLLA DRS) provides a single source for data reporting guidance for projects implemented under the Great Lakes Legacy Act (GLLA). Information provided in this document should serve as a basis for developing a Quality Assurance Project Plan (QAPP) and associated field and laboratory standard operating procedures (SOPs) to clearly identify and describe how the project will meet GLLA data reporting requirements and help ensure that all required data are correctly collected and reported throughout each phase of the GLLA project.

The 2023 version of the GLLA DRS supersedes the 2020 GLLA DRS and provides updated references and minor revisions to the 2020 GLLA DRS.A more substantial revision was completed in 2020 to update the 2010 GLLA DRS, with major changes in the 2020 GLLA DRS including:

- updates to locational, metadata and geospatial data reporting requirements;
- updates to instructions for reporting data using the Environmental Protection Agency (EPA) Region 5 Electronic Data Deliverable (EDD), the National Oceanic and Atmospheric Administration (NOAA) Data Integration, Visualization, Exploration, and Reporting (DIVER) tool, the Scribe software tool developed by the Environmental Response Team (ERT), the Staged Electronic Data Deliverable (SEDD) format, and simplified spreadsheet formats;
- clarification on differences in reporting requirements for routine analytical chemistry data, biology/toxicology data and data from experimental methods;
- instructions for submitting final project data sets via the GLLA Data Submission Site on GLNPO.net; and
- elimination of the requirement for hard-copy reports and documentation.

Since the publication of the 2010 GLLA DRS, the Region 5 EDD Specifications and SEDD Specifications have been comprehensively updated, and NOAA Query Manager has been replaced by NOAA DIVER. The 2023 GLLA DRS provides updated guidance (including links to source documents along with additional clarification and helpful hints for data providers) to ensure that GLLA data reporting requirements align with these updated reporting formats. Data providers should carefully review the 2023 GLLA DRS to: 1) ensure familiarity with any revisions made since the release of the 2020 GLLA DRS, and 2) ensure that updates or addendums to previously approved QAPPs and SOPs are compliant with the 2023 GLLA DRS.

All GLLA data providers, including prime site contractors, subcontractors, laboratories, states, non-federal sponsors, and others are expected to follow the GLLA DRS requirements. The 2023 GLLA DRS details required data components and accepted data reporting formats to facilitate comprehensive data verification, validation and usability reviews (identified by the GLLA Quality Plan (QP) as Data Quality Assessments (DQAs)), as well as the submission of complete project data sets for upload to the Great Lakes Sediment Database (GLSED). Any data-generating project that is wholly or partially funded under the GLLA should follow reporting requirements identified in the GLLA DRS, unless an exception is approved by the GLLA Quality Assurance (QA) Manager.

Requirements identified in the 2023 GLLA DRS should be implemented for all future GLLA projects and GLLA final data deliverable packages. Special consideration should be applied to the updated geospatial database requirements. Current and past GLLA projects with approved quality documentation are exempt from these new or updated requirements but should incorporate them to the extent practical. Any questions on the requirements of the 2023 GLLA DRS should be directed to the GLLA QA Manager, Mark Loomis (loomis.mark@epa.gov), 312-886-0406.

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1.0 Introduction

The U.S. Environmental Protection Agency (EPA) Great Lakes National Program Office (GLNPO) collects a variety of environmental data to support sediment assessment and remediation projects implemented under the Great Lakes Legacy Act (GLLA). Projects implemented under the GLLA involve a number of collaborators, which may include EPA Regions, states, non-federal sponsors, state contractors, EPA contractors, non-federal sponsor contractors, state laboratories, commercial laboratories, university laboratories, and others. To facilitate the accuracy, integrity, objectivity, consistency and completeness of these data sets, GLNPO has developed a data reporting standard for all projects administered under the GLLA. These requirements are consistent with common data reporting standards that have been developed by GLLA partner organizations and are familiar to GLLA project partners. The common data reporting formats accepted by GLNPO include: the EPA Region 5 Electronic Data Deliverable (EDD) format; the National Oceanic and Atmospheric Administration (NOAA) Data Integration, Visualization, Exploration, and Reporting (DIVER) templates; and the Staged Electronic Data Deliverable (SEDD), Version 5.2 format. When SEDD is used for laboratory data, data providers often provide accompanying field data in the Scribe format. Data may also be provided in a mini-GLSED format. Laboratories directly contracted with GLNPO for experimental or research methods may provide data in the simplified spreadsheet format. GLLA-specific requirements build upon the source data requirements. Data reporting standardization facilitates efficient upload to GLNPO's Great Lakes Sediment Database (GLSED), which is used to manage and publicly disseminate data collected under the GLLA through the Great Lakes Environmental Database (GLENDA).

This 2023 GLLA Data Reporting Standard (DRS) details data reporting requirements for both field and laboratory data, and addresses the most common types of environmental information generated under the GLLA. As directed by the *Quality Assurance Considerations for Great Lakes Legacy Act Projects* (August 2023), project-specific Quality Assurance Project Plans (QAPPs) should contain detailed information about the data reporting requirements for each specific project, as well as roles and responsibilities for providing project data to GLNPO. Each QAPP must be approved by the GLLA Project Manager and GLLA Quality Assurance (QA) Manager. GLLA projects often involve site-specific data collection requirements that cannot be fully captured by the standards described in this document. In these cases, to ensure GLSED compatibility, the data provider and the GLLA Project Manager should discuss these requirements with the GLLA QA Manager during project planning and QAPP development to ensure these reporting requirements are sufficiently addressed in the project-specific QAPP, and during data collection and reporting. Adhering to requirements in the GLLA DRS facilitates generation of consistent, usable, and reliable data to support GLLA sediment assessment and remediation projects and is mandatory for projects wholly or partially funded under the GLLA.

The original version of the GLLA DRS was published in 2010 and since then, EPA Region 5, NOAA, EPA Superfund, and other partner organizations have developed and implemented comprehensive updates to their data reporting requirements that serve as a basis for GLLA data reporting requirements. This 2023 GLLA DRS reflects the current requirements and as such, identifies options that can: 1) increase flexibility for data providers through the use of several common data reporting formats, and 2) streamline the data reporting process by allowing research laboratories to use simplified data reporting options when submitting directly to GLNPO. Additional modifications have been made to update source links and provide additional clarification and helpful hints to data providers.

2.0 Purpose

The 2023 GLLA DRS provides data reporting guidance for projects being implemented under the GLLA. Any project wholly or partially funded under the GLLA should follow the reporting guidance in the GLLA DRS, unless an exception is approved by the GLLA QA Manager. While GLNPO provides several options for reporting data, to maintain consistency, transparency, and reproducibility, laboratory and field

data submittals must include specific minimum components. In addition, data providers must submit additional supporting information to GLNPO (e.g., optional EDD fields, field reports, additional sample information, final project reports), when available.

For many projects implemented under the GLLA, a site contractor or collaborator is responsible for planning and overseeing field and laboratory data generation, and subsequently reporting the final verified and validated data to GLNPO. These data must be submitted in one of the accepted deliverable formats for upload to GLSED. GLNPO typically does not re-validate final project data when project documentation confirms that project-specific data quality objectives (DQOs) have been met. However, as the final owner of all data generated under GLLA, it is imperative that GLNPO maintain complete information about a data set to both ensure compliance with EPA Information Quality Guidelines and preserve their ability to fully investigate data integrity should future concerns arise. The GLLA QA Process diagrams are provided in Attachment 1. More information on the GLLA QA process and GLLA project-specific QA requirements for prime (i.e., site) contractors can be found in the *Quality Assurance Considerations for Great Lakes Legacy Act Projects* (August 2023), (i.e., QA Considerations) which is available from the GLLA QA Manager.

The remaining sections of this 2023 GLLA DRS are summarized below:

- Section 3: **Overview of GLLA Data Reporting Requirements.** This section provides a brief overview of the accepted reporting formats, GLLA-specific reporting requirements, the relevance and applicability of the Superfund Contract Laboratory Program (CLP) requirements, the use of the Scribe software tool for field data collection, and considerations related to data submission.
- Section 4: **Field and Laboratory Data Deliverable Requirements.** This section of the GLLA DRS details the required components of the data deliverable, including the EDD and supplemental field (Attachment 2) and laboratory information. Supplemental information (e.g., chains of custody, field logs, laboratory raw data, validation reports, final project reports) should be provided in a portable document format (PDF) or in the original electronic file format (e.g., Microsoft Word, Microsoft Excel). To facilitate efficient data storage and retrieval, GLNPO accepts data and reports in electronic format only (i.e., hard copy deliverables are not accepted).
- Section 5: **Accepted Formats for Electronic Data Deliverables.** This section provides information on accepted formats for the EDD submissions and defines minimum information that must be included for each accepted reporting format. Links to guidance documents, user guides, and references to helpful resources are included.
- Section 6: **Geospatial Database Deliverables.** This section provides a summary of the advantages and technical specifications for geospatial database deliverables.
- Section 7: **Data Deliverable Package Checklist.** This section provides a description and reference to a checklist (Attachment 3) that can be used by data providers when preparing their deliverable packages to help avoid common data quality issues.
- Section 8: **Contacts.** This section provides a list of individuals that data providers may contact if questions arise during sample analysis and/or report preparation.
- Section 9: **Resources.** This section provides a list of sources with additional information that may be helpful to data providers, laboratories and other stakeholders.

The following attachments to this GLLA DRS provide additional information to help ensure adherence to GLLA data reporting requirements:

- Attachment 1: **The GLLA QA Process.** These figures depict the GLLA QA and Data Management Process from project planning to final data reporting and identify major requirements and roles/responsibilities of the prime contractor, GLLA Project Manager and GLLA QA Manager.
- Attachment 2: **Minimum Requirements for the Submission of Field Data for GLLA Sediment Projects.** This attachment summarizes the required field data elements for information on the data provider contact, study area, station and samples.
- Attachment 3: **GLLA Final Data Deliverable Package Checklist.** This checklist can be used to help ensure deliverables submitted to the GLLA Data Submission Site on GLNPO.net are complete and speed the GLSED review and approval process.
- Attachment 4: **Locational Data Checklist and Metadata Recording Form.** Data providers should review this form prior to collecting data and upload the completed form to the GLLA Data Submission Site on GLNPO.net along with the GLLA final data deliverable package.
- Attachment 5: **Data Element Dictionary for the Simplified Spreadsheet EDD File.** This file provides a description of each data field in the simplified spreadsheet EDD, which is used by laboratories submitting data directly to GLNPO (if pre-approved by the GLLA QA Manager and documented in the project QAPP).

3.0 Overview of GLLA Data Reporting Requirements

Due to the variety of partners supporting GLLA sediment assessment and remediation projects, GLNPO allows flexibility in the submission of final project data sets. GLNPO encourages data providers to be efficient and practical in their data collection and evaluation efforts, and allows providers to submit data in formats they are typically already familiar with, including the Region 5 EDD format (typically used to report analytical chemistry data as described in Section 5.1) and the NOAA DIVER templates (typically used to report biological, toxicity and habitat data, but may also be used for analytical chemistry data as described in Section 5.2).

Data providers should collect, evaluate, and provide data in a format that meets project-specific DQOs and data management needs, and need not limit data reporting to a single format if the format does not meet project-specific requirements. For example, it is not uncommon for GLLA sediment and remediation projects to collect field data using Scribe and request that the laboratory submit a SEDD file. The data provider may then use EPA's Electronic Data Exchange & Evaluation System (EXES) to merge the field and laboratory data, conduct an automated data review, and complete data verification and validation using EXES Data Manager (EDM) (see Section 5.3). EXES can generate Region 5 EDDs. Supporting toxicity, bioaccumulation, and benthic or fish community data may be reported using NOAA DIVER Templates to support the development of clean-up goals and identify ecological restoration targets. Each of these deliverables are acceptable provided they meet the GLLA DRS minimum requirements for each reporting format, and all supporting information is included in the final data submission to GLNPO.

3.1 Comparison of GLNPO Accepted Reporting Formats

The data reporting format used by the project team is typically chosen based on the type of data generated (e.g., chemistry, biology) as well as laboratory capability. Table 1 provides a summary of accepted reporting formats and describes how each format may support GLLA sediment projects to assist data providers with identifying and selecting the appropriate format during project planning.

Table 1: Laboratory Reporting Format Options and Considerations

Laboratory EDD Format Option	Compatible Field Data Formats	Considerations
Region 5 EDD Format	Scribe, Region 5 EDD Field forms	• Most common format for submitting data to GLLA • Flexible in reporting a variety of site parameters, including geotechnical data and associated quality control (QC) criteria • Files are compatible with EQuIS • Does not allow reporting of benthic community or toxicity data
NOAA DIVER Lab EDD Template	NOAA DIVER Templates (ChemTox or BioLab)	• Useful for site assessments that incorporate toxicity, bioaccumulation, benthic/fish community, or ecological assessments, but may not provide sufficient information on geotechnical parameters to support comprehensive remedial investigations
SEDD	Scribe if using EXES for automated data review	• Uniform, non-proprietary format that can support instrument calibration information and link batch QC to associated samples, allowing automated data review up to Stage 3 • Recommended laboratory deliverable if using EXES and EDM or other automated review program to assist data validation, increasing consistency and saving resources • EXES automatically merges field and laboratory data (if Scribe XML file is provided) and outputs a GLNPO CLP Reporting Format and Universal Data Deliverable, which meets all GLLA DRS EDD requirements • EXES can be used to generate additional project-specific EDDs and preliminary data qualifiers and validation reports • Can be directly mapped to most data management systems, including EQuIS and Scribe • Does not currently support field information
Mini-GLSED Access Database	N/A	• Data providers may provide data in the mini-GLSED Access Database format (this is not typical or required, and does not speed data processing by GLNPO's Data Management Contractor) • Providing a mini-GLSED may be useful if the data are not already in another format accepted by GLNPO (e.g., in a propriety database format)
Simplified Spreadsheet for Laboratory Data	Simplified Spreadsheet for Field Data	• Simplified way for research laboratories directly contracted with GLNPO to provide environmental data when other data reporting options are not practical

Note: The NOAA Query Manager Template is no longer an option to submit data to GLNPO.

3.2 Project Planning to Meet GLLA Data Reporting Requirements

Project planning documentation, particularly the project QAPP, must describe all project-specific data reporting requirements, as well as the process and requirements for data verification and data validation. Project data sets must include certain sample and method quality control data, supporting information, and the results of data verification and validation (to ensure that both data quality and usability are fully evaluated and documented). Project data sets are uploaded to the GLSED only when the GLLA QA Manager has confirmed that minimum data and data quality requirements have been met.

To ensure the data produced are of known and documented quality, all data generated under the GLLA must undergo formal data verification and validation per the requirements in the project QAPP. Field and laboratory data must be correctly merged and verified, and data validation must be performed in accordance with EPA Information Quality Guidelines, Superfund CLP National Functional Guidelines for

Data Review (NFGs), and project-specific DQOs. Data verification and validation procedures, including acceptance criteria and laboratory- and validator-applied qualifiers, must be stated clearly in the QAPP and approved by the GLLA Project Manager and GLLA QA Manager prior to data collection. The results of data verification and validation are typically documented with a data review narrative, which should be provided to GLNPO with the verified and validated final project data set. Many projects document the overall results of data review and evaluate findings in terms of project DQOs. These findings are documented in a separate data usability technical memorandum or provided within a final project report. When available, technical memoranda, final project reports and any other information supporting data quality and usability should be provided to GLNPO when submitting the final project data set.

3.3 GLLA-Specific Data Reporting Requirements

In addition to all minimum data fields required by the Region 5 EDD, NOAA DIVER Templates, and SEDD, GLNPO requires data providers to report the following additional information for all GLLA final data deliverable packages, which may not be required by other EPA programs or NOAA. Section 4.1 provides information on how this information should be reported in the EDD for each data reporting format.

GLLA-Specific Data Reporting Requirements:

- **Contact Information**: Provide contact information for the data provider, including name, email and phone number should questions arise concerning the data.
- **Field Data**: Include sample collection time, sample delivery group, water depth with units, total sediment depth with units, method for measuring total sediment depth (e.g., graduated pole, vibracore), and whether sediment was collected to refusal (Section 4.1.4).
- **Laboratory Information:** Include lab name, sample delivery group, lab sample identification (ID), lab qualifiers, method detection limits, sample specific detection limits, all associated methods and sample QC results (as required by the QAPP), all analyte-specific results from dilutions, and re-extractions and re-analyses with a single reportable result clearly identified (Section 4.3).
- **Data Validation:** Include validator qualifiers, final interpreted data qualifiers, qualifier definitions, and information on summing schemes or correction factors, including how non-detects and data qualifiers were accounted for (e.g., for Total polycyclic aromatic hydrocarbons (PAHs), Total polychlorinated biphenyls (PCBs)).
- **Spatial Data:** Submit in the ESRI File Geodatabase format with a defined coordinate system. If applicable, submissions should include the collection method(s). Each geodatabase should be accompanied by Federal Geographic Data Committee (FGDC) compliant metadata as described in Section 4.2.

Attachment 2 provides additional information on minimum data required for final submission to GLNPO for field data.

3.4 The Superfund Contract Laboratory Program

While not affiliated with the Superfund program, GLNPO closely follows Superfund data quality requirements and data review procedures, including the Superfund CLP requirements and procedures, when practical. The CLP is a national program managed by the U.S. EPA Analytical Services Branch (ASB). CLP Statements of Work (SOWs) contain the technical, contractual, and reporting requirements that must be met by the CLP laboratories. More information on this program is available at https://www.epa.gov/clp.

Use of CLP processes and procedures provide benefits to GLLA projects, including implementation of standardized EPA methods and procedures, consistent method and sample QC requirements, automated data review using EXES, standardized data reporting, and consistent method QA/QC and documentation requirements. Laboratories in the CLP are typically qualified to meet the rigorous data quality and data reporting requirements of the GLLA. However, final decisions on laboratory capability, analytical method and data quality requirements, and project DQOs are determined by the GLLA project team, in consultation with the GLLA QA Manager, to ensure that project-specific DQOs are met.

3.5 The GLNPO CLP Reporting Format and Universal CLP Deliverable

GLNPO has worked with the Superfund CLP Program to develop a customized reporting format for data reported to GLNPO following EXES automated data review, which is known as the "GLNPO CLP Reporting Format." The CLP also provides a "Universal Data Deliverable" for all projects that use EXES automated data review. The GLNPO CLP Reporting Format and CLP Universal Data Deliverable include all of the laboratory analytical information required for upload to the GLSED. Scribe Extensible Markup Language (XML) field data exports can be automatically mapped to the GLNPO CLP Reporting Format and CLP Universal Data Deliverable using EXES. The collaborator may then use EDM to access data in a central location, allowing multiple data reviewers to complete data validation simultaneously. When using EXES automated data review, the original laboratory SEDD file does not need to be provided to GLNPO. Only the validated GLNPO CLP or CLP Universal Data Deliverable with complete field information needs to be provided. The supplemental information should include a PDF of the laboratory raw data package and all EXES-generated and validator-edited reports along with associated data review narratives.

Please note that the Universal Data Deliverable and GLNPO CLP Reporting Format are only accepted as EDDs if EXES is used. These files are specific outputs of EXES and are not accepted by GLNPO as stand-alone templates.

3.6 Scribe for Field Data Collection

Scribe, a software tool developed by EPA's ERT, captures sampling, observational and monitoring field data. Examples of Scribe field tasks include soil sampling, water sampling, air sampling and biota sampling. While Scribe can also import and store analytical laboratory results, GLNPO does not utilize this capability.

Scribe outputs include labels for collected samples and chain-of-custody (COC) generation, which help ensure samples are sent to the correct laboratory and analyzed for the correct parameters. Scribe labels are often used in addition to laboratory provided labels. Scribe supports electronic field data exports in the XML format, which can be automatically combined with the laboratory EDD when using automated data review software, such as EXES, or other mapping software. Field data for GLLA projects can be delivered in the Scribe database format regardless of whether laboratories are delivering analytical data in a format that can be mapped to the field data or if EXES is used. Additional information on the Scribe format for field data can be found at http://www.ertsupport.org/scribe_home.htm.

The Scribe Quick Start Guide provides information for creating new projects, adding samples, and creating labels and chains of custody. The Scribe User Manual provides additional information on where to store data and Geographic Information System (GIS) information in Scribe. Data providers may submit the study-specific Scribe database to GLNPO in lieu of providing a supplemental field data file.

***Helpful Hint**: GLNPO encourages the use of Scribe and EXES automated data review to increase consistency and save costs by streamlining field data collection as well as data verification and validation processes. Use of these free EPA software tools requires up front planning, system access, and laboratory EDD testing. As such, data providers should alert the GLLA QA Manager and CLP program during QAPP development to receive assistance and training on these systems, which can help reduce potential delays.*

3.7 Submission of Final Project Data sets via the GLLA Data Submission Site

GLNPO collaborators should submit final project data, including final EDDs and complete field and laboratory supporting information, to GLNPO (via the GLLA Data Submission Site on GLNPO.net). Data providers requiring access to the GLLA Data Submission Site or training on the data submission process should contact the GLLA QA Manager.

A GLLA Final Data Deliverable Package Checklist is included as Attachment 3.

GLSED final Data Submissions must be uploaded to the GLLA Data Submission Site on GLNPO.net and include the following items:

- Final verified and validated EDD(s) with merged field and laboratory data in one of the accepted formats identified in Section 5
- The complete laboratory raw data package identified in Section 4.3
- Data verification and validation narratives
- Definitions of any validator and laboratory qualifiers applied to the data
- Final QAPPs, Field Sampling Plan (FSP) and Addendums

When available, the data provider should also submit the following items:

- Data Quality/Usability Assessments
- Technical Memoranda
- Final Project Reports
- Any additional information supporting project data quality or usability

Upon receipt of a GLLA final data deliverable package, GLNPO's Data Management contractor pre-loads project data into a project-specific GLSED (i.e., mini-GLSED). Data submissions are first assessed for adherence to basic completeness and compliance requirements during the Data Submission Compliance Overview (DSCO) review. A completed DSCO form identifying data gaps and issues is forwarded to the data provider. Upon receipt of DSCO form responses, the data then undergo various checks, including data consistency checks, a Quality Information Completeness (QIC) review, and a mini-GLSED review. Data quality issues requiring further clarification or confirmation are forwarded to the data provider. GLNPO requires that all data providers comply with these requests for additional information.

Once project-specific mini-GLSEDs are developed and thoroughly reviewed, they are submitted to the GLLA QA Manager for approval prior to final upload to GLSED. GLNPO does not allow project close-out until final project data sets are approved by the GLLA QA Manager. Data providers should compare these project-specific databases against their project data sets to ensure accuracy and completeness prior to project close-out. This final step helps ensure that quality data used to support GLLA decisions are available to the public and in compliance with EPA Information Quality Guidelines.

4.0 Field and Laboratory Data Deliverable Requirements

This section details the minimum components that must be included in the field and laboratory data deliverables. GLNPO requires two distinct data deliverable components. The first is the EDD, which must be provided in one of the accepted formats detailed in Section 5. The second is the supplemental information, to include final project QA documentation, field logs, laboratory raw data, data validation reports, final project reports, and/or other supplemental information necessary to confirm the quality and usability of the data and whether the data meet project data quality requirements. As the final owner of the data, GLNPO must retain this information to 1) comply with EPA Information Quality Guidelines, and 2) maintain the ability to fully investigate data sets in the event that data integrity questions arise after final project decisions have been made.

4.1 Field Data Deliverable Components

Accurate field information and locational data are critical to mapping, modeling and interpreting environmental data. For all GLLA projects, collaborators are required to complete and submit the *Locational Data Checklist and Metadata Recording Form* for each sampling event (Attachment 4), which can be submitted as a fillable/electronically-signed form (preferred) or as a scanned version of the completed form.

To facilitate the use of existing field forms used by GLNPO data providers, a list of data field reporting requirements, *Minimum Requirements for the Submission of Field Data for GLLA Sediment Projects* is included as Attachment 2. When reporting data in the allowable report formats discussed in Section 5, all required elements must be reported according to the specific EDD reporting specifications along with the additional components required by GLLA. Any additional field information requirements based on project objectives must be detailed in the project QAPP. The data provider should discuss any special field data reporting requirements with the GLLA Project Manager and document the final decisions in the project QAPP.

4.1.1 Data Provider Contact Information

The data provider is the person responsible for submitting the EDD and will serve as the contact person to answer any questions that may arise regarding the field and laboratory data provided in the EDD. Required data provider information includes their affiliation, first and last name, physical address information (street address or post office box number, city, state, and zone improvement plan [ZIP] code), and data provider e-mail address and phone number.

4.1.2 Study Information

Study information consists of data that describe a study. These include geographical data, such as: site or location name, site or location description, site or location purpose, EPA region, state, district, county, watershed, and reach, when available.

4.1.3 Station Information

Station information consists of data that describe a particular sampling station. These data include the station identifier; latitude and longitude of sampling stations in World Geodetic System (WGS) 84 or North America Datum (NAD) 83; and a description of the station. These data are critical for projects using geostatistics to map the extent of site contamination. GLLA requirements for locational data include:

- **Horizontal data information:** x-coordinate, y-coordinate, horizontal datum, horizontal collection method, horizontal accuracy value, and horizontal units of measure

- **Vertical data information:** water surface elevation, sediment surface elevation, bedrock elevation, elevation collection method, elevation accuracy value, and vertical datum code
- **Reference point(s):** a structure or recognizable feature within or near the study area that is viewable using aerial photography. See Attachment 4.
- **Control point(s):** a previously surveyed location with known coordinates (e.g., a benchmark). See Attachment 4.

Alternatively, if the vertical data information as listed above is not available, data providers should provide the water and sediment depth measurements and the method for measuring sediment depth.

4.1.4 Field Sample Information

Field sample information consists of data associated with specific environmental samples. These data include the unique sample identifier, sample type (e.g., routine field sample, field duplicate sample, and equipment blank), collection date and time, upper and lower sample collection elevation or depth (z-coordinate), and elevation/depth units. Additional information required by GLNPO for sediment samples includes water depth with units, sediment sampling technique, whether the sample is a composite or a grab sample, a list of sediment sample observations (e.g., color, texture, odor), and whether the sample was collected to refusal.

When using the Region 5 EDD Format, report required field sample station information in the Field EDD location file (EPAR5LOC_v3) as follows:

- Report water depth with the applicable unit of measure in the "remark" data field
- Report sediment depth in the "depth_to_bedrock" data field
- Report observation time along with the date in the "observation_date" data field
- Report the method for collecting sediment depth (graduated pole, vibracore, etc.) and whether the sample was collected to refusal in the "loc_purpose" data field

Report the following sample-specific information in the Lab EDD sample file (EPAR5SMP_v3):

- Report sediment sample observations (e.g., color, texture, odor) in the "comment" data field
- Identify whether the sample was a composite or grab sample in the "composite_yn" data field

More information on the Region 5 EDD format reporting requirements is provided in Section 5.1.

When using the NOAA DIVER ChemTox and BioLab Templates, report this information in the "Notes" data field of the SampleMaster worksheet. More information on this reporting format is provided in Section 5.2.

If more convenient for the data provider, this additional information may be submitted in a supplemental spreadsheet file regardless of EDD format. The only requirement is to use sample and station IDs in the spreadsheet file that are consistent with those used in the main EDD.

4.2 GIS Data Collection and Reporting Requirements

The data provider should follow appropriate guidelines prior to, during and after field data collection for the purpose of maximizing the accuracy of data collected using a global positioning system (GPS). Failure to follow these procedures may result in spatial measurements with reduced accuracy and a failure to meet project objectives. GLNPO recommends that project participants collecting and processing spatial data undergo certified GPS training and spend time familiarizing themselves with each specific GPS unit being used for each project prior to the start of field activities.

To document and facilitate the collection and reporting of locational data, collaborators are required to complete and submit the *Locational Data Checklist and Metadata Recording Form* (Attachment 4). Data providers must review this form prior to writing the project QAPP and complete and submit the form subsequent to field data collection. For a more complete description of GPS data collection policy and guidance, please refer to Section 1 (Standard Operating Procedure for Global Positioning System Data Collection) of EPA's Interim Guidance for Developing Global Positioning System Data Collection Standard Operating Procedures and Quality Assurance Project Plans, dated February 2008.

Technical Specifications for all GLLA GIS Data Deliverables:

1. All horizontal locational data not in a GIS file format (e.g., CSV, Microsoft Excel) must be submitted in decimal degrees in the WGS 84 or NAD 83, specifying which is used. Inclusion of projected coordinates (e.g., Universal Transverse Mercator (UTM)) is optional, but complete metadata regarding projection parameters and datum must accompany projected coordinates.
2. All GIS files (e.g., raster, vector) should be formatted into a standard ESRI format, with the file geodatabase preferred, and must have spatial reference information that describes the projection and datum.
3. All GIS files are required to have associated FGDC metadata. The Content Standard for Digital Geospatial Metadata can be found at http://www.fgdc.gov/metadata/geospatial-metadata-standards#csdgm. An add-on editor for ESRI software, EPA Metadata Editor (EME), complies with the FGDC metadata requirement and is available at https://edg.epa.gov/EME/. Versions are available for both ArcMap and ArcGIS Pro.
4. All map documents should be submitted in the ESRI format (.mxd or .mapx), with all supporting map data submitted as described above, as a map package (.mpk or .mpkx), or as a project package (.ppkx), with the supporting map data included as part of the package. Before saving or exporting, ensure the resultant file includes a relevant title and brief summary, and store relative path names to data sources.
5. Any GIS files and map documents that cannot be formatted into a standard ESRI format must be included in their native editable file format. This includes computer-aided design (CAD) files or any other files that are part of the data submission and support map documents.

4.3 Laboratory Analytical Data Deliverable Components

GLLA data providers are required to submit two laboratory data deliverables to GLNPO: 1) electronic data in an allowed EDD format (Section 5), and 2) a supplemental data package containing laboratory raw data and supporting information. The supplemental laboratory data package facilitates the DSCO and QIC review process and upload to GLSED. GLNPO requires that the laboratory raw data package and supporting information be provided as PDF files or as original file formats (e.g., Microsoft Word) and uploaded with the full data submission to the GLLA Data Submission Site.

The required minimum components of a laboratory EDD and supplemental data package are detailed in this section. In most GLLA projects, the collaborator or site contractor will contract directly with an analytical laboratory. The analytical laboratory should be identified during the project planning and QAPP development process so that the laboratory may provide important feedback on their capability to meet method acceptance criteria and data reporting requirements as documented in the QAPP. The laboratory statement of work should dictate specific GLLA data reporting requirements for the analytical laboratory. When using the Region 5 EDD format, NOAA DIVER, or SEDD, the data provider should carefully review the source documents to ensure all required information is included and the correct valid values are used. The QA Considerations document contains additional information regarding laboratory competency, certification requirements, preferred analytical methods, Performance Evaluation (PE) sample use, and data verification and validation requirements that must be considered when identifying an

analytical laboratory to meet project DQOs. In rare instances when GLNPO contracts directly with a laboratory (usually an academic laboratory for research purposes), the laboratory has the option of submitting data in the simplified spreadsheet format.

> ***Helpful Hint:*** *The analytical laboratory is an important member of the project team and should provide feedback on the project-specific DQOs and how they can be achieved using current analytical technology, prior to QAPP submission.*

While GLNPO does not perform a Stage 4/Tier 2 (described in the GLLA QP) data validation on all laboratory data, as the final data owner, GLNPO requires all laboratories submitting routine analytical chemistry data to submit a complete data package (commonly called a "Level IV" package), which allows an independent reviewer to complete a Stage 4/Tier 2 data validation if future concerns about the data arise. To conduct a comprehensive Stage 4/Tier 2 data validation, independent data reviewers must comprehensively evaluate the following six data deliverable components: a narrative summary, COC forms (or traffic reports), calibration information, raw data, electronic results, and other supporting information. These items are described in detail below. At a minimum, GLNPO requires field data, analytical results, and sample QA/QC data delivered in a machine-readable format (e.g., CSV, XML, Microsoft Excel, Microsoft Access), in one of the accepted EDD formats, to facilitate upload of the data submission to GLSED. Other supporting information and raw data should be provided as an electronic PDF or in the original file format (e.g., Microsoft Word) unless otherwise agreed upon in advance by the GLLA Project Manager and GLLA QA Manager. Scanned photocopies of original sample documentation, such as air bills and COCs should be provided.

For routine organic, inorganic, and high-resolution chemistry parameters in water, sediment, soil and tissue (i.e., CLP or relevant SW-846 methods), GLNPO evaluates data against CLP NFGs, with modifications specific to sediment as described in the GLLA QP. For these types of data, laboratories must provide sufficient information to allow an independent data reviewer to reproduce results and calculations from the raw laboratory data (i.e., a Level IV deliverable like a CLP data package). This information is typically provided in a machine-readable EDD, which contains sample and methods QA/QC information, and a supplemental PDF laboratory data package that contains calibration information and raw data. The SEDD reporting format allows laboratories to report additional information in the EDD, such as calibration information, which is evaluated by an automated data review program, and can speed and streamline the data validation and verification process.

For analyses where NFGs are not available or supplemental GLLA data validation guidance does not exist (e.g., bioassays, habitat evaluations, toxicity tests), the QAPP must identify clear QA/QC acceptance criteria to ensure the data are of sufficient quality to support project DQOs. The QAPP should detail the reporting requirements and supporting information required from the laboratory necessary to complete appropriate data verification and validation to support the intended decision. The GLLA QP provides additional information on the verification and validation of this type of non-routine data.

4.3.1 Narrative Summary

Each laboratory report must contain a narrative summary detailing the samples that were analyzed, the specific methods and procedures followed, any problems or issues encountered with the analysis, and corrective actions taken.

At minimum, the laboratory narrative should include the following components:

- Contact information for the laboratory, including the individual responsible for the data submission

- Reference to relevant test methods and the QAPP
- Copies of (or references to) any communications from the GLLA Project Manager or data provider that: 1) provide specific directions that necessitate a deviation from the QAPP in the sample analysis, or 2) address specific questions posed by the laboratory
- Discussion of problems upon sample delivery, such as broken containers or mislabeling
- Sample issues, such as biphasic nature or insufficient volume for testing
- Testing issues, such as quality control failures or unusual observations during the testing process (e.g., extract solidified on concentration)
- A discussion of how issues were resolved, including any corrective actions taken
- A statement from the Laboratory Director or Laboratory Project Manager attesting to the results and their completeness and providing contact information should questions arise.

4.3.2 Chain-of-custody Forms or Traffic Reports

Copies of sample COC forms or traffic reports and any other paperwork (e.g., air bills, field reports) arriving with the samples should be included in the report. Any sample package receipt logs generated at the laboratory should be included. Internal COC (within the laboratory) is not generally required.

4.3.3 Calibration and Raw Data

For routine analyses, data reviewers must be able to track sample results throughout the analytical process to determine whether the analysis was performed correctly and recalculate reported results (i.e., a Stage 4/Tier 2 review). All data pertinent to reconstructing the sample result must be included in the data package unless otherwise approved by the GLLA Project Manager and GLLA QA Manager and documented in the QAPP. These data include, but are not limited to, calibration information, extraction logs, instrument run logs and chromatograms. PDF files containing this information that are generated directly from the instrument are preferred since it allows for clear interpretation of the chromatograms. Photocopies must be clearly legible and of appropriate resolution to allow the data reviewer to complete the evaluation. Note that if the laboratory is submitting an SEDD file for automated data review using EXES, an SEDD Stage 3 is preferred when possible.

4.3.4 Laboratory EDD

EDDs must be delivered in a machine-readable file format to facilitate data validation and GLSED upload processes. Please note that a PDF file is not considered a machine-readable format. Typically, EDDs are provided as Microsoft Excel, Microsoft Access, XML or CSV files. EQuIS-compatible formats are accepted for validation purposes, but other proprietary formats are not allowed. GLNPO may require data providers to utilize a certain file format, detailed in the project QAPP, to meet project DQOs or to share data with other collaborators.

Files that include laboratory results must include all associated method and sample QC results as required by the QAPP, such as matrix spike or matrix spike duplicate percent recoveries, surrogate recoveries, and laboratory duplicate results. The laboratory should report the results of all associated dilutions, re-extractions, and re-analyses in the EDD, and must clearly identify the reportable result for each specific analyte.

> ***Helpful Hint:*** *Data providers should specify which results should be considered reportable when results for two or more analyses of the same sample are reported in the EDD. When samples are analyzed more than once for a given parameter (dilutions, re-extractions, re-analyses, or cross-over between methods), the laboratory must choose the most appropriate result for reporting purposes. The reportable result is analyte-specific based on the best professional judgement of the laboratory. Reportable results for multiple analytes in a single sample are often reported from multiple runs and not a single analysis. Laboratories that do not select the appropriate reportable result (e.g., only reporting results from the undiluted run when many analytes are outside calibration range) may be asked to revise and resubmit the data at no cost to GLNPO.*

Laboratories are expected to carefully evaluate the method and sample QC results for each analyte and use best professional judgement to determine the reportable result for each analyte in a given sample. Laboratories that do not identify the reportable result may be asked to revise and resubmit the data with the appropriate reportable result identified at no cost to GLNPO. Data validators may identify a different reportable result based on data validation and should provide justification in the data review narrative. The laboratory must report all diluted and undiluted results in the EDD. However, the laboratory **is not** required to run a sample undiluted if high concentrations could cause damage to lab instrumentation. In these cases, laboratories should provide documentation in the laboratory narrative and supplemental data package to explain why the undiluted sample could not be analyzed (e.g., screening chromatograms, pictures, sample descriptions). Calibration data are generally not reported in the EDD unless the laboratory is submitting an SEDD Stage 3 EDD for automated data review.

Many GLLA studies include the analysis of investigation-derived waste (IDW) and confined disposal facility (CDF) samples to characterize these wastes/sediments for disposal purposes (e.g., SW-846 Method 1311: Test Method Toxicity Characteristic Leaching Procedure (TCLP), ignitability). These data are not required to undergo a Stage 4/Tier 2 data validation process because waste disposal typically precedes completion of the data review process. However, the resulting data must be compliant with relevant regulations, such as the Toxic Substance Control Act (TSCA) or the Resource Conservation and Recovery Act (RCRA). Waste characterization tests should be performed on IDW and CDF samples for disposal purposes only and are generally not valid site characterization methods. While the GLLA does not require retention of data for these samples in the master GLSED, the data may be stored within study-specific mini-GLSEDs. When data are submitted electronically for these samples, the data provider must clearly identify which information in the data package corresponds to these samples in the EDD (e.g., by identifying them within the comment data fields in the sample and result level EDD submissions).

Information on the acceptable format for laboratory and field EDD submissions is provided in Section 5.

4.3.5 Additional Supporting Information for the Laboratory Supplemental Data Package

Laboratory data submittals should include any supporting information that may assist data validators or data users in evaluating data quality and usability. Project-specific requirements for reporting supporting information should be detailed in the project QAPP, including laboratory qualifier definitions, schemes for handling non-detect data and qualifiers when summing analytes (e.g., when summing individual PCB Aroclor concentrations to calculate total PCB concentrations), and any correction factors applied. This information is required for GLSED to facilitate reproducibility and use by secondary data users. Requirements for this type of supporting information should be detailed the project QAPP and delivered along with the complete data submission to the GLLA Data Submission Site on GLNPO.net.

Supporting information provided by the laboratory for GLNPO projects must include the following components (as applicable):

- Definitions of any laboratory flags applied to the data
- The number of significant figures used in data reporting
- The scheme or approach used to determine: 1) total concentrations for specific parameters including (but not limited to) PCB Aroclors, PAHs, and PCB congeners (detail the analytes included in each total, how non-detects and qualifiers were handled, if any adjustment factors were applied, etc.); and/or 2) other relevant calculations such as the mean Probable Effect Concentration quotient (PEC-Q)
- Detailed information on all null codes used in the EDD (including definitions and usage)

A GLLA Data Deliverable Package checklist is included as Attachment 3 and should be provided to the laboratories during the project planning/QAPP development phase of the project to ensure the laboratory is able to provide this information.

4.4 Format of the Laboratory Supplementary Data Package

Laboratory supplemental data packages should be submitted as PDF files. Original electronic formats (i.e., Microsoft Word) are also acceptable. Paper copies (i.e., hard copies) are not accepted by GLNPO. Laboratory data package formats can vary depending on laboratory procedures. However, for routine analytical chemistry parameters (i.e., CLP or SW-846 methods), the laboratory data package must contain all information necessary to reconstruct the sample result from the raw data (i.e., perform a Stage 4/Tier 2 data validation on a Level IV data package). For analytical analysis where a Level IV laboratory data package is not practical or possible (i.e., bioassays, toxicity tests or waste disposal samples), the QAPP should clearly describe the supporting information required from the laboratory to ensure that the data meet project data quality requirements.

Two common formats for laboratory data packages are: 1) the chronological format, and 2) the standardized format in the style of the CLP. Other formats are acceptable if approved by the GLLA Project Manager and the GLLA QA Manager as documented in the project QAPP. These two formats are described further below.

4.4.1 Chronological Format

A common option for reporting laboratory data packages is a chronologically assembled data package. In this case, the results of each analysis are followed by the supporting information (Section 4.3) in chronological order. For instance, in the case of semi-volatile analysis of sediment samples, sample results would follow the narrative report, the results would be followed by extraction logs, calibration data and sample analysis data (according to the date performed). In this case, instrument logbook pages for the date of the data included should precede that data. Any information recorded in the logbook that is not relevant to the project samples (i.e., sample results from another project) should be crossed out.

4.4.2 Standardized Form in the Style of the CLP

The CLP SOWs provide specific guidance on data package preparation for laboratories working under a CLP contract to the EPA Superfund Program and requires that laboratories submit a PDF and hard copy of the data package to EPA if required by the EPA Region. GLNPO does not accept hard copy data packages but otherwise accepts the CLP format. GLNPO does not require the submission of CLP or CLP-equivalent forms provided the information is submitted in the associated EDD and the supporting raw data are clearly identified.

The CLP SOW details the correct assembly of data in CLP format and can be found at https://www.epa.gov/clp/superfund-clp-analytical-services.

5.0 Accepted Formats for EDDs

GLNPO allows several options for the submission of the EDD for field and laboratory data. Field data must be verified and correctly merged with associated laboratory data. Complete data sets submissions are accepted in the following formats: 1) the EPA Region 5 Comprehensive EDD format; 2) NOAA DIVER templates (Microsoft Excel or Access versions); 3) a GLNPO CLP Reporting Format/Universal Data Deliverable (when using EXES for electronic data validation); 4) SEDD Version 5.2 file; or a 5) mini-GLSED EDD (Microsoft Access Database). Because SEDD does not currently allow the reporting of field information, if SEDD is used, supplementary field data should be provided via a Scribe XML file or an alternative acceptable reporting format. A Microsoft Excel flat file is acceptable from laboratories directly contracted by GLNPO to perform experimental methods or research. If an alternate EDD format is proposed or anticipated, contact the GLLA QA Manager, Mark Loomis, for approval prior to submission.

Regardless of which format is used, the minimum field requirements detailed in this section and in Attachment 2 must be reported in addition to any project-specific data requested by the GLLA Project Manager. Required EDD data fields should be populated according to the associated source data standard instructions. GLNPO encourages data providers to submit all available field and laboratory information (rather than only the minimum requirements) since additional field and laboratory data reported for a specific project improves data usability in the long-term. Additional information about each allowable report format is provided in the following sections.

The EPA Region 5 EDD is the most common format for submitting routine laboratory analytical chemistry data to GLNPO. Toxicity, bioaccumulation, and benthic/fish community data should be submitted according to the associated NOAA DIVER Templates (ChemTox and/or BioLab) unless otherwise agreed upon with the GLLA Project Manager and GLLA QA Manager. NOAA's Query Manager template has been discontinued and is no longer an acceptable format for GLLA final data deliverable packages. Toxicity and bioaccumulation data should be submitted using the most current ChemTox Template and benthic or fish community data should be submitted using the BioLab Template (Section 5.2). Sediment chemistry results analyzed in conjunction with toxicity data may be reported using the NOAA DIVER Lab EDD Template, which can be appended to the ChemTox template, but are also accepted in the other formats described in this section.

5.1 The EPA Region 5 EDD Format

The EPA Region 5 Superfund Program publishes comprehensive EDD specifications for providing environmental data to the EPA Region 5 Superfund Division. This standard includes reporting requirements for sampling location and mapping information, as well as analytical chemistry results. EPA Region 5 provides detailed instructions on how to report data in the *EPA Region 5 Electronic Data Deliverable Comprehensive Specification Manual, Version 4.3, February 2023*, which can be found at: https://www.epa.gov/superfund/region-5-superfund-electronic-data-submission-documents. The EDD Comprehensive Specification Manual was extensively updated in 2020, and requirements should be reviewed thoroughly by data providers prior to QAPP submission and data collection.

5.1.1 GLNPO Required Files for Region 5 EDD

The *EPA Region 5 EDD Comprehensive Specification Manual* describes the requirements for reporting Superfund environmental data to EPA Region 5, thus not all reporting requirements are applicable to GLNPO. While the Region 5 EDD provides many options for submitting various types of data, GLLA projects normally generate data applicable to just six of the files including: data provider

(EPAR5DATAPROVIDER_v3), sub-facility **(EPAR5SUBFACILITY_v3)**, location **(EPA5LOC_v3)**, chemistry sample **(EPAR5SMP_v3)**, chemistry test result with QC **(EPAR5TRSQC_v3)**, and batch **(EPAR5BAT_v3).** However, a project may generate data that are applicable to more than these standard six files. The data provider should discuss special data reporting requirements and the specific files to be reported with the GLLA Project Manager and document the final decisions in the project QAPP.

Region 5 Files used for GLLA Projects

Field: The field EDD files contain general information about the site, data provider contact information for questions about the EDD, and information regarding the sample locations.

Required Field EDD Files:

- Data Provider (named as "EPAR5DATAPROVIDER_v3) provides sampler contact information for information in the EDD.
- Sub-facility (named as "EPAR5SUBFACILITY_v3) provides general information about the site.
- Location (named as "EPAR5LOC_v3) provides information regarding the sampling locations.

Lab: The Lab EDD files contain data related to chemistry field measurements, sample collection information, and sample results, QC and batch information.

Required Lab EDD Files:

- Sample (**EPAR5SMP_v3**) contains information about sample collection (including some field sample details).
- Test Result QC (**EPAR5TRSQC_v3**) contains the information about the analytical test performed on samples with quality control data elements. ***Note: GLNPO Data Submitters must use this file instead of the Test Result file (EPAR5TRS_v3) to allow for the reporting of spike and surrogate %R information.***
- Batch (**EPAR5BAT_v3**) contains the data that relates individual samples to the batch identifier.

> ***Helpful Hint:*** *Data providers should submit all Sample and associated Test Result QC and Batch files, and ensure that sample IDs, results, QC, and batch information are clearly linked to allow the data reviewer to complete the DSCO and QIC reviews.*

The process for creating Field and Lab EDDs, including required fields and valid values, are thoroughly described in the Region 5 EDD Comprehensive Manual. Laboratories typically produce the Lab files while site contractors/data providers produce the required Field files. When Scribe is used to collect field information, the Scribe XML exports should be provided to facilitate the DSCO review.

5.1.2 GLNPO Required Fields and Valid Values for the Region 5 EDD

Unless otherwise specified in the GLLA DRS, data providers should follow the detailed instructions on how to report data in the *EPA Region 5 Electronic Data Deliverable Comprehensive Specification Manual, Version 4.3, February 2023,* (or most current version) available at: https://www.epa.gov/superfund/region-5-superfund-electronic-data-submission-documents

All Region 5 EDD project files contain required data fields as well as data fields that are applicable when the information is available. Some data fields required by EPA Region 5 are pertinent only to Superfund, and thus not applicable to GLLA final data deliverable packages. GLNPO requires that GLLA project data submissions contain the data fields required by EPA Region 5 (as applicable) as well as specific information as described below (see Exhibit 1). Data providers should submit all available information,

including any project-specific data requirements in the final verified and validated EDD. The minimum additional components necessary for GLLA Data Submission in the Field and Laboratory files are detailed below and in Exhibit 1.

Required Field Files and GLLA Required Data Fields (in addition to R5 requirements):

- Data provider **(EPAR5DataProvider_v3):** data_provider, data_provider_contact_person, data_contact_email, and data_contact_phone
- Subfacility **(EPAR5SUBFACILITY_v3):** subfacility_code (AOC name), subfacility name (use the site name from the QAPP)
- Location **(EPA5LOC_v3):** loc_purpose (state whether collected to refusal and method used), loc_county_code, loc_state_code, remark (water depth), depth_to_bedrock (sediment depth).

Required Lab Files and GLLA Required Data Fields (in addition to R5 requirements):

- Chemistry sample **(EPAR5SMP_v3):** sample_delivery_group, sample_date, sys_loc_code, start_depth, end_depth, depth_unit, sampling_company_code, composite_yn, comment (report sample observations: e.g., color, texture, odor)
- Chemistry test result with QC **(EPAR5TRSQC_v3):** lab_name_code, lab_sample_id, lab_qualifiers, validator_qualifiers, interpreted_qualifiers, method_detection_limit, reporting_detection_limit (regardless of whether or not the result value is non-detect), quantitation_limit, qc_spike_recovery (applicable for all spiked analytes including labeled compounds and surrogates), and qc_dup_spike_recovery (applicable for all spiked analytes including labeled compounds and surrogates).
- Batch (**EPAR5BAT_v3):** Same as R5 requirements

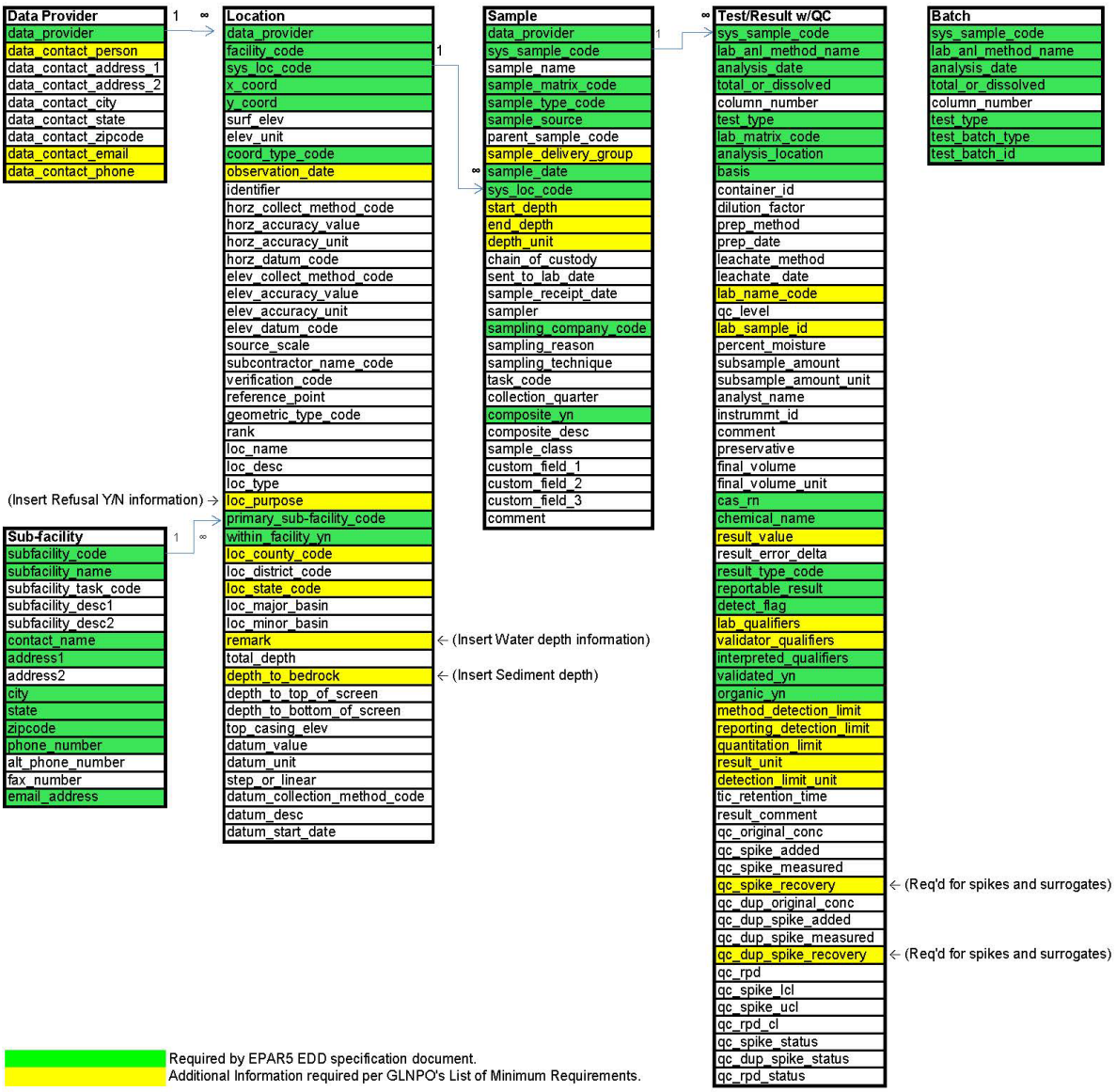


Exhibit 1: EPA Region 5 EDD Templates for Field and Lab Data, and Data Fields Required to Meet GLLA Minimum Data Requirements

> ***Helpful Hint:*** *Please include time information in data fields that include date information (e.g., sample_date, analysis_date, prep_date). Analysis date and time information are critical to link batch information to the corresponding results in the EPA Region 5 EDD structure.*

The Region 5 EDD Valid Values Reference Tables for EDDs (October 2020) lists allowable values for all Region 5 EDD data fields, and should be followed for GLLA final data deliverable package. GLNPO does not require that Region 5 EDDs prepared for GLNPO be checked by the EQuIS Data Processor (EDP) as specified in the Region 5 EDD Comprehensive Specification Manual. The EDP was developed for EDDs being prepared for the Region 5 Superfund Program and requires registration with that

program. However, if the data provider has access to the EDP via their work with the Superfund program, use of the tool to check the EDD prior to submission to GLNPO is highly encouraged.

Please note that GLNPO requires laboratories delivering electronic data in the Region 5 format to submit PDF files of raw data packages and supplemental information (Section 4.3). Laboratories are urged to make certain that data files contain header rows to avoid confusion.

> ***Helpful Hint:*** *Definitions of reporting limits are included in GLSED for each sample result in the form of reporting limits codes. The required reporting detection limit data field must be completed in accordance with the Region 5 EDD Comprehensive Manual. If applicable, any alternative reporting limits must be provided along with a detailed description of how that limit was generated. Compliance with the EDD standard will provide the information needed to complete the reporting limit codes.*

5.2 NOAA DIVER Templates

DIVER is a NOAA application for the integration and distribution of response, assessment, and restoration data, as well as historical data collected from hazardous sites around the country to support the Natural Resources Damage Assessment (NRDA) program. NOAA DIVER includes field and laboratory protocols, data templates, COC forms, and field data collection forms to help standardize data collection. These forms and templates generally meet GLLA field data reporting requirements. Current documents can be downloaded by document type or environmental resource through the NOAA DIVER Dashboard. NOAA DIVER has replaced NOAA's Query Manager template, which is no longer accepted by GLNPO.

> ***Helpful Hint:*** *Field forms developed by NOAA aid sample collectors in accurately collecting and documenting required field information for transcription into the appropriate NOAA DIVER Template. GLNPO does not require the use of field forms, but does require field data to be submitted in a machine-readable electronic format (e.g., Microsoft Excel, CSV, XML). When using field forms to collect information, the data provider should submit PDF copies of any field forms used along with final project data.*

The most current NOAA Templates and Field Forms are continually updated, and the most current version should be obtained directly from the NOAA DIVER website prior to data collection. Both the Microsoft Excel and Microsoft Access version of these templates are acceptable. As NOAA is continually updating these templates to meet the evolving needs of the NRDA program, the most current version of these templates should be downloaded from the NOAA DIVER Dashboard.

5.2.1 Accepted NOAA DIVER Field and Lab EDD Templates

The DIVER ChemTox and BioLab templates are the only way to report toxicity and benthic community data to GLNPO. The NOAA Diver FieldObs Template provides a useful means of recording non-sample specific ecological data (e.g., bird counts, percent cover). Sediment chemistry may be reported using the NOAA Lab EDD Template provided that a supplemental PDF containing required laboratory raw data and supplemental information is included.

The following table provides the Template Name, Example Data, and General Information for the NOAA DIVER Templates that are typically used to report electronic data to GLNPO. Additional information can be found in the NOAA Template Guidance, dated June 1, 2021.

All NOAA DIVER Reporting Templates can be found on NOAA's Field Forms, Data Templates, and Guidelines page. Table 2 identifies the NOAA DIVER Templates accepted for GLLA final data deliverable packages and provides general information on each accepted template.

Table 2: NOAA DIVER Templates Accepted by GLNPO

Template Name	Example Data	General Information
ChemTox Sediment Chemistry and Toxicity Results	• Sediment chemistry • Tissue chemistry • 10-Day *Hyalella* survival • Other benthic toxicity results	This template is intended only for sediment, water, oil, tar, or tissue chemistry (only field data, no laboratory QC data), or bioassay data. Analytical laboratory results are typically appended to this template using the NOAA Lab EDD Template.
BioLab Biological, Habitat and other non-chem laboratory analysis (sample-based)	• Benthic invert community metrics • Fish histopathology • Fish deformities • Stable Isotope analysis	This template is used when submitting biological metrics (e.g., fish histopathology, benthic invertebrate community structure data), which may be stand-alone or paired with contaminant chemistry samples (entered in the ChemTox template).
FieldObs Field measurements and biological surveys (not sample-based)	• Bird Counts • Vegetation Surveys • Percent Cover Estimates • Water Quality Parameters	This template is used to submit biological metrics (e.g., bird counts, vegetation surveys) which are not sample based. These data may be stand-alone or paired with contaminant chemistry samples (entered in the ChemTox template) and/or biological and other non-chemistry laboratory data (entered in the BioLab template).
NOAA Tester Automated Template review program	• ChemTox • BioLab • FieldObs	This application is used to check the internal consistency of the ChemTox, BioLab and FieldObs Templates.
NOAA Lab EDD	• ChemTox	This EDD format can be easily appended to data in the ChemTox Template. All NOAA Lab EDD data fields are required for GLNPO deliverables, but null values are accepted if data are not applicable for the project.

While the file names and locations of these files are current as of publishing, these templates are continually updated. Data providers should check the NOAA DIVER website prior to data collection to verify that the most current version of the templates is used.

5.2.2 GLNPO Required Data Fields and Valid Values for the NOAA DIVER Data Templates

The NOAA DIVER templates include both required data fields and valid values, which are accepted by GLNPO. GLNPO requires the following additional pieces of data, if available in the template, which are not required by NOAA DIVER:

Study Worksheet:

- Contact Name
- Contact Phone Number
- Contact Email
- County
- State

SampleMaster Worksheet:

- Sample Collection Time
- Water Depth/Water Depth Units
- Notes (if applicable: provides sediment depth, whether sediment was collected to refusal, and method used to collect sediment depth).

If the study did not require sample-specific measurements (i.e., habitat data) please state in the "Notes" data field of the appropriate worksheet that this information was not collected.

While sediment chemistry information may be provided in the NOAA Diver Lab EDD Template, which can be appended to the ChemTox template, complete sediment chemistry information is more typically provided in the Region 5 EDD format.

5.2.3 GLNPO Required Data Fields for the NOAA Lab EDD Template

The NOAA Lab EDD Template allows laboratories to report sediment chemistry data in a format that can be easily appended to field data recorded in the ChemTox Template. **GLNPO requires all data fields in this EDD, including those listed as "optional" in the NOAA Lab EDD Template, but allows null values if the data field is not applicable for the project.**

Sediment chemistry data provided in the NOAA Lab EDD are typically appended to the NOAA ChemTox Template prior to data validation and delivered to GLNPO as a single EDD. Please note that when toxicity samples are collected in support of a GLLA sediment assessment or remediation project, the laboratory often provides sediment chemistry for surface-collected samples in the NOAA Lab EDD format as well as full sediment chemistry results (including sub-surface samples). The Region 5 EDD or SEDD format helps facilitate complete data evaluation and minimize manipulation and manual transcription that may be required to add data to the project data management system. The data provider should determine the most appropriate approach for data reporting to meet project DQOs and document the approach and associated laboratory reporting requirements in the QAPP.

5.3 SEDD Format

The SEDD format was developed as part of an inter-agency effort to create a uniform, non-proprietary reporting format for the electronic delivery of analytical chemistry data that are conducive to automated data review. The data deliverable generated by SEDD is an XML file that can be mapped directly to GLSED or to other EPA accepted EDDs. The SEDD Specifications, Version 5.2 (March 2019) provides detailed information on this data deliverable. Please note that GLNPO only accepts SEDD Version 5.2 file formats.

SEDD is a hierarchical format based on a model of laboratory activities, the linkages between these activities, and the data that these activities produce. Laboratories typically provide data in one of the three primary stages of SEDD, based on the level of detail required.

- **Stage 1** contains the minimum amount of data required to transmit "results-only" data. Only limited method QC data (or no QC data at all) are reported in Stage 1.
- **Stage 2** has two distinct levels that build on Stage 1. Stage 2a adds method QC data, and Stage 2b adds instrument QC data.
- **Stage 3** builds on Stage 2 by adding additional measurement and calibration data that allows for independent recalculation of reported results.

Additional assistance in preparing this deliverable, including the SEDD Valid Values, can be found at https://www.epa.gov/clp/staged-electronic-data-deliverable-sedd.

When submitting SEDD files for automated data review, laboratories are urged to deliver:

- SEDD Stage 2a electronic deliverables for dioxin and PCB congeners,
- SEDD Stage 2b or 3 electronic deliverables for Aroclors, pesticides, volatile organic compounds, and semi-volatile organic compounds (including polycyclic aromatic hydrocarbons), and
- Stage 2b or 3 electronic deliverables for metals, mercury and cyanide.

Laboratories utilizing the Stage 2a or 2b deliverable will be required to also submit PDF files containing the raw data (Section 4.4). The CLP SOWs, Exhibit H provide guidance to the CLP laboratories for delivery of SEDD files.

Laboratories submitting SEDD files should process their deliverables through EXES or similar automated data review software. EXES is a free tool that can be accessed through the Sample Management Office (SMO) Portal at smoclpss.com. Access is granted for each user account as approved by the GLLA QA Manager. If all information is complete and compliant with SEDD and GLNPO requirements, EXES will automatically generate a GLSED and EQuIS-compatible data deliverable, electronic data validation reports, and preliminary data qualifiers. If Scribe is used to collect field data, Scribe XML files should be uploaded to the SMO Portal within 72 hours of sample collection. EXES will automatically merge field and laboratory data if all provided information is complete. Site contractors are encouraged to follow this process (as practical) to streamline data verification and validation.

5.4 Mini-GLSED Data Deliverable

Completed GLLA study data are stored in GLNPO's GLSED. GLSED is a relational database that includes field and analytical result information for a number of matrices and analyte classes. In addition to GLSED, which stores data from multiple GLLA projects, single-study data are stored within a "mini-GLSED." While data submissions are typically imported into a mini-GLSED upon completion of the DSCO review, the data provider may populate data directly into a mini-GLSED. This is typically completed when the data provider stores data in a proprietary database or a data format not accepted by GLNPO, and exports the data to a mini-GLSED format. A data element dictionary may be obtained from the GLLA QA Manager.

5.4.1 Mini-GLSED Field Data

The mini-GLSED format includes multiple field sample tables for which sample-level and field-specific information can be stored:

- ptbl – STATION (location information for each collected sample)
- ptbl – SAMPLE (sample level information for field samples for all matrices other than tissue)
- ptbl – SAMPLE_QC (sample level information for field QC samples)
- ptbl – SMPTISS (sample level information for tissue field samples)
- ptbl – SMPTISS_QC (sample level information for tissue QC samples)
- ptbl – PROBE (field probe depth measurement data)

5.4.2 Mini-GLSED Lab Data

GLSED includes ten tables for which laboratory data can be stored, listed below:

- ptbl – CHEM (sediment chemistry sample results for all analytes other than acid volatile sulfide/simultaneously extracted metals (AVS/SEM)

- ptbl – CHEM_AQ (aqueous chemistry sample results for all analytes other than AVS/SEM)
- ptbl – AVS_SEM (sediment and/or aqueous chemistry sample results for AVS/SEM)
- ptbl – CHEMTISS (tissue chemistry sample results)
- ptbl – CHEM_QC (QC data associated with all non-tissue samples for all analytes)
- ptbl – CHEMTISS_QC (QC data associated with tissue samples for all analytes)
- ptbl – CHEM_TCLP (TCLP Extract Sample Results)
- ptbl – BIOSUMM (toxicity and bioaccumulation sample results)
- ptbl – BENTHOSREP (fish and benthic community replicate data)
- ptbl – BENTHOSAMPLE (fish and benthic community data, including mean, standard deviation, and summary data)

A GLSED data element dictionary and an example of a mini-GLSED with populated laboratory data tables is available from the GLLA QA Manager.

5.5 Simplified Spreadsheet Format

GLNPO may contract directly with a laboratory to support research needs. These laboratories typically submit data directly to GLNPO but may not have the data management capabilities to generate the relational or hierarchical data submission formats described in the preceding sections. In these circumstances, directly contracted laboratories have the option of submitting data in a single spreadsheet to meet minimum data requirements. A data element dictionary for the simplified spreadsheet EDD is included as Attachment 5. The spreadsheet should include all the information required for laboratory data as detailed in Section 4.3.4 and encompass all matrices and both field sample and sample-level QC results.

The spreadsheet should also include field information and must include station IDs, sample matrix, sampling date and time, and sampling depths. All available values should be populated in appropriate data fields in the delivered spreadsheet.

> ***Helpful Hint:*** *When populating spreadsheet files, avoid the use of formatting to convey information, because formatted information may be lost when data are imported or transported. For example, entering a percent solid result as 39% could mean that the imported value would become 0.39. The "%" should be placed in the separate RESULT_UNIT data field.*

6.0 Geospatial Database Deliverables

Significant time and effort are invested by GLLA project teams to create geospatial databases necessary to model the nature and extent of contamination and produce the maps required for project plans and reports. Advantages of having a study's geodatabase available to GLNPO are:

- storage of multiple feature classes, raster datasets, and tables in a single location (folder), making it easier for GIS users to share data; and
- increased capabilities for importing, mapping and sharing data on EPA's GeoPlatform.

Technical Specification for Geodatabase Deliverables:

Spatial data should be submitted to GLNPO in the ESRI File Geodatabase format with a defined coordinate system. If applicable, submissions should include the collection method(s). Each geodatabase

should be accompanied by FGDC compliant metadata as described in Section 4.2. The geodatabase deliverable should be submitted as part of the GLSED deliverable package.

7.0 Data Deliverable Package Checklist

To date, over two hundred final data packages have been delivered to GLNPO for upload to GLSED, in support of sediment assessment and remediation projects conducted under GLLA. Some deficiencies noted in the data packages received were repeated across multiple studies. Included in Attachment 3 is a checklist that can be used by laboratories and data providers when preparing the deliverable packages to avoid these common deficiencies. Nearly 95 percent of the EDDs by GLNPO received to date have been in the Region 5 EDD format, so many suggested resolutions to these issues reference the Region 5 data elements. GLNPO recommends providing this checklist to the laboratory during the project planning/QAPP development phase of the project to ensure the chosen lab is able to meet these reporting requirements.

8.0 Contacts

Laboratories, field crews and all other data providers are responsible for providing complete and compliant data of acceptable quality in a format useable by GLNPO. As questions arise during the analysis of samples and their report preparation, laboratories should contact the following representatives in the following order:

1. GLLA Project Manager(s)
2. Mark Loomis, GLLA QA Manager, (312) 886-0406, loomis.mark@epa.gov
3. Louis Blume, GLNPO Quality Manager, (312) 353-2317, blume.louis@epa.gov
4. Kenneth Klewin, GLENDA Manager, (312) 886-4794, klewin.kenneth@epa.gov

9.0 Resources

The following resources provide additional information concerning GLLA data deliverables and data quality requirements:

1. The *Quality Assurance Considerations for Great Lakes Legacy Act Projects* (August 2023), which provides information for GLLA site contractors on important GLLA QA requirements for project planning, DQO development, project analytical method and acceptance criteria, data validation, and final data submission. This document is available from the GLLA QA Manager.
2. The Superfund CLP provides SOWs for many analytical methods used by GLLA. The SOWs detail the correct assembly of data in CLP format. The methods can be found at: https://www.epa.gov/clp/superfund-clp-analytical-services
3. The EPA has produced several documents on the data review process, including:
   - *Guidance on Environmental Data Validation and Verification (EPA QA/G-8)*, EPA-240-R-02-004: https://www.epa.gov/sites/production/files/2015-06/documents/g8-final.pdf
   - *Guidance for Labeling Externally Validated Laboratory Analytical Data for Superfund Use,* EPA-540-R-08-005: https://www.epa.gov/system/files/documents/2021-10/guidance-for-labeling-externally-validated-laboratory-analytical-data-for-superfund-use-2009.pdf
   - *EPA Contract Laboratory Program National Functional Guidelines for Organic Superfund Methods Data Review*, EPA-540-R-20-005, November 2020:

https://www.epa.gov/sites/default/files/2021-03/documents/nfg_for_organic_superfund_methods_data_review_november_ 2020. pdf

- *EPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Methods Data Review*, EPA-540-R-20-006, November 2020: https://www.epa.gov/sites/default/files/2021-03/documents/nfg_for_inorganic_superfund_methods_data_review_november_2020.pdf
- *EPA Contract Laboratory Program National Functional Guidelines for High-Resolution Superfund Methods Data Review*, EPA 542-R-20-007 https://www.epa.gov/sites/default/files/2021-03/documents/nfg_for_hrsm_superfund_methods_data_review_november_2020.pdf

4. Federal Geographic Data Committee

   http://www.fgdc.gov/metadata/geospatial-metadata-standards. This site lists the content standard for digital geospatial metadata.

ATTACHMENT 1

GLLA QA PROCESS DIAGRAMS

GLLA QA Planning Process: August 2023

Project Partners

GLNPO/NFS Project Agreement, Inter-Agency Agreement or GLRI Assistance Agreement

Develop DQOs and Sampling Plan

Review QAPP

Field Sampling

GLLA Project Manager

Develops SOW/IGCE

Sends SOW, IGCE, QARF to C-COR and QA Manager

Approve DQOs/Sampling Plan

Review QAPP

Prime Contractor

Negotiations

Task Order Award

EXES training and SEDD testing for laboratory (if applicable)

Draft QAPP

Revise QAPP

Final or Contingent QAPP

Data

EPA Contracts Office

C-COR Reviews SOW/IGCE/QARF

CO-CS Review SOW/IGCE

Proposal

Contract Level Audit (as necessary)

GLLA QA Manager/ QA Group

Review SOW/IGCE and QARF Signs and loads to QA Track

Pre-QAPP meeting to discuss project-specific questions or concerns and identify technical assistance needs. Communication with contractor QA Lead throughout process

Review QAPP Request updates as needed

Re-review/ Approve QAPP

QA Field Audit or TSA (as necessary)

QA Support Contractor

Provides EXES access for lab (if applicable)

Reviews QAPP and provides comments

Reviews QAPP and provides comments

Technical Assistance for TSA

GLLA Data Evaluation Process: August 2023

Site Contractors

Field/Geospatial data verification by Prime Contractor

Data

Field Data

Lab Data

Manual Data Validation

Lab reports data in accordance with QAPP requirements and GLLA Data Reporting Standard

Complete manual data validation (100% GLLA Tier 1 / 20% GLLA Tier 2) by EPA QA Contractor or sub-contractor using lab EDD and PDF data package

or

EXES Data Validation

Lab uses EXES to validate SEDD deliverable against QAPP requirements. EXES must be setup as a part of sub-contracting analytical work. Coordinate with QA Manager and QA Support Contractor.

Supplemental manual validation by EPA QA Contractor or sub-contractor using EXES EDM, preliminary validation reports, and PDF data package

Final Merged Data Set

Final lab data including validation narratives

Data Usability Report or Final Project Report

GLSED Deliverable

See GLSED Diagram and GLLA Data Submission Site

Approve Final Data

Closeout

GLLA QA Manager & QA Support Contractors

Request analysis of single-blind PE sample provided by GLLA QA Contractor

GLLA QA Manager may communicate with Contractor's QA Lead throughout process as needed.

GLLA Project Manager

Ensure compliance with SOW and QAPP
Ensure adequate contract funding
Review/approve invoices (or advise C-COR)

Evaluate Data Quality and Usability, Reconcile DQOs, Make Decision

[1] Data verification evaluates completeness, correctness and conformance/compliance of the data set against method, procedural or contractual specifications

[2] Data validation is an analyte/sample specific evaluation beyond data verification to determine the quality of the data relative to its end use

GLLA Final Data Reporting Process: August 2023

Site Contractor

Upload final data package to GLLA Data Submission Site per requirements of the Data Reporting Standard. Submit Final QAPP and any addenda

Respond to DSCO and submit revised data or supplemental information as needed

Respond/Resolve – submit new versions or supplemental information as needed

QA Support Contractor

Enter project into tracking system with expected data delivery date

Receive final data package via data submission site

Evaluate final data package using DSCO and final QAPP. Perform locational data checks

Submit DSCO

Initiate DRT which tracks resolution of all issues identified throughout the data management process

Upload to Mini-GLSED

Evaluate completeness and compliance of GLSED upload using a standard checklist

Import mini into SAS, evaluate completeness of upload against original source files, and conduct data integrity checks

Conduct QIC review, draft report, and provide identified issues

Receive information and update DRT and mini GLSED

Prepare mini GLSED and finalize QIC report

Update Master GLSED with approved mini GLSEDs

MASTER GLSED

GLLA Data Status Report tracks project data in real time

GLLA QA Manager

Provide copy of final QAPP upon approval

Direct or refer Site Contractor to work with QA Contractor on GLSED upload

Review DSCO

Issue a DMR to Project Manager if DSR indicates excessive delays in response from prime contractor

Mini GLSED Review and Approval

GLLA Project Manager

Forward DMR to Contractor and facilitate resolutions

Mini GLSED Review (as needed)

Contractor can proceed with project closeout after mini GLSED is approved by GLLA QA Manager

Retrieve Data from GLSED:

- Data converted to **GLENDA** standard and uploaded **via CDX**
- **NOAA DIVER** (periodic transfers)
- To obtain project-specific exports/data: **Complete a DRF** and submit to GLLA QA Manager. DRFs track distribution and version control of data

Acronyms

CDX – Central Data Exchange
DMR – Data Management Request
DRF – Data Request Form
DRT – Discrepancy Resolution Tracking*
DSR – Data Status Report
DSCO – Data Submittal Compliance Overview*
GLENDA – Great Lakes Environmental Database
GLSED – Great Lakes Sediment Database
QAPP – Quality Assurance Project Plan
QC – Quality Control
QIC – Quality Information Completeness*
SAS – Statistical Analysis System

*Provided in GLSED with project data

ATTACHMENT 2

MINIMUM REQUIREMENTS FOR THE SUBMISSION OF FIELD DATA FOR GLLA SEDIMENT PROJECTS

Attachment 2: Minimum Requirements for the Submission of Field Data for GLLA Sediment Projects

This document provides the minimum requirements for the submission of field data for sediment samples collected in support of Great Lakes Legacy Act (GLLA) projects. This document supplements information in the *Great Lakes Legacy Act Data Reporting Standard, April 2023*. Please note that GLLA projects may require the collection and submission of additional field data elements, including field data for other types of samples; these additional data elements should be documented in the project-specific qualify assurance project plan. Project participants also are required to complete the *Locational Data Checklist and Metadata Recording Form* (Attachment 4) for each sampling event.

The minimum field data elements to be reported are provided below for four categories: 1) data provider contact information, 2) study area information, 3) station information, and 4) sample information.

Data Provider Contact Information

Provide contact information for data provider including:

- First and last name
- Affiliation
- E-mail address and phone number

Study Area Information

Provide information on the study area, including:

- Site name
- County (or county code)
- State
- Coordinates, date and time of collection, and brief description of at least one control point (or reference location point, if control point is not available)[1]

Station Information

For each sampling station, provide:

- Unique station identifier
- Latitude and longitude coordinates in decimal degrees in WGS 84 or NAD 83
- Water depth with units of measure (reported according to the Electronic Data Deliverable (EDD) specification)[2]

[1] See Section 4.1.3 in the Data Reporting Standard for a description of reference locations. This information should be reported in the Locational Data Checklist and Metadata Recording Form (Attachment 4).

[2] If depth measurements were collected as elevation, please provide a description of the associated reference.

- Sediment depth with units of measure and method for measuring sediment depth (pole, vibracore, etc.) and whether sample was collected to refusal[3]
- Documentation for samples not collected at a proposed station (due to issues such as insufficient amount of sediment, thick layer of ice, etc.) by recording zero for sediment depth along with all other available station information

Sample Information

For each sample, provide:

- Associated station identifier
- Unique sample identifier
- Sample type (e.g., routine field sample, field duplicate sample, equipment blank)
- Sample collection date and time
- Upper and lower sediment collection depths with units of measure
- Sediment sampling technique
- Whether the sample was a composite or a grab
- Sediment sample observations (e.g., color, texture, odor)

[3] When submitting data using the NOAA ChemTox/BioLab Templates, report this information in the "Notes" data field of the SampleMaster worksheet. When submitting data using the EPA Region 5 EDD, report this information in the "depth_to_bedrock" and "loc_purpose" data field of the Location file (EPAR5LOC_v3). Please provide the method used for determining sediment depth in a cover letter or supplemental file.

ATTACHMENT 3

GLLA FINAL DATA DELIVERABLE PACKAGE CHECKLIST

Attachment 3: GLLA Final Data Deliverable Package Checklist

This document complements the *GLLA Data Reporting Standard, April 2023.* Over two hundred final data deliverable packages have been delivered to the U.S. Environmental Protection Agency's (EPA's) Great Lakes National Program Office (GLNPO) for upload to the Great Lakes Sediment Database (GLSED) in support of sediment assessment and remediation projects conducted under the Great Lakes Legacy Act (GLLA). Many data quality issues noted in these final data deliverable packages were repeated across multiple studies and subsequently addressed prior to upload. GLNPO recommends that laboratories and data providers use this checklist when preparing final data deliverable packages for upload to the GLLA Data Submission Site on GLNPO.net to avoid common issues and speed the GLSED review and approval process. Nearly 95 percent of the Electronic Data Deliverables (EDDs) delivered to GLNPO to date have been in the EPA Region 5 EDD format; therefore, references to Region 5 data elements are included in the required supporting information described in the checklist. GLNPO recommends providing this checklist to the laboratory during the project planning/Quality Assurance Project Plan (QAPP) development phase of the project to ensure the chosen laboratory is able to meet these reporting requirements. GLNPO encourages data providers to submit a completed version of the checklist as part of the final data deliverable package.

GLLA Final Data Deliverable Package Checklist		
Required Supporting Information		**Included (✓)**
Final QAPP, FSP and Addendums	Include the final, approved QAPP and FSP (if separate) and any addendums to the QAPP or FSP; make sure to provide document tables, figures, and appendices.	☐
Final Project Reports and Technical Memoranda	Include final project reports and project technical memoranda (i.e., site characterization reports, data usability assessments, reconciliation with DQOs, etc.), especially those identified as project deliverables in the QAPP. This information assists the reviewer in reconciling data quality issues and QAPP deviations.	☐
Deviations from Sampling Plan	In the deliverable cover letter or in a supplemental file, please discuss briefly, as appropriate, why 1) the number of samples collected significantly deviated from those proposed in the sampling plan, or 2) sample locations were off set from proposed sample locations.	☐
Deviations from Analytical Plan	In the deliverable cover letter or in a supplemental file, please discuss briefly, as appropriate, why 1) the list of target analytes differs from the list provided in the QAPP, or 2) analyses were conducted by laboratories different than those specified in the QAPP.	☐
Calculated Totals for Derived Results (e.g., Total PCBs, Total PAHs)	In the deliverable cover letter or in a supplemental file, describe how the totals (e.g., PAHs, PCBs, Dioxins,) provided in the EDD were calculated noting: 1) how non-detects were treated (e.g., ND=0, ND=1/2RL), 2) how qualifiers were handled (e.g., whether "J" flags were carried over to totals), 3) who calculated the totals (e.g., lab, sampling contractor), 4) the individual compounds included, and 5) how co-eluting compounds were treated.	☐

GLLA Final Data Deliverable Package Checklist		
Locational Data Checklist and Metadata Recording Forms	Provide copies of completed *GLLA Locational Data Checklist and Metadata Recording Forms* and ensure the following information is included: • Control point or reference point information (page 2 of *GLLA Locational Data Checklist and Metadata Recording Forms)* • GPS brand and model number(s) • Model accuracy • Final post-processed accuracy of the data	☐
Qualifier Definitions	Provide definitions for all laboratory and validator applied qualifiers, even if common qualifiers were used.	☐
Data Verification/Validation/ Usability Reports	Provide copies of all available data verification, validation, and usability reports prepared for the study as well as any automated review reports.	☐
Site Name	Provide site name information in a supplemental file or in the "subfacility_name" data field of the EPA Region 5 **Field** EDD subfacility file (EPAR5SUBFACILITY_v3).	☐
County	Provide "County" information in a supplemental file or the "loc_county_code" data field of the EPA Region 5 **Field** EDD location data file (EPAR5LOC_v3).	☐
State	Provide "State" information in a supplemental file or in the "loc_state_code" data field of the EPA Region 5 **Field** EDD location data file (EPAR5LOC_v3).	☐
Water Depth	Provide water depth in a supplemental file (with units of measure) or in the "remark" data field of the EPA Region 5 **Field** EDD location data file (EPAR5LOC_v3), expressed in units of feet.	☐
Sediment Depth	Provide sediment depth in a supplemental file (with units of measure) or in the "depth_to_bedrock" data field of the EPA Region 5 **Field** EDD location data file (EPAR5LOC_v3), expressed in units of feet.	☐
Method for Sediment Depth Collection	Identify the method used to determine sediment depth (e.g., graduated pole, vibracore) and whether sediment was collected to refusal in a supplemental file or in the "loc_purpose" data field of the EPA Region 5 **Field** EDD location data file (EPAR5LOC_v3).	☐
Bathymetric Survey (if conducted)	Provide a PDF copy of bathymetric survey report(s), if a bathymetric survey was conducted	☐
Toxicity or Bioaccumulation Data (if generated)	Provide toxicity and/or bioaccumulation data, when generated, using the NOAA DIVER ChemTox Template. If toxicity/bioaccumulation data are not able to be provided in the NOAA DIVER ChemTox Template, provide a PDF copy of the laboratory report covering the toxicity/bioaccumulation tests.	☐

GLLA Final Data Deliverable Package Checklist		
Benthic/Fish Community Data (if generated)	Provide community data, when generated, using the NOAA DIVER BioLab Template. If benthic or fish community data are not able to be provided in the NOAA DIVER BioLab Template, provide a PDF copy of the laboratory report covering the fish/benthic community findings.	☐
Additional EDD Checks		**Included (✓)**
AVS_SEM Data	Report AVS and SEM metals data in units of umoles/g.	☐
QC Batch IDs	Provide Batch IDs that relate field sample result records and their associated QC results.	☐
Unique Station Identifiers	Confirm that station identifiers are unique to each sampling location.	☐
Coordinate Information with Associated Datum	Provide coordinate information in WGS84 or NAD83 latitude and longitude in a supplemental file or in the "X_coord" and "Y_coord" data fields of the EPA Region 5 **Field** EDD location data file (EPAR5LOC_v3), and specify the Datum provided in the "horz_datum_code" data field.	☐
	Identify the coordinate system used and associated Datum for projected coordinates, when provided.	☐
Method and Field QC Results	Provide all associated method and field QC results for each analytical method reported as specified in the project QAPP: • Method blanks • LCS/LCSDs • MS/MSDs • Lab duplicates • Field duplicates • Equipment blanks	☐
Spike and Surrogate Results in %R	Provide percent recovery (%R) information or the data needed to calculate percent recoveries for QC spikes and surrogates.	☐
Reportable Results	Complete the "reportable_result" data field of the EPA Region 5 **Lab** EDD chemistry test result with QC file (EPAR5TRSQC_v3) when results for multiple dilutions and/or re-analyses are reported for a sample. Confirm that only one result was reported as reportable (i.e., "Yes") for each analyte for each sample.	☐
	Provide a complete set of reportable results for each analytical method and sample. Confirm that all target analytes portable result (i.e., no missing analyte results).	☐
Sample-adjusted MDLs or RLs, and QLs	Report the adjusted MDLs or RLs and QLs for all sample results for all methods as appropriate.	☐

GLLA Final Data Deliverable Package Checklist		
Laboratory Locations	Confirm that the laboratory is reported in the "lab_name_code" data field of the EPA Region 5 **Lab** EDD chemistry test result with QC file (EPAR5TRSQC_v3) and is consistent with the laboratory identified in the QAPP.	
Final Data Qualifiers	Provide final qualifiers based on an interpretation of the lab and validator flags in the "interpreted_qualifier" data field of the EPA Region 5 **Lab** EDD chemistry test result with QC file (EPAR5TRSQC_v3).	☐
Non-detect Reporting	The preferred format for reporting non-detects is to set the concentration to the sample-specific detection limit and the final interpreted flag to "U." Alternatively, it is acceptable to leave the concentration field for non-detected analytes blank as required by the EPA Region 5 EDD format. Describe in the deliverable cover letter how non-detected analytes were reported if not reported in one of these formats.	☐

ATTACHMENT 4

LOCATIONAL DATA CHECKLIST AND METADATA RECORDING FORM

Attachment 4: Locational Data Checklist and Metadata Recording Form

This document accompanies the *Great Lakes Legacy Act (GLLA) Data Reporting Standard* , which provides detailed data reporting guidance for GLLA projects, including electronic data deliverables (EDD) requirements. GLLA data providers should review this document prior to collecting data. Copies of completed forms should be uploaded to the GLLA Data Submission Site on GLNPO.net with the complete GLLA data submission.

Contact Information

Contact Name: _____ Phone Number: _____

Affiliation: _____ E-mail Address: _____

Study Information

Project Title: _____

Site Name: _____

Sampling Start Date: _____ Sampling End Date: _____

Preparation Activities (please confirm each activity in the boxes to the right)

1. Sampling staff are trained in GPS Field Data Collection and have familiarized themselves with the GPS unit used for this project (certified training recommended). ☐

2. Determined window of satellite availability. https://www.gnssplanning.com ☐

3. Locate at least one control point to assess GPS accuracy. ☐
   For assistance locating control points, visit http://www.ngs.noaa.gov/datasheets or http://www.geocaching.com/mark/. This may not be feasible if the GPS unit is mounted to a vessel.*

4. If a control point cannot be located, establish at least one reference point.* ☐

Data Collection Activities (please confirm each activity in the boxes to the right)

1. GPS unit was configured to collect data only when the following requirements were met:
   a. A minimum of four satellites ☐
   b. Position dilution of precision (PDOP) ≤ 6 ☐
   c. Satellite elevation ≥ 15° above the horizon ☐
   d. A minimum signal-to-noise ratio (refer to GPS user manual for recommendation) ☐

2. Collected point data based on the nearest base station's logging interval. ☐

3. Collected point data for a period of at least 1 minute per location. ☐

4. Reported locational data in WGS 84 or NAD 83 (please specify_____). ☐

Please provide an explanation if a box was not checked for any of the responses above and specify deviations (include sample IDs if applicable):

_____

_____

_____

*At minimum, collect on the first day of sampling prior to beginning data collection and on the last day of sampling following data collection. It is recommended to collect at the start and end of each sampling day. Record on page 2.

GPS Unit Specifications

GPS brand and model number: _____

Model accuracy: _____

Data Processing

Which of the following best describes any data correction that may have been performed:

☐ real-time correction - specify type _____ ☐ post processed differential correction - provide base station id and location _____

☐ no correction ☐ other, please specify _____

Quality Information

Describe any difficulties in collecting locational data: _____

_____

Data Provider:

Confirm required information has been provided.

_____ _____

Signature Date

Locational Data Checklist and Metadata Recording Form

Locate at least one control point to assess GPS accuracy. If a control point cannot be located, establish at least one reference point. At minimum, collect on the first day of sampling prior to beginning data collection and on the last day of sampling following data collection. It is recommended to collect at the start and end of each sampling day.

Project Title: _____

Date: _____

Control Point 1

Benchmark ID: _____ Time: _____

Established x-coordinate: _____ Measured x-coordinate: _____

Established y-coordinate: _____ Measured y-coordinate: _____

Horizontal distance offset (include UOM): _____

Established elevation: _____ Measured elevation: _____

Vertical distance offset (include UOM): _____

Control Point 2

Benchmark ID: _____ Time: _____

Established x-coordinate: _____ Measured x-coordinate: _____

Established y-coordinate: _____ Measured y-coordinate: _____

Horizontal distance offset (include UOM): _____

Established elevation: _____ Measured elevation: _____

Vertical distance offset (include UOM): _____

Reference Point 1

Time: _____

Location description: _____

Estimated x-coordinate: _____ Measured x-coordinate: _____

Estimated y-coordinate: _____ Measured y-coordinate: _____

Horizontal distance offset (include UOM)*: _____

Reference Point 2

Time: _____

Location description: _____

Estimated x-coordinate: _____ Measured x-coordinate: _____

Estimated y-coordinate: _____ Measured y-coordinate: _____

Horizontal distance offset (include UOM)*: _____

Control point: a previously surveyed location with known coordinates (e.g., a benchmark).

Reference point: a recognizable feature within or near the study area that is viewable using aerial photography.

UOM: unit of measure - preferably recorded in feet or meters.

*Calculated by estimating the reference point on aerial photography and measuring the distance to the recorded position.

ATTACHMENT 5

DATA ELEMENT DICTIONARY FOR THE SIMPLIFIED SPREADSHEET EDD FILE

Attachment 5: Data Element Dictionary for the Simplified Spreadsheet EDD File

This document provides the Data Element Dictionary for the Simplified Spreadsheet EDD. Use of this EDD must be pre-approved by the GLLA QA Manager and documented in the project QAPP. The Simplified Spreadsheet EDD is typically only accepted when GLNPO contracts directly with a laboratory to support research needs and the laboratory is submitting data directly to GLNPO. These laboratories may not have the data management capabilities to generate the relational or hierarchical data submission formats required by other data reporting formats, and thus have the option of submitting data in a single spreadsheet to meet minimum data requirements.

The Simplified Spreadsheet EDD should include all of the information required for laboratory data, and encompass all matrices and both field sample and sample-level QC results. In addition to laboratory-level information, the simplified spreadsheet format should include field information when available. Required field information for this file format are: 1) Station ID, 2) sample matrix, 3) sampling date and time, and 4) sampling depths. All available values should be populated in the delivered spreadsheet.

Field # -- Column	**Field Name**	**Description**	**Comments**
1 – A	DATA_PROVIDER	Name of company or agency submitting EDD	
2 – B	STATION_ID	Station Location	
3 – C	SAMPLE_NAME	Unique Sample ID	
4 – D	MEDIUM	Sample Matrix	
5 – E	SAMPLE_MATRIX_CODE	Sample Matrix Code	Water = W Soil = S Sediment = SED Porewater = PW
6 – F	SAMPLE_TYPE_ CODE	Sample Type Code	Field Sample, Performance Evaluation Sample = N Field Blank = FB Field Duplicate = FD Laboratory Control Sample = LCS Laboratory Control Sample Dup = LCSD Laboratory Duplicate = LD Matrix Spike, Post Digestion Spike = MS Matrix Spike Duplicate = MSD Method Blank = MB Storage Blank = SB

Field # -- Column	Field Name	Description	Comments
7 – G	SAMPLE_SOURCE	Identifies where the parent sample originated.	Field Sample, Field Blank, Performance Evaluation Sample, Matrix Spike, Matrix Spike Duplicate = FIELD Laboratory Control Sample, Method Blank, Storage Blank= LAB
8 – H	SAMPLE_COLL_ PROC_ID		Field is null
9 – I	SAMPLE_ID_ DUPLICATE		Field is null
10 – J	SAMPLE_DEL_ GROUP	Sample Delivery Group (SDG) Number	
11 – K	SAMPLE_DATE	Date sample was collected; reported in MM/DD/YYY format	
12 – L	SAMPLE_TIME	Time sample was collected; 24-hr format HH:MM	
13 – M	UPPER_DEPTH	Upper depth of sample collection interval from sediment surface	
14 – N	LOWER_DEPTH	Lower depth of sample collection interval from sediment surface	
15 – O	DEPTH_UNIT	Unit of measure for upper and lower depths	
16 – P	CHAIN_OF_ CUSTODY	Chain of Custody Identifier	
17 – Q	SENT_TO_LAB_ DATE	Date sample was shipped; reported in MM/DD/YYYY HH:MM format. Time must be in 24-hr format.	
18 – R	SAMPLE_RECEIPT_DATE	Date sample received by the lab; reported in MM/DD/YYYY HH:MM format. Time must be in 24-hr format.	
19 – S	SAMPLER_NAME	Sampler Name or Initials	
20 – T	TASK_CODE		Field is null

Field # -- Column	Field Name	Description	Comments
21 – U	QAPP_APPROVAL_DATE		Field is null
22 – V	SAMPLE_ COMMENT	Any comments regarding the sample	
23 – W	SAMPLE_ID	Sample Number	
24 – X	CHEMICAL_NAME	CAS Chemical Name	Specific Guidance Regarding Chemical Names available upon request
25 – Y	CAS_RN	CAS Number	CAS Registry Number
26 – Z	FRACTION	Analytical Fraction	VOA_Low_Med = V VOA_Trace = VT VOA (SIM) = VS BNA = B BNA (SIM) = BS Aroclor = A PEST = P Inorganic = I Note: if another value is used, please define in the narrative or cover letter
27 – AA	VALUE_TYPE	Value Type	"ACTUAL"
28 – AB	STAT_TYPE		Field is null
29 – AC	DURATION_BASIS		Field is null
30 – AD	TEMPERATURE_ BASIS		Field is null
31 – AE	RESULT_VALUE	Reported Result	
32 – AF	RESULT_UNIT	Concentration Units	
33 – AG	CONTAINER_ TYPE		Field is null
34 – AH	CONTAINER_SIZE		Field is null
35 – AI	CONTAINER_SIZE_UNITS		Field is null
36 – AJ	CONTAINER_ COLOR		Field is null
37 – AK	TEMP_ PRESERVE_ TYPE	Sample Temperature	
38 – AL	CHEM_ PRESERVATIVE	Preservative Name	
39 – AM	LAB_ANL_ METHOD_ CODE	Name of Analytical Method	
40 – AN	ANALYSIS_DATE	Date sample was analyzed; reported in MM/DD/YYY format	
41 – AO	ANALYSIS_TIME	Time sample was analyzed; 24-hr format HH:MM	
42 – AP	TEST_TYPE	Test Type	Initial, Dilution, Re-analysis, etc.

Field # -- Column	Field Name	Description	Comments
43 – AQ	LAB_MATRIX_ CODE	Sample Matrix Code	Water = W Soil = S Sediment = SED Porewater = PW
44 – AR	ANAL_ LOCATION	Analysis Location	Lab or Field
45 – AS	WET_OR_DRY_ BASIS	Results Basis	"WET" for wet-weight basis reporting. "DRY" for dry-weight basis reporting. "NA" where not applicable.
46 – AT	DILUTION_ FACTOR	Effective test dilution factor	
47 – AU	PREP_METHOD_ NAME	Preparation Method	
48 – AV	PREP_DATE	Beginning date when sample preparation started; reported in MM/DD/YYY format	
49 – AW	PREP_TIME	Time sample preparation began; 24-hr format HH:MM	
50 – AX	LAB_NAME_CODE	Laboratory Name Code	
51 – AY	QC_LEVEL	QC Level	"SCREEN" for screening data. "QUANT" for quantitative data.
52 – AZ	LAB_SAMPLE_ID	Laboratory Sample ID	
53 – BA	PERCENT _MOISTURE	Percent Moisture	Report in numeric format (i.e., 70.1% should be reported as 70.1 not as 70.1%)
54 – BB	SUBSAMPLE_ AMOUNT	Amount of sample used for test.	
55 – BC	SUBSAMPLE_ AMOUNT_UNIT	Unit of measure for Subsample amount.	
56 – BD	TEST_COMMENT		Field is null
57 – BE	FINAL_VOLUME	Final Volume of sample after preparation.	
58 – BF	FINAL_VOLUME_ UNIT	Final Volume Units	
59 – BG	RESULT_ERROR_ DELTA		Field is null
60 – BH	RESULT_TYPE_ CODE	Result Type Code	TRG: Target SC: Spiked Compound SUR: Surrogate Compound IS: Internal Standard TIC: Tentatively Identified Compounds

Field # -- Column	Field Name	Description	Comments
61 – BI	REPORTABLE_ RESULT	Reportable Result Flag (Y/N)	
62 – BJ	DETECT_FLAG	Analyte Detection Flag (Y/N)	
63 – BK	LAB_QUALIFIERS	Laboratory Qualifiers	
64 – BL	VALIDATOR_ QUALIFIERS	Validator Qualifiers	
65 – BM	ORGANIC_YN	Organic analysis Flag (Y/N)	
66 – BN	REPORTING_ DETECTION_ LIMIT	Adjusted Method Detection Limit (MDL)	
67 – BO	QUANTITATION_ LIMIT	Adjusted Contract Required Quantitation Limit (CRQL)	
68 – BP	DETECTION_ LIMIT_UNIT	CRQL/MDL Units	
69 – BQ	RESULT_ COMMENT	Laboratory comments regarding reported result	
70 – BR	QC_ORIGINAL_ CONC	The concentration of the analyte in the original (unspiked) sample.	
71 – BS	QC_SPIKE_ADDED	Amount of Spike Added	
72 – BT	QC_SPIKE_ MEASURED	Amount of Spike Measured	
73 – BU	QC_SPIKE RECOVERY	Spike Percent Recovery	
74 – BV	QC_DUP_ ORIGINAL_ CONC	Matrix Spike Duplicate Original Concentration	
75 – BW	QC_DUP_SPIKE_ ADDED	MSD or LCSD Added Amount	
76 – BX	QC_DUP_SPIKE_ MEASURED	MSD or LCSD Concentration	
77 – BY	QC_DUP_SPIKE_ RECOVERY	MSD or LCSD Percent Recovery	
78 – BZ	QC_RPD	MSD or LCSD RPD	
79 – CA	QC_SPIKE_LCL	Spike Lower Control Limit	

Field # -- Column	Field Name	Description	Comments
80 – CB	QC_SPIKE_UCL	Spike Upper Control Limit	
81 – CC	QC_RPD_CL	RPD Control Limit	
82 – CD	QC_SPIKE_ STATUS	Matrix Spike Result Qualifier	
83 – CE	QC_DUP_SPIKE_ STATUS	Duplicate Result Qualifier	
84 – CF	QC_RPD_STATUS	Duplicate RPD Qualifier	
85 – CG	TEST_BATCH_ TYPE	Test Batch Type	
86 – CH	TEST_BATCH_ID	Preparation Batch ID	
87 – CI	CASE	Case Number	
88 – CJ	SAMPLE_DEL_ GROUP	Sample Delivery Group (SDG) Number	
89 – CK	PH	Sample pH	

Appendix D Health and Safety Plan with Community Air Monitoring Plan

GEI Safety
EVERYONE LIVE
SAFE EVERYDAY

GEI Consultants

Consulting
Engineers and
Scientists

Appendix D
Health and Safety Plan

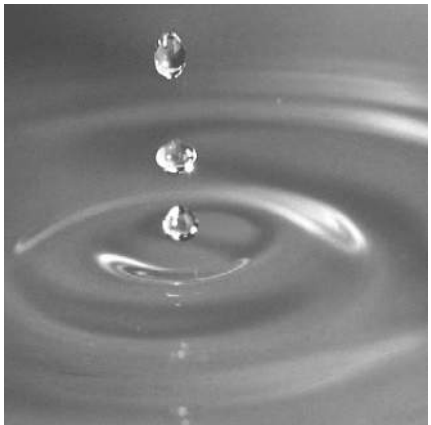
Scajaquada Creek Operable Unit RI/FS- GLLA Program
Buffalo, New York

National Fuel Gas Distribution Corporation
6363 Main St
Williamsville, New York, 14221

Prepared by:
GEI Consultants, Inc.
100 Sylvan Parkway, Suite 400
Amherst, NY, 14228
716.204.7154

February 2025

Project No.2406810



Michael A. Cummings, P.G.
Project Manager

Steven Hawkins, CSP
Safety Director

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1. Emergency Contact Information

Table 1. Emergency Contact Information

Site Information	
Project Name:	Scajaquada Creek Operable Unit
Project Address:	1660 Niagara Street, Buffalo, NY, 14207
Project Number:	2406810
Important Phone Numbers	
Emergency (Police/Fire/Medical):	911
Local Police:	911
Fire Department:	911
Hospital and Occupational Clinic Information *(See Attached Map and Directions in Appendix A)*	
Erie County Medical Center: 462 Grider St Buffalo, NY 14215 **Urgent Care/Occupational Health Clinic: Contact Medcor Triage**	(716) 898-3000 Call Medcor Triage 1-800-775-5866
Contacts	
Project Manager: Michael Cummings	(716) 204-7155 office (716) 572-4362 cell
Safety Director: Steve Hawkins	(860) 368-5348 office (860) 916-4167 cell
Regional Safety Manager: Jessie Papageorge	(973) 873-7117 office (862) 432-2283 cell
Site Safety Manager: Michael Cummings	(716) 204-7155 office (716) 572-4362 cell
Medcor Triage	1-800-775-5866
Client Contact: Tanya Alexander	(716) 857-7410 office (716) 864-5318 cell
Other Information	
Contractor Requesting/Performing Utility Clearance: TBD	Utility Clearance Ticket Number: TBD

2. GEI Safety Practices to Live By

Safety is what we do and how we do it every day. These Everyday Practices to Live Safe are simple yet concise reminders to our employees, clients, contractors, visitors of the steps that must be taken to avoid injury, illness, and incident so everyone can live safe every day. To maintain a safe work environment, GEI has established an organizational structure and a Corporate Health and Safety Program along with these safety practices.

1. Stop work if it is unsafe to continue, after any incident, injury, or near miss.

2. Prepare before starting work. Complete safety training and project-related safety requirements, such as preparation and review Health and Safety Plans (HASPs) as required by project or job-related duties.

3. Assess and control safety hazards/risks before starting any tasks and when previously unidentified safety hazards are observed.

4. Be attentive and aware of your environment. Constant focus and awareness will avoid complacency.

5. Properly use and maintain GEI-approved and required PPE in all appropriate circumstances.

6. Do not work or drive impaired, including under the influence of alcohol/drugs or while fatigued.

7. Follow all safety practices when operating a vehicle. Always wear your seatbelt while in any vehicle. Do not drive distracted, including using hand-held cell phones, when operating any vehicle.

8. Use tools, equipment, and safety devices in accordance with manufacturer's recommendations and GEI expectations. Never modify or override safety devices.

9. When activities involve chemicals or hazardous substances, follow GEI's hazard communication requirements, including labeling, reviewing Safety Data Sheets (SDS), and keeping proper protections in place.

10. Be aware of and keep clear of equipment moving in all work areas, at all times.

11. Follow GEI's incident reporting procedure in the event of safety incidents, including injuries, illness, near misses, or observation of unsafe behaviors.

3. Site Background

This Health and Safety Plan (HASP) establishes policies and procedures to protect GEI Consultants, Inc. (GEI) personnel from the potential hazards posed by the activities performed during a Remedial Investigation/Feasibility Study at the Lower Scajaquada Creek Operable Unit property (site) in Buffalo, New York (Figure 1). Reading, understanding, and compliance with the contents of the HASP is required for on-site GEI personnel and will be reviewed by GEI subcontractors. Subcontractors will prepare their own site-specific HASP but may use this HASP as a guide. This HASP identifies measures to minimize accidents and injuries which may result from site conditions or activities. A copy of this HASP will be maintained on site for the duration of the work.

3.1 Site Description

The Scajaquada Creek Operable Unit (OU) encompasses the lands adjacent to Scajaquada Creek, extending from south of the West Avenue Bridge to the creek's mouth where it discharges into the Black Rock Canal. The Scajaquada Creek Slip Area (Inlet Area), which was historically the creek's discharge location before being blocked during the construction of the I-190 New York State Thruway in the 1950s, is also part of the OU.

Upland areas include properties owned by the former Penn Central Railroad (now CSX Transportation), New York State, and City of Buffalo that are used for transportation and infrastructure corridors including a railroad passenger and freight train corridor, the NYS Thruway I-190, the Route 198 Scajaquada Expressway, and City of Buffalo roadways (West Avenue, Tonawanda Street, and Niagara Street). The upland areas also include privately owned properties, many of which were former industrial properties with environmental concerns, many of which have been investigated, and some remediated and developed for reuse in the NYSDEC Brownfield Cleanup Program (BCP) or former Voluntary Cleanup Program (VCP). Most properties in this area are zoned for commercial/industrial use.

3.2 Scope of Field Work

DNAPL associated with former MGP operations is present in the subsurface and locally in shallow sediments. Although significant data have been collected, data gaps identified in Remedial Investigation/Feasibility Study need to be addressed. The scope of the RI has been designed to fill the identified data gaps and support the preparation of feasibility study to evaluate remedial alternatives. Field activities to be performed as part of the RI include the following:

- Infrastructure assessment
- Subsurface soil investigation

- Groundwater investigation
- Sediment investigation
- DNAPL Mobility Assessment

Each of these activities is described in detail below.

- 1. Engineering Source Control and Infrastructure Assessment
    - Field reconnaissance to identify potential utility/infrastructure discharges to Scajaquada Creek that could be affecting surface water and sediment quality (e.g., combined sewer overflows and historical drain line discharges from adjacent properties).
- 2. Subsurface Soil Investigation
    - To delineate DNAPL in the subsurface soil, a series of soil borings will be completed with a drill rig in the Scajaquada OU.
- 3. Groundwater Investigation
    - Installation and sampling of groundwater monitoring wells
    - Hydraulic conductivity testing of wells.
- 4. Creek and Inlet area Sediment Investigation
    - Probing of Creek sediments to be performed from a boat, but where the water is shallow (less than 3 feet deep), or the boat cannot access the probing locations this task will be performed by wading into the creek.
- 5. DNAPL Mobility Assessments
    - Conduct NAPL mobility testing in newly-installed wells and in creek.

Table 2. GEI Employee Site Tasks and Descriptions

Task Number	Task Titles	Task Descriptions
1.	Utility and Infrastructure Assessment	Identification of infrastructure (sewer, utilities, etc.) which have potential DNAPL migration pathways, or utilities which discharge into the creek which could be affecting sediment.
2.	Subsurface Investigation	Oversight of drilling of soil borings to and through clay, soil characterization and sampling.

Task Number	Task Titles	Task Descriptions
3.	Groundwater Investigation	Oversight of monitoring well installation, well development, hydraulic conductivity testing, groundwater sampling
4.	Creek and Inlet area Sediment Investigation	Probing will occur from the barge to disturb sediment to observe characteristics and possible NAPL presence. Oversight of sediment borings with drill rig, collect sediment samples.
5.	DNAPL Mobility Assessments	Conduct NAPL mobility testing in newly-installed monitoring wells.

Notes:
Task Titles correlate to a prepared site-specific JHAs.
Task Descriptions are a brief summary of the task being performed by GEI.

4. Potential Hazards

The potential hazards associated with site conditions and activity hazards related to GEI on-site activities have been identified in this section. Detailed information for these hazards and their control methods are discussed further in the Table 3 and the job hazard analysis (JHAs) included in Appendix B.

4.1 General Site Hazards

General hazards and control measures that are applicable to all site activities have been identified in Table 3.

Table 3. General Site Hazards

General Hazards These Hazards Apply to All Site Activities	Control Measure
Cell Phone Use – On Project Site	• Look and listen for potential hazards while using a cell phone for work related activities. • Limit your cell phone use for personal calls or other activities to non-working hours in areas free of hazards. • Do not use your cell phone when safety hazards are present. • Do not use headphones or ear buds while performing work related activities. Headphone or ear bud use can prevent you from hearing hazards around you. • Noise canceling headphones or earbuds are not to be used in place of hearing protection. • Never use a cell phone while operating any equipment or vehicle. • Consider using the "do not disturb" or "silent" functions to allow you to focus on your task
Chemical/ Contaminant Exposure – Skin and eye injury/ irritation	• Wear protective coveralls (e.g. Tyvek ®) with shoe covers, safety glasses, face shield, Nitrile gloves. • Dispose of gloves after use and wash hands. • Avoid contact with pooled liquids and limit contact with contaminated soils/groundwater.

General Hazards These Hazards Apply to All Site Activities	Control Measure
Cold Stress – Hypothermia, Frostbite	• Take breaks in heated shelters when working in extremely cold temperatures. • Drink warm liquids to reduce the susceptibility to cold stress. • Wear protective clothing (recommended three layers: an outside layer to break the wind, a middle layer to provide insulation, and an inner layer of cotton of synthetic weave to allow ventilation). • Wear a hat and insulated boots. • Keep a change of dry clothing available in case clothes become wet. • Do heavy work during the warmer parts of the day and take breaks from the cold. • If possible, shield work areas from drafts of wind and use insulating material on equipment handles when temperatures are below 30°F • Watch for symptoms of cold stress.
Driving	• Employees must wear their safety belt while in a moving vehicle. • Vehicle accidents will be reported. • Vehicles will be properly maintained and safely operated. • Employees will follow safe driving behaviors, which include limiting distractions such as manipulating radios or other equipment that may cause a distraction. Employees should not exceed the posted speed limit and should maintain a safe distance between other vehicles. • Use defensive driving techniques. • Driving distance and time after a 12-hour shift should not exceed 30 miles or 30 minutes (whichever is greater). • See SOP HS-004
Dusty Conditions – Eye and respiratory irritation	• Avoid travel at extreme times • Wear protective gear – dust masks, safety glasses
Heat Stress - Fainting, Fatigue, Heat Stroke	• Increase water intake while working. • Increase number of rest breaks and/or rotate workers in shorter work shifts. Rest in cool, dry areas. • Watch for signs and symptoms of heat exhaustion and fatigue. • Plan work for early morning or evening during hot months. • Use ice vests when necessary. • In the event of heat stroke, bring the victim to a cool environment and initiate first aid procedures.

General Hazards These Hazards Apply to All Site Activities	Control Measure
Inclement Weather	• Listen to local forecasts for warnings about specific weather hazards such as tornados, thunderstorms, and flash floods. • If the storms produce thunder and/or lightning, leave the work area immediately and move to a safe area. • Discuss an action plan prior to the severe weather. • Wear appropriate PPE for the type of weather that could be encountered. • Stop work until conditions are suitable. Take cover in vehicles or shelter as appropriate. • See SOP HS-010
Insects – Bites, Stings, Allergic Reactions	• Apply insect repellent prior to performing field work and as often as needed throughout the work shift • Wear proper protective clothing (work boots, socks and light-colored clothing) • Wear shoes, long pants with bottoms tucked into boots or socks, and a long-sleeved shirt when outdoors for long periods of time, or when many insects are most active (between dawn and dusk). • When walking in wooded areas, avoid contact with bushes, tall grass, or brush as much as possible. • Field personnel who may have insect allergies should have bee sting allergy medication on site and should provide this information to the SSO and the CHSO prior to commencing work. • Field personnel should perform a self-check at the end of the day for ticks. • See SOP HS-001
Noise	• Maintain distance from the source of the noise. • Wear appropriate hearing protection when working in load environments.

General Hazards These Hazards Apply to All Site Activities	Control Measure
Physical Injury – Slips, Trips, and Falls	• Wear PPE that properly fits, is in good condition and appropriate for the activities and hazards. • Maintain good visibility of the work area. • Avoid walking on uneven, steeply sloped or debris ridden ground surfaces. • Plan tasks prior to preforming them including an activity hazard analysis. • Keep trafficked areas free from slip/trip/fall hazards. • Maintain weed growth in sampling areas, especially on slopes. • Wear shoes with traction. • Avoid traversing steep areas in slippery conditions. • Do not carry heavy objects to sampling areas, on steeply sloped areas, or where steep areas must be traversed to arrive at sample points.
Repetitive Motion Injury- Standing, Squatting, and Bending Over	• Take regular breaks and do not work in unusual positions for long periods of time. • Walk and stretch between tasks.
Sun Exposure	• Liberally apply sunscreen, with a minimum broad-spectrum sun protection factor (SPF) of 30 · Wear safety glasses that offer protection from ultraviolet A and B (UVA/UVB) rays. • Bring shade to the site to reduce exposure. • When possible, wear long-sleeved shirts and long pants. • Clothes made from tightly woven fabric and darker colors offer the best protection. Some clothing is certified as offering UV protection. • Wear a hat that has a brim all the way around that shades your face, ears, and the back of your neck. A tightly woven fabric, such as canvas, works best to protect your skin from UV rays. • Sunscreen wears off. Put it on again if you stay out in the sun for more than 2 hours. • Check the sunscreen's expiration date. Sunscreen without an expiration date has a shelf life of no more than 3 years.

General Hazards These Hazards Apply to All Site Activities	Control Measure
Unsecured or High Crime Areas	• Be aware of your surroundings. • Use the buddy system. Do not remain on site alone. Accompany or be accompanied by others to vehicles. • Request police detail when appropriate. • Let the Site Safety Manager (SSM) know when you begin work in these areas and when you leave. • Call in regularly. • If you arrive in an area and it does not look safe to get out of your vehicle, lock the doors and drive off quickly but safely.
Vehicular Traffic –Struck by injury, crushing	• Do not turn your back on traffic. • Use situational awareness to identify concerns like erratic motorist movements, sudden stops, squealing tires, or other indicators that motorists are having difficulty in maneuvering through the work zone. If observed notify the workers and suspend work until safe restart work. • Have a bail-out plan in case of an errant vehicle. Warn other workers of errant vehicles. Increase visibility of the work area to others by using cones, flags, barricades, proper lighting, and caution tape to define work area. • Use a "spotter" to locate oncoming vehicles. • Use vehicle to block work area. • Engage police detail for all work conducted in appropriate areas. • Wear high-visibility, reflective vest at all times. • Maintain minimum DOT defined distances to other traffic lanes. • See SOP HS-016.

4.1.1 Hazard Controls

On-site safety equipment to control the hazards listed above will include:

Site-Specific Safety Equipment *(check all that apply)*		
☒ Drinking water/electrolyte fluids	☒ Hand cleaner/sanitizer	☒ Tick removal kit
☒ Eye wash bottles	☒ Insect repellent	☐ Other:
☐ Fire extinguisher	☐ Phone charger	☐ Other:
☒ First aid kit	☐ Poison ivy/oak cleanser	☐ Other:
☐ Flashlight/head lamp	☒ Sunscreen	

Personal protective equipment (PPE) is discussed in further detail in Section 5.

4.1.2 Personal Safety

Field activities have the potential to take employees into areas which may pose a risk to personal safety. The following websites have been researched to identify potential crime activity in the area of the project:

- www.cityrating.com/crimestatistics.asp

Crime in Buffalo, NY is significantly above both the New York State and National averages.

Employees must not knowingly enter into a situation where there is the potential for physical and violent behaviors to occur. If employees encounter hostile individuals or a confrontation develops in the work area, suspend work activities, immediately leave the area of concern, and contact local 911 for assistance. Notify the Site Safety Manager (SSM) and Safety Team (Safety Director and Regional Safety Managers – Safety_Team@geiconsultants.com)of any incidents once you are out of potential danger.

In the event of an emergency, prompt communications with local emergency responders are essential. At least one charged and functioning cell phone to enable emergency communications will be on site. Confirmation of cellular phone operation will be confirmed at the start of each working day.

4.1.3 Vehicle Safety

Mobilization to and from the field will likely require the use of a personal, GEI fleet, or rental vehicle. When operating a vehicle while conducting business on behalf of GEI, employees must follow safe driving practices as outlined in the GEI Safe Driving SOP.

In conjunction with the hazard controls listed in the Site Mobilization JHA, GEI employees should follow these guidelines to operate a vehicle safely:

- Do not drive distracted, including using hand-held cell phones, when operating any vehicle.
- Complete a 360° inspection of the vehicle and surrounding area to identify vehicle safety issues and hazards that could be within the travel path.
- Confirm cell phones, tablets, or other potentially distracting equipment are safely secured and put away prior to operating the vehicle.
- Park in designated areas or a safe area away from heavy equipment.
- When parking a vehicle at a job site, the employee should make an effort to position the vehicle in a manner which reduces or eliminates the need to operate the vehicle in reverse.
- Use a spotter whenever possible.

GEI personnel will follow the GEI Incident Reporting (and client specific reporting) procedures if a vehicle accident occurs involving another vehicle, results in injury, or the damage to property.

4.1.4 Communicable Diseases

Communicable diseases are illnesses caused by viruses or bacteria that people spread to one another through contact with contaminated surfaces, bodily fluids, blood products, insect bites, or through the air. Examples of communicable diseases include influenza, coronavirus 2019, hepatitis B, salmonella, measles, and blood-borne illnesses. Most common forms of spread include food, insect bites, droplets, or skin contact. Infections may range in severity from asymptomatic (without symptoms) to severe and fatal. Transmission of these biologic agents can occur in a variety of ways including airborne (inhalation), direct physical contact with an infectious person, consuming contaminated foods or beverages, contact with contaminated body fluids, contact with contaminated inanimate objects, or being bitten by an infected insect or tick. Below are ways to prevent the infection or spread of communicable diseases:

1. Distancing - Maintain distance from others when possible. Minimize the number of employees in one location to the extent possible.
2. Wash Hands Often - Frequent hand washing with soap and warm water for 20 seconds. If soap and water are not readily available, use hand sanitizer (containing 60% alcohol) until soap and water can be used.
3. Clean and Disinfect Commonly Used Surfaces - Wipe down surfaces with disinfectant on a routine basis. This includes field equipment and other items that may have previously been used by others. This is especially important while working in construction trailers. When using company and personal vehicles, wipe surfaces including the steering wheel, gear shifter, controls, and door handles before and after use.
4. Use Good Hygiene Practices – These include washing hands frequently, avoid touching your eyes, nose, and mouth, and cover coughs and sneezes.
5. Get Vaccinated - Vaccines can prevent many infectious diseases. There are also vaccines that are recommended or required for travel to certain parts of the world.
6. Avoid Touching Wild Animals - Be cautious around wild animals as they can spread infectious diseases.
7. Stay home when you are sick.

4.2 Job Hazard Analysis

The site-specific tasks, potential hazards, and control measures established to reduce the risk of injury or illness are identified in step-by-step JHAs included in Appendix B. Prior to the

start of work, project team members will determine what tasks are covered in the scope of work (Table 2) and then develop a JHA for each of these tasks and have them reviewed by the Project Manager (PM) or their designee and approved by a member of the Safety Team. Indicated in each JHA are the specific PPE, training, equipment, health and safety SOPs and programs that apply to each task. Additional information on hazard controls can be found in GEI's SOPs and programs that apply to this project which are indicated in Appendix E.

4.3 Heat and Cold Stress

4.3.1 Heat Stress

Employees may be exposed to the hazards associated with heat stress when ambient temperatures exceed 80ºF. To prevent heat-related illness, Project Managers (PMs) should plan for proper hydration (drinking plenty of water), acclimatization (getting used to weather conditions), and schedules that alternate work with rest. Employees should also be trained to recognize the symptoms of heat related illnesses and know how to administer first aid for heat-related illnesses and activate emergency medical services quickly when needed. Water and shade will be available to all project employees and located as close as practicable to the work areas when temperatures exceed 80°F.

Prior to each workday, the forecasted temperature and humidity for the worksite will be reviewed and will be compared against the National Weather Service Heat Index to evaluate the risk level for heat illness. When the temperature equals or exceeds 95°F, or during a heat wave, high heat procedures will be used which include additional preventive measures including pre-shift meetings to encourage employees to drink plenty of water, working in the buddy system or regular communication so observations can be made for heat related illness, and to remind employees of their right to take a cool-down rest when necessary.

CDC Heat Planning Tool
For the most recent details and tools for heat stress, use your smart phone to access the Centers for Disease Control for the latest information. Additional details can be found in GEI's Heat Stress Program located on the Safety Resources page of GEI Connections.

4.3.2 Cold Stress

Employees may be exposed to the hazards associated with cold stress when working in cold, wet, and/or windy conditions. Potential hazards in cold environments include frostbite, trench foot or immersion foot, hypothermia, as well as slippery surfaces, brittle equipment, and poor judgment.

CDC Cold Stress Info
For the most recent details and tools for cold stress, use your smart phone to access the Centers for Disease Control for the latest information. Additional details can be found in GEI's Cold Stress Program located on the Safety Resources page of GEI Connections.

4.4 Constituents of Concern

The characteristics of constituents of concern (COC) at the site are discussed below for safety information purposes. A COC is any substance classified or defined as hazardous, extremely hazardous, toxic, or dangerous. The COC included in this health and safety plan will be the primary constituents that have been detected, are anticipated to be detected, or are being evaluated for the presence of, on the project site. These COC will be used to determine the action levels and PPE necessary for site personnel. Adherence to the safety and health guidelines in this HASP should reduce the potential for exposure to the COC discussed below.

4.4.1 Site-Specific COC

4.4.1.1 Volatile Organic Compounds

Volatile organic chemicals (VOCs), such as benzene, toluene, ethyl benzene, and xylene (BTEX) are commonly present as soil and groundwater contaminants, and in some cases chemical components in non-aqueous phase liquids (NAPL) such as oil or tar within soils and abandoned pipelines. These compounds are not expected to be at concentrations that exposure symptoms would occur. These compounds generally have a depressant effect on the Central Nervous System (CNS), may cause chronic liver and kidney damage, and some are suspected human carcinogens. Benzene is a known human carcinogen. Acute exposure may include headache, dizziness, nausea, and skin and eye irritation. The primary routes of exposure to VOCs are through inhalation and direct contact, therefore air monitoring, gloves, and respiratory protection are the primary controls against exposure to VOCs.

4.4.1.2 Semi-Volatile Organic Compounds

Semi-volatile organic compounds (SVOCs) usually consist of a mixture of acenaphthene, acenaphthylene, anthracene, benz(a)anthracene, benzo(b)fluoranthene, benzo(k)fluorethene, benz(a)pyrene, benzo(e)pyrene, benzo(g,h,i)peryline, chrysene, dibenz(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3cd)pyrene, 2-methyl naphthalene, naphththalene, phenanthrene, phenols, and pyrene.

SVOCs are not expected to be present at concentrations that exposure symptoms would occur. SVOCs such as those listed above may cause contact dermatitis. Direct contact can be irritating to the skin and produce itching, burning, swelling, and redness. Direct contact or exposure to the vapors may be irritating to the eyes. Conjunctivitis may result from prolonged exposure. Many SVOCs are considered to be very toxic, if ingested. High levels of exposure to SVOCs, though not anticipated during work activities conducted during this project, may increase the risk of cancer including lung, kidney, and skin cancer. Naphthalene is also an eye and skin irritant and can cause nausea, headache, fever, anemia, liver damage,

vomiting, convulsions, and coma. Poisoning may occur by ingestion of large doses, inhalation, or skin absorption.

The primary routes of exposure to SVOCs are through inhalation and direct contact, therefore air monitoring, gloves, and respiratory protection are primary controls against exposure to SVOCs.

4.4.1.3 Coal Tar and Coal Tar Products

Coal tar products, which are semi-volatile organic compounds (SVOCs) consist of a mixture of acenaphthene, acenaphthylene, anthracene, benz(a)anthracene, benzo(b)fluoranthene, benzo(k)fluorethene, benz(a)pyrene, benzo(e)pyrene, benzo(g,h,i)peryline, chrysene, dibenz(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3cd)pyrene, 2-methyl naphthalene, naphththalene, phenanthrene, phenols, pyrene.

Coal tar products and other SVOCs are present at the Site within impacted soil and groundwater and as a dense non-aqueous phase liquid (DNAPL) by-product of gas production within soils, former manufactured gas plant (MGP) structures, and abandoned pipelines.

Coal tar products such as those listed above may cause contact dermatitis. Direct contact can be irritating to the skin and produce itching, burning, swelling, and redness. Direct contact or exposure to the vapors may be irritating to the eyes. Conjunctivitis may result from prolonged exposure. Coal tar is considered to be very toxic, if ingested. High levels of exposure to coal tar, though not anticipated during work activities conducted during this project, may increase the risk of cancer including lung, kidney, and skin cancer. Naphthalene is also an eye and skin irritant and can cause nausea, headache, fever, anemia, liver damage, vomiting, convulsions, and coma. Poisoning may occur by ingestion of large doses, inhalation, or skin absorption.

The major route of entry for the work activities to be conducted at this Site is through direct contact. Exposure is most likely when handling soil and water samples. Inhalation may occur when the soil is disturbed causing respirable and nuisance dust particles to become airborne.

4.4.1.4 Heavy Metals

This Site may contain elevated levels of metals including arsenic, copper, iron, manganese, mercury, selenium, and zinc.

Exposure to high concentrations of arsenic can cause dermatitis, gastrointestinal disturbances, peripheral neuropathy, respiratory irritation, and hyper pigmentation of skin. Chronic exposure to arsenic has resulted in lung cancer in humans.

Exposure to high concentrations of copper through inhalation can cause irritation of the eyes, nose, pharynx, nasal septum. Ingestion may cause a metallic taste. Skin irritation may result from direct contact with skin. Damage to the liver and kidneys may occur.

No adverse health effects are associated with environmental exposure to iron. Target organs for iron via ingestion of iron (most often in supplement form) are the liver, cardiovascular system, and kidneys. Exposure to high concentrations of iron through ingestion can cause salivation nausea, vomiting, diarrhea, and abdominal pain.

Exposure to high concentrations of manganese can cause manganism, metal fume fever, flu-like fever, and kidney damage.

Exposure to mercury can cause dizziness, salivation nausea, vomiting, diarrhea, constipation, emotional disturbance, and kidney injury. Chronic exposure to mercury can cause CNS damage.

Exposure to high concentrations of selenium can cause mucous membrane irritation, coughing, sneezing, shortness of breath, chills, headaches, hypotension, and CNS depression. Chronic exposure to selenium could cause bronchial irritation, gastrointestinal distress, excessive fatigue, and skin discoloration.

Exposure to high concentrations of zinc through ingestion can cause abdominal pain, nausea, vomiting, and diarrhea. Chronic exposure can lead to low blood pressure, jaundice, and seizures.

Heavy metals are not expected to be present at concentrations that exposure symptoms would occur. The primary route of exposure is through inhalation of dust particles when soil is disturbed and becomes airborne, therefore, particulate monitoring, respiratory protection, and gloves are primary controls against exposure to heavy metals.

4.4.1.5 Cyanide

Cyanide compounds are common by-products of manufactured gas plant (MGP) production. Hydrogen cyanide is toxic because it is a chemical asphyxiate. It replaces the oxygen in the blood and thereby suffocates the cells. Ferro cyanides are not considered toxic because the hydrogen cyanide ion is bound too tightly to the iron and cannot therefore replace the oxygen. It takes a great amount of heat and/or acid to release cyanide gas from the ferro cyanide molecule; therefore, hydrogen cyanide is not a concern at this Site.

4.4.1.6 Hydrogen Sulfide

Hydrogen sulfide is a common by-product of manufactured gas plant (MGP) production. Exposure to lower concentrations can result in eye irritation, a sore throat and cough, shortness of breath, and fluid in the lungs. These symptoms usually go away in a few weeks.

Long-term, low-level exposure may result in fatigue, loss of appetite, headaches, irritability, poor memory, and dizziness. Breathing very high levels (> 800 parts per million [ppm]) of hydrogen sulfide can cause death within just a few breaths. The primary route of exposure is through inhalation and therefore respiratory protection is the primary control against exposure to hydrogen sulfide.

Table 4. Primary Constituents of Concern Data

Constituent	Exposure Limit(s)	Route of Exposure	Primary Hazard/ Symptoms of Exposure
(Link to COC Data Table) If a COC for your site is not included in this table, use The CDC NIOSH Guide			
Arsenic	0.01 mg/m3 A.L. 0.005mg/m3	Inhalation, skin absorption, ingestion, skin contact	Ulceration of nasal septum, dermatitis, GI disturbances, peripheral neuropathy, respiratory irritation, hyperpigmentation of skin, potential carcinogen
Benzene	1 ppm TWA 5 ppm STEL	Inhalation, skin absorption, ingestion, skin contact	Irritation of eyes, skin, nose, respiratory system, giddiness, headache, nausea; staggering gait, fatigue, anorexia, weakness, dermatitis, bone marrow depression, potential carcinogen
Ethylbenzene	100 ppm	Inhalation, ingestion, skin contact	Eye, skin, mucous membrane irritation; headache; dermatitis, narcosis; coma
Hydrogen sulfide (H2S)	20 ppm C 50 ppm [10-min. Maximum peak]	Inhalation, skin/eye contact	Irritation eyes, respiratory system; apnea, coma, convulsions; conjunctivitis, eye pain, lacrimation (discharge of tears), photophobia (abnormal visual intolerance to light), corneal vesiculation; dizziness, headache, fatigue, irritability, insomnia; gastrointestinal disturbance; liquid: frostbite
Iron	Iron oxide dust and fume: 10 mg/m3	Inhalation, ingestion, eye contact	Respiratory tract irritation, coughing, shortness of breath, overdose of iron may cause vomiting, abdominal pain, bloody diarrhea, vomiting blood, lethargy, and shock; acidity in the blood, bluish skin discoloration, fever, liver damage, and possibly death; eye and cornea irritation and discoloration
Manganese	C 5 mg/m3	Inhalation, ingestion	Manganism; asthenia, insomnia, mental confusion; metal fume fever: dry throat, cough, chest tightness, dyspnea (breathing difficulty), rales, flu-like fever; low-back pain; vomiting; malaise (vague feeling of discomfort); lassitude (weakness, exhaustion); kidney damage

Constituent	Exposure Limit(s)	Route of Exposure	Primary Hazard/ Symptoms of Exposure
(Link to COC Data Table) If a COC for your site is not included in this table, use The CDC NIOSH Guide			
Mercury	0.10 mg/m3	Inhalation, ingestion, skin contact/absorption	Irritates eyes and skin, chest pain, cough, difficulty breathing, bronchitis, pneumonitis, tremor, insomnia, irritability, indecision, headache, fatigue, weakness, stomatitis, salivation, Gastrointestinal disturbance, weight loss, proteinuria
Naphthalene	10 ppm (50 mg/m3) TW	inhalation, skin absorption, ingestion, skin and/or eye contact	Irritation eyes; headache, confusion, excitement, malaise (vague feeling of discomfort); nausea, vomiting, abdominal pain; irritation bladder; profuse sweating; jaundice; hematuria (blood in the urine), renal shutdown; dermatitis, optical neuritis, corneal damage
Selenium	0.2 mg/m3	Inhalation, ingestion, skin contact	Irritant to eyes, skin, nose and throat, visual disturbance, headache, chills, fever, breathing difficulty, bronchitis, metallic taste, garlic breath, GI disturbance, dermatitis, eye, and skin burns
Toluene	200 ppm	Inhalation, skin absorption, ingestion, skin contact	Eye, nose irritation; fatigue, weakness, confusion, euphoria, dizziness, headache; dilated pupils, tearing of eyes; nervousness, muscle fatigue, insomnia, tingling in limbs; dermatitis
Xylene	100 ppm	Inhalation, skin absorption, ingestion, skin contact	Eye, skin, nose, throat irritation; dizziness, excitement, drowsiness; incoordination, staggering gait; corneal damage; appetite loss, nausea, vomiting, abdominal pain; dermatitis
Zinc	TWA 15 mg/m3 (total dust) TWA 5 mg/m3 (resp dust	Inhalation	Metal fume fever: chills, muscle ache, nausea, fever, dry throat, cough; lassitude (weakness, exhaustion); metallic taste; headache; blurred vision; low back pain; vomiting; malaise (vague feeling of discomfort); chest tightness; dyspnea (breathing difficulty), rales, decreased pulmonary function

Notes:
A.L. - Action Level
C - ceiling limit, not to be exceeded
Ca – carcinogen
f/cc - fibers per cubic centimeter
mg/m^3 - micrograms per cubic meter
mppcf - millions of particulates per cubic foot of air
ppm - parts per million
STEL - Short-term exposure limit (15 minutes)

TWA - Time-weighted average (8 hours)

4.4.2 Chemicals Brought on Site

Potential hazards associated with chemicals brought on site (e.g., decontamination chemicals, sample preservatives, fuels, calibration fluids) for the work will be mitigated through training, administrative controls (e.g., proper labeling and storage), and proper use of PPE. Safety data sheets (SDSs) for all chemicals brought on site shall be maintained by the SSM and are included in Appendix C.

5. Personal Protective Equipment

The PPE required to be worn on the project site is listed in the table below. Additional PPE required for the tasks to be performed is listed on the JHAs in Appendix B.

Site Required PPE *(check all that apply)*		
☒ Hard Hat	☐ Respirator	☒ Tyvek clothing/boots
☒ Safety Glasses	☐ Flame Resistant Clothing	☒ Hearing Protection
☒ Safety Boots	☒ Personal Flotation Device	☐ Long Sleeve Shirt
☒ Gloves (task specific)	☐ Snake Chaps	☐ Other:
☒ High Visibility Safety Vest	☐ EH-Rated Boots	☐ Other:

If site conditions suggest the existence of a situation more hazardous than anticipated, the site personnel will evacuate the area to a safe distance. The hazard, the level of precautions, and the PPE will then be reevaluated with the assistance and approval of the Safety Director and the PM. If conditions indicating the need for Level A or Level B PPE are encountered, personnel will leave the site and notify the PM or a member of the Safety Team. GEI's PPE Program can be found on the Safety Resources page of GEI Connections.

5.1 Respiratory PPE

GEI personnel who have the potential to don a respirator must have a valid fit test certification and medical clearance. Both the respirator and cartridges specified for use in Level C protection must be fit-tested prior to use in accordance with OSHA regulations (29 CFR 1910.134). Air purifying respirators cannot be worn under the following conditions:

- Oxygen deficiency (less than 20.7%).
- Imminent Danger to Life and Health (IDLH) concentrations.
- If contaminant levels exceed designated use concentrations.

Upgrades to respiratory protection may be required based on the designated Action Levels found in Section 9.

6. Responsibilities and Lines of Authority

6.1 GEI Personnel Responsibilities

The implementation of health and safety at this project location will be the shared responsibility of the Safety Director, Regional Safety Director, PM, the Site Safety Manager (SSM), and each GEI personnel implementing the proposed scope of work.

6.1.1 GEI Safety Director

The Safety Director is responsible for the overall management of GEI's safety programs, policies, and procedures. Modifications to this HASP which may result in the reduction in the identification, evaluation, and control of safety and health hazards cannot be undertaken by the project team without the approval of the Safety Director.

6.1.2 GEI Project Manager

The PM is responsible for confirming that the requirements of this HASP are implemented. The PM's specific responsibilities include:

- Conducting and documenting the Project Safety Briefing.
- Verifying that the GEI staff and subcontractors selected to work on this program are sufficiently trained for site activities and have reviewed this HASP.
- Maintaining regular communications with the SSM and, if necessary, the Safety Director.

6.1.3 GEI Regional Safety Manager

The RSM is responsible for supporting the safety needs and requirements specified in this HASP. The RSM's specific responsibilities include:

- Reviewing and approving the HASP and applicable JHAs.
- Working with the PM and SSM to meet client safety requirements.
- Providing approval for fall protection plans and confined space entries (permit numbers), as applicable.
- Providing safety support regarding safety programs and procedures as applicable to the project.

6.1.4 GEI Site Safety Manager

The SSM is responsible for implementing and enforcing the safety requirements specified in this HASP and will be on-site during activities covered in the HASP. The SSM's specific responsibilities include:

- Enforcing the requirements of this HASP and notify the PM of noncompliance.
- Conduct daily Safety Tailgate meetings for site-related work.
- Maintaining a high level of health and safety consciousness among employees implementing the proposed activities.
- Procuring the air monitoring instrumentation, PPE, and safety equipment needed for GEI project employees and verifying that each is in good working order.
- Verifying that GEI subcontractors are utilizing the correct PPE and safety equipment.

6.1.5 All GEI Field Personnel

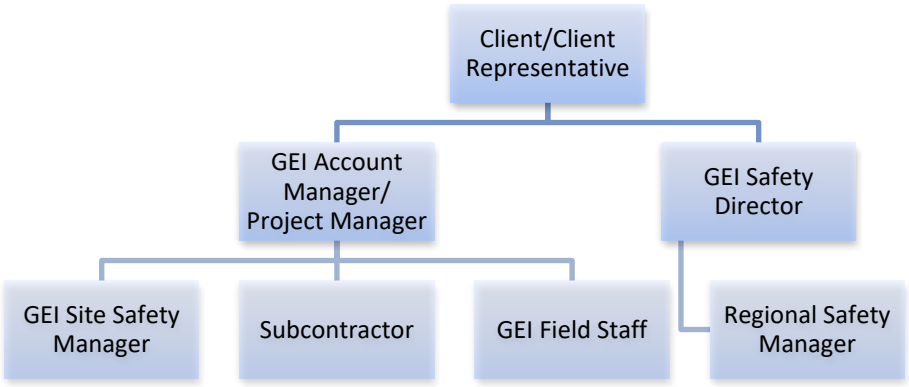
All GEI field personnel (including the PM and SSM) covered by this HASP are responsible for following the health and safety procedures in this HASP and for performing their work in a safe and responsible manner. The specific responsibilities that apply to all field personnel include:

- Reading and signing the HASP prior to the start of on-site work.
- Bringing forth any questions or concerns regarding the content of the HASP to the PM or the SSM.
- Attending and actively participating in the required Project Safety Briefing prior to beginning on-site work and any subsequent safety meetings.
- Complying with the requirements of this HASP and the requests of the SSM.
- Stopping work in the event that an immediate danger situation is perceived.
- Reporting accidents, injuries, and illnesses, regardless of their severity by following GEI's incident reporting procedures.

6.2 Lines of Authority

GEI will have responsibility for safety of its employees during the work performed at the site. GEI's SSM will have a cell phone available to contact the appropriate local authorities, in the event of an emergency. GEI's SSM will be available for communication with the GEI PM and with the Client's representative.

Project Lines of Authority



6.2.1 Stop Work Authority

GEI employees have the authority to stop work activities if an unanticipated hazard is encountered or a potential unsafe condition is observed. The GEI employee should contact the Safety Director and the Project Manager to discuss the stop work conditions and potential control methods that can be implemented.

6.3 Subcontractors

GEI has subcontracted the following firms to assist in performing work on this project:

Subcontractor Information	
Company Name/Address: TBD	
Contact Name:	Cell: (XXX) XXX-XXXX
Scope of Work:	

GEI requires its subcontractors to work in a responsible and safe manner. Subcontractors hired by GEI are required to submit documentation of their safety practices as part of GEI's Subcontractor Safety Prequalification for evaluation and approval before the start of work. Subcontractors for this project will be required to develop their own HASP for protection of their employees, but, at a minimum, must adhere to applicable requirements set forth in this HASP. The PM will obtain applicable safety certifications and training records from the subcontractor's site supervisor prior to the initiation of work.

7. Training Requirements

Prior to commencement of field activities, the PM or their designee will verify GEI field personnel assigned to the project will have completed training that will specifically address the activities, procedures, monitoring, and equipment used in the site operations. This training will be documented on the applicable JHAs (Appendix B). Personnel that have not received project-specific training will not be allowed on site.

Applicable Site-Specific Training Requirements *(check all that apply)*		
✔ HAZWOPER (8Hr Refresher)	☐ Railroad Specific Training	☐ Other:
☐ HAZWOPER (Site Supervisor)	☐ Transportation Worker Identification Credential (TWIC)	☐ Other:
✔ First Aid/CPR	☐ MSHA (Mine Safety and Health Administration)	☐ Other:

7.1 On-Site Safety Briefings

GEI personnel will be given health and safety briefings daily (or as frequently as needed) by the SSM or their designee to plan for conducting work activities safely. The briefing will include GEI subcontractors and others as appropriate. The briefings can include information on:

- Applicable JHAs
- Changes in work practices
- Changes in environmental conditions
- Anticipated weather
- Evacuation/emergency procedures
- Air monitoring results
- Safety inspection results

Documentation of these briefings will be recorded in the GEI field book or the Tailgate Safety Briefing Form (Appendix D). For long-term projects, the Tailgate Safety Briefing Form is preferred.

8. Medical Surveillance Program

GEI maintains a medical surveillance program under the supervision of the Safety Director that includes a plan designed specifically for field personnel engaged in work at sites where hazardous materials may be present. Field personnel undergo an initial physical examination, including a detailed medical and occupational history before they are able to engage in work at hazardous waste sites. Upon successful completion of the examination, personnel are provided a medical clearance from an occupational health physician stating their fitness to perform the specified work activities. Employees who are part of this program will schedule and attend annual exams 12 months from the date of their previous exam.

If a GEI employee or other project worker shows symptoms of exposure to a hazardous substance and wishes to be seen by a doctor, GEI will consult with their third-party medical administrator and provide access to the nearest area hospital or medical facility.

GEI subcontractor personnel that will enter any hazardous waste sites must certify that they are participating in a medical surveillance program that complies with OSHA regulations for hazardous waste operations (i.e., 29 CFR 1910.120 and 29 CFR 1926.65). A copy of their medical clearance will be submitted to the GEI PM or SSM prior to the start of field activities.

9. Personal/Work Zone Air Monitoring

Personal/Work Zone Air Monitoring Required?	
☒ Yes	☐ No

Air monitoring, in the form of personal or work zone monitoring, will be performed to identify and quantify airborne levels of hazardous substances and atmospheric hazards to determine the appropriate level of worker protection needed on-site. A Community Air Monitoring Plan (CAMP) which describes air quality monitoring for receptors at the Site perimeter (outside the work zone) is provided as Appendix A.

Work on this project requiring air monitoring includes:

Personal/Work Zone Air Monitoring Tasks *(check all that apply)*		
☒ Excavation	☐ Confined Space Entry	☐ Other:
☒ Soil Sampling	☐ Indoor Drilling	☐ Other:
☒ GW Monitoring Well Headspace	☒ Product Sampling	☐ Other:

The following air monitoring equipment will be on site:

Personal/Work Zone Air Monitoring Equipment *(check all that apply)*	
☒ PID with 10.6 eV lamp or equivalent	☐ Particulate Meter (PM-10 capable)
☐ Drager Chip Measurement System (CMS) with appropriate gas detection chips	☐ Multi-gas meter: lower explosive limit (LEL) / oxygen (O_2) / hydrogen sulfide (H_2S) / hydrogen cyanide (HCN) or carbon monoxide (CO) meter
☐ Sensidyne Gas Detection Pump with appropriate gas detector tubes (or equivalent colorimetric tube)	☒ Other: Dust Trak DRX (upwind and downwind locations) to evaluate PM10 and TSP concentrations at the Site perimeter as part of the CAMP

GEI will conduct and document on-site work zone monitoring and will inform GEI employees of the results. ***If Action Levels are exceeded, immediately implement site action(s) according to Action Table below and notify the PM***.

9.1 Calibration

Air monitoring equipment will be calibrated and maintained in accordance with manufacturer's requirements. Calibrations will be recorded in the project notes daily or on a daily calibration form.

9.2 Action Levels

The tables below provide a summary of real time air monitoring Action Levels and contingency plans for work zone activities. The below Action Levels are determined by halving the Permissible Exposure Limits (PELs) or Threshold Limit Values (TLVs) as set forth by OSHA and the American Conference of Government Industrial Hygienists (ACGIH).

9.2.1 VOC Monitoring and Control

Air monitoring reduces the risk of overexposure by indicating when action levels have been exceeded and when PPE must be upgraded or changed. Based on the volatile organic compounds (VOCs) listed in Table 4, determine which constituent has the lowest permissible exposure limit (PEL). This data is used to determine the action levels needed including respiratory protection at the project site. GEI's action level is half of the PEL listed in Table 4 (exception is made for benzene).

Exposure to organic COC can be evaluated and/or controlled by:

- Monitoring worker breathing zone atmospheric concentrations for organic COC in the breathing zone with a photoionization detector (PID).
- When possible, engineering control measures will be utilized to suppress the volatile organic vapors. Engineering methods can include utilizing a fan to promote air circulation, utilizing volatile suppressant foam, providing artificial ground cover, or covering up the impacted material with a tarp to mitigate volatile odors.
- When volatile suppression engineering controls are not effective and organic vapor meters indicate concentrations above the action levels, then appropriate respiratory protection (i.e., air purifying respirator with organic vapor cartridge) will be employed.

Air Monitoring Instrument	Action Level (above background)	Site Action
Action Levels for the following parameters are 15-minute time weighted averages (TWA), not a single exceedance.		
PID (Monitoring for VOCs)	0.0 – 50 ppm	No respiratory protection is required if VOCs are not present. (If benzene or naphthalene are constituents of interest at this site, follow the action levels below for benzene and/or naphthalene.)
	> 50 ppm	Stop work, withdrawal from work area, institute engineering controls, if levels persist, upgrade to Level C. Notify PM and Safety Team.
PID	0.0 - 1.0 ppm	No respiratory protection is required if benzene is not present. Use detector tube for benzene to verify if concentration of 1.0 ppm or greater is benzene.

Air Monitoring Instrument	Action Level (above background)	Site Action
(Monitoring for benzene)	1.0 – 50 ppm	If benzene is present (confirmation via detector tube), stop work and contact your PM. If work continues and benzene is present, or no detector tubes are used to determine presence, upgrade to Level C. No respiratory protection is required if benzene is not present.
	> 50 ppm	Stop work, withdraw from work area, notify PM and Safety Team.
PID (Monitoring for naphthalene)	0.0 - 5 ppm	No respiratory protection is required if naphthalene is not present. Use Sensodyne detector tube for naphthalene to verify if concentration of 5 ppm or greater is naphthalene.
	5 – 50 ppm	If naphthalene is present (confirmation via detector tube), stop work and contact your PM. If work continues and naphthalene is present or no detector tubes are used to determine presence, upgrade to Level C. No respiratory protection is required if naphthalene is not present.
	> 50 ppm	Stop work, withdraw from work area, notify PM and Safety Team.

9.2.2 Dust Monitoring and Control

Some COC hazards may become hazardous when they are associated with dust/particles and become airborne. For worker safety, dust levels must be managed to eliminate this hazard. Dust generated during activities can cause irritation to the respiratory system and eyes. Contaminants can also be carried in airborne dust causing potential exposure to workers through skin contact and inhalation. Constituent concentrations on site are expected to be low therefore the exposure hazard through inhalation should be minimal; however, contaminant contact through skin and clothing can introduce additional exposures.

For dust generated during site activities which exceed site-specific limits, engineering controls such as water application will be used to control dust concentrations. However, if excessive dust concentrations cannot to be handled through engineering controls, then respirators will be required to be worn.

Air Monitoring Instrument	Action Level (above background)	Site Action
Action Levels for the following parameters are 15-minute time weighted averages (TWA), not a single exceedance.		
Particulate Meter	150 µg/m^3	Implement work practices to reduce/minimize airborne dust generation, e.g., spray/misting of soil with water. Don respirator with particulate filters if action levels remain in exceedance.

10. Site Control

10.1 Site Zones

Site zones are intended to control the potential spread of contamination and to assure that only authorized individuals are permitted into potentially hazardous areas. This project is being conducted under the requirements of 29 CFR 1910.120, and any personnel working in an area where the potential for exposure to site contaminants exists, will only be allowed access after proper training and medical documentation.

10.2 Buddy System

GEI personnel should be in line-of-site or communication contact with another on-site person. The other on-site person should be aware of his or her role as a "buddy" and be able to help in the event of an emergency. Some projects may not support the need for the buddy system to be implemented. If this is the case, the PM is required to conduct regular check-ins with the employee on site.

10.3 Sanitation for Temporary Work Sites

Sanitation requirements identified in the OSHA Standard 29 CFR 1926.51 "Sanitation" specifies that employees working at temporary project sites have at least one sanitary facility available to them.

10.4 Illumination

Illumination requirements identified by OSHA are directed to work efforts inside buildings and/or during non-daylight hours. Activities planned for the site are anticipated to occur outside during daylight hours. However, if work areas do not meet illumination requirements, they will be equipped with appropriate illumination that meets or exceeds requirements specified in OSHA Standard 29 CFR 1926.56. Employees will not work on sites that are not properly lighted.

10.5 Smoking

Smoking is prohibited at or in the vicinity of hazardous operations or materials. Where smoking is permitted, safe receptacles will be provided for smoking materials.

10.6 Alcohol and Drug Abuse Prevention

Alcohol and drugs will not be allowed on the site. Project personnel under the influence of alcohol or drugs will not be allowed to enter the site. All GEI employees must comply with

GEI's Controlled Substance Use & Alcohol Misuse Policy found on the Safety Resources page of GEI Connections. Employees may be subject to random drug and/or alcohol testing if required by a client at a project site.

11. Incident Reporting

GEI will report incidents involving GEI personnel or subcontractor personnel, such as: lost time injuries, injuries requiring medical attention, near miss incidents, fires, fatalities, accidents involving the public, chemical spills, vehicle accidents, and property damage. The following steps must be followed when an incident occurs:

1. For incidents involving life-threatening situations or serious injury that require emergency response personnel (Police, Fire, EMS), call 9-1-1 from a safe area.
2. **Stop work** activity to address any injury, illness, property damage, spill, or other emergency.
3. Call Medcor Triage at 1-800-775-5866 to speak with a medical professional following any injury or illness.
4. Notify your Supervisor/Project Manager of the incident or injury.
5. Complete an incident report using the GEI Incident Report Form located on the GEI Safety Smartphone App, GEI Connections intranet page, or in the project HASP.
6. Resume work activity if all steps above have been completed and it is safe to do so.

For vehicle accidents involving another vehicle or damage to property, the employee will take pictures of each vehicle or property involved in the incident and obtain a police report. In some municipalities police will not be dispatched to a non-injury accident, but every effort needs to be made to try and obtain the report.

The Incident Report Form and the Near Miss Reporting Form can be found in Appendix D, on the GEI Health and Safety smartphone app, or on the Safety Resources page of GEI Connections. To report subcontractor injuries or incidents, follow the same verbal reporting procedures and submit an email describing the event to the PM and the Safety Team.

11.1 Injury Triage Service

If a GEI employee experiences a work-related injury that is not life-threatening, the employee will initiate a call to Medcor Triage at 1-800-775-5866. The injured employee will detail any medical symptoms or complaints which will be evaluated by a Registered Nurse (RN) specially trained to perform telephonic triage. The RN will recommend first aid self-treatment or refer the injured employee for an off-site medical evaluation by a health professional at a clinic within GEI's workers compensation provider network. GEI employees are still required to follow our Accident Reporting procedures as listed above.

12. Decontamination Procedures

Site decontamination procedures are designed to achieve an orderly, controlled removal or neutralization of contaminants that may come in contact with personnel or equipment. These procedures minimize contact with contaminants and protect against the transfer of contaminants to clean areas. They also extend the useful life of PPE by reducing the amount of time that contaminants contact and can permeate PPE surfaces. This project is being conducted under the requirements of 29 CFR 1910.120(k), and any personnel or equipment that are exposed to site contaminants will follow applicable decontamination procedures.

12.1 Personnel and PPE Decontamination

Contaminated PPE (gloves, suits, etc.) will be decontaminated and stored for reuse or placed in plastic bags (or other appropriate containers) and disposed of in an approved facility. Decontamination wastewater and used cleaning fluids will be collected and disposed of in accordance with applicable state and federal regulations.

12.2 Equipment Decontamination

All tools, equipment, and machinery that have come into contact with contaminated media, will be decontaminated on site prior to departure. Equipment decontamination procedures are designed to minimize the potential for hazardous skin or inhalation exposure and to avoid cross-contamination and chemical incompatibilities.

13. Emergency Response

13.1 Evacuation

Prior to the start of work, emergency procedures must be identified and communicated to workers on site. This includes evacuation routes, safe areas, and/or muster points (Figure 1). Also communicate how employees will be notified that an emergency or evacuation of the site is occurring (audio alarms, visible (light) alarms, radios, sirens, etc.) Upon discovering an emergency situation, personnel will notify the SSM, who will initiate an appropriate response. Once the scene is safe, use the incident report procedures to report the evacuation to the PM and Safety Team.

13.2 Fire

In the event of a fire personnel will evacuate the area to the muster point located on Figure 1. GEI's SSM will contact the local fire department and report the fire. The SSM will account for GEI personnel and subcontractor personnel and report their status to the PM. Incident reporting procedures will be followed once the scene is safe.

13.3 Spills or Material Release

If a hazardous waste spill or material release occurs, if safe, the SSM or their representative will immediately assess the magnitude and potential seriousness of the spill or release based on the following:

- SDS for the material spilled or released.
- Source of the release or spillage of hazardous material.
- An estimate of the quantity released and the rate at which it is being released.
- The direction in which the spill or air release is moving.
- Personnel who may be or may have been in contact with the material, or air release, and possible injury or sickness as a result.
- Potential for fire and/or explosion resulting from the situation; and
- Estimates of area under influence of release.

If the spill or release is determined to be within the on-site emergency response capabilities, the SSM will verify implementation of the necessary remedial action. If the release is beyond the capabilities of the site personnel, personnel will be evacuated from the immediate area and the fire department will be contacted. The SSM will notify the PM and follow the incident reporting procedures.

13.4 Medical Support

In case of minor injuries, on-site care will be administered with the site first aid kit. A GEI employee certified by the American Red Cross or other American Health & Safety Institute (ASHI) will be on-site at all times. For serious injuries, call 911 and request emergency medical assistance. Seriously injured persons should not be moved unless they are in immediate danger. Notify the PM of the emergency and follow incident reporting procedures.

In the event of an emergency, prompt communications with local emergency responders are essential. At least one charged and functioning cell phone to enable emergency communications will be on site. Confirmation of cellular phone operation will be confirmed at the start of each working day.

Table 1 of this HASP contains detailed emergency information, including directions to the nearest hospital, and a list of emergency services and their telephone numbers. In addition, Appendix A includes a map to the local hospital/emergency room and Figure 1 indicates the evacuation route (including muster point).

13.5 Severe Weather

The contingency plan for severe weather includes reviewing the expected weather to determine if severe weather is in the forecast. Severe weather includes high winds over 40 miles per hour (mph), heavy rains or snow squalls, thunderstorms, tornados, and lightning storms. If severe weather is approaching, the decision to evacuate GEI personnel and subcontractor personnel from the site will be the responsibility of GEI's SSM. Notification of evacuation will be made to the PM. The SSM will account for GEI personnel and subcontractor personnel and report their status to the PM. If safe, work can resume 30 minutes after the last clap of thunder or flash of lightning.

13.6 Hazard Communication Plan

GEI personnel have received hazard communication (HAZCOM) training as part of their annual safety training and new employee safety orientation training. Hazardous materials brought on site will be properly labeled, stored, and handled. SDSs for each chemical will be included in this HASP in Appendix C. GEI's HAZCOM program can be found on the Safety Resources page of GEI Connections (Appendix E).

14. Health and Safety Plan Sign-Off

GEI personnel conducting site activities will be familiar with the information in this HASP. After reviewing this plan, please sign the copy in the project files, and bring a copy of the plan with you to the site. By signing this site-specific HASP, you are agreeing that you have read, understand, and will adhere to the provisions described in this plan while working on the site below.

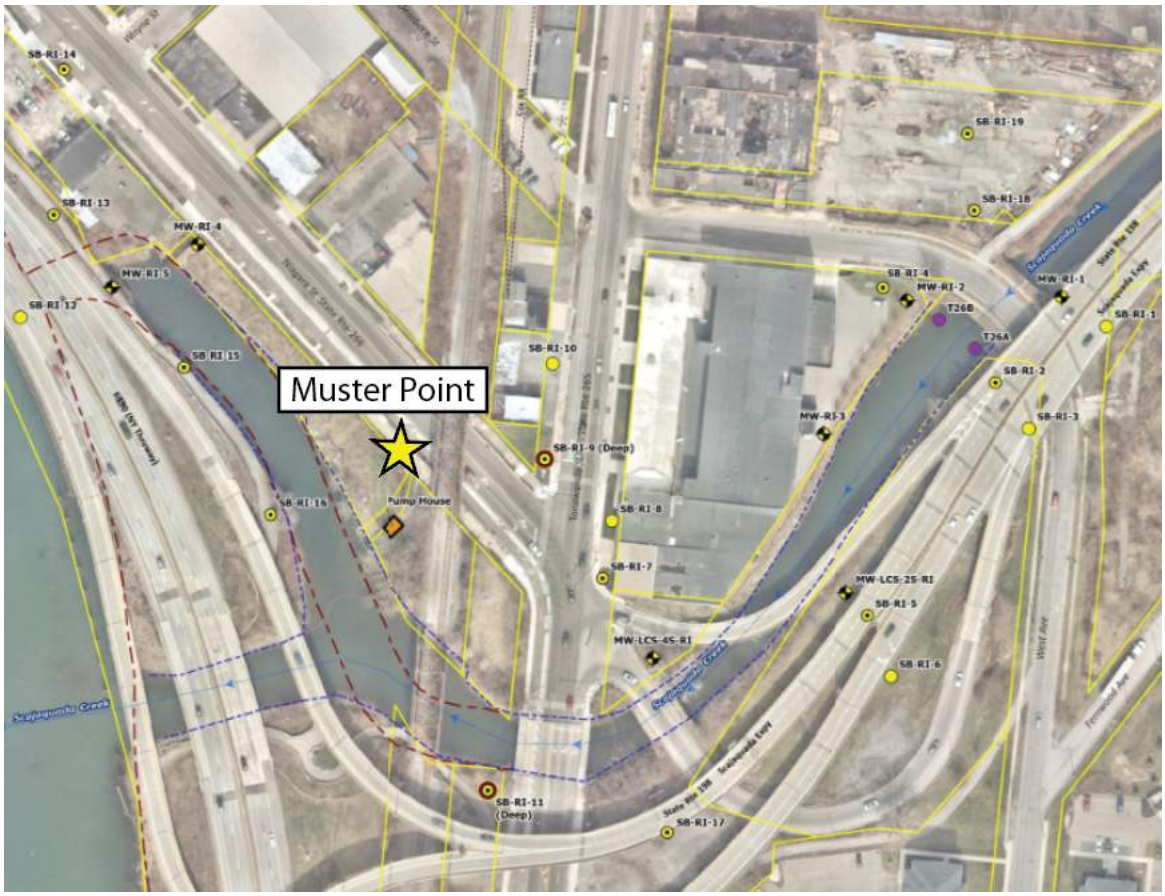
Site Name: Scajaquada Creek Operable Unit

GEI Project No: 2408160

Print Name	Signature
Project Manager: Richard Frappa	

Figure

Figure 1. Site Location and Muster Point



Appendix A

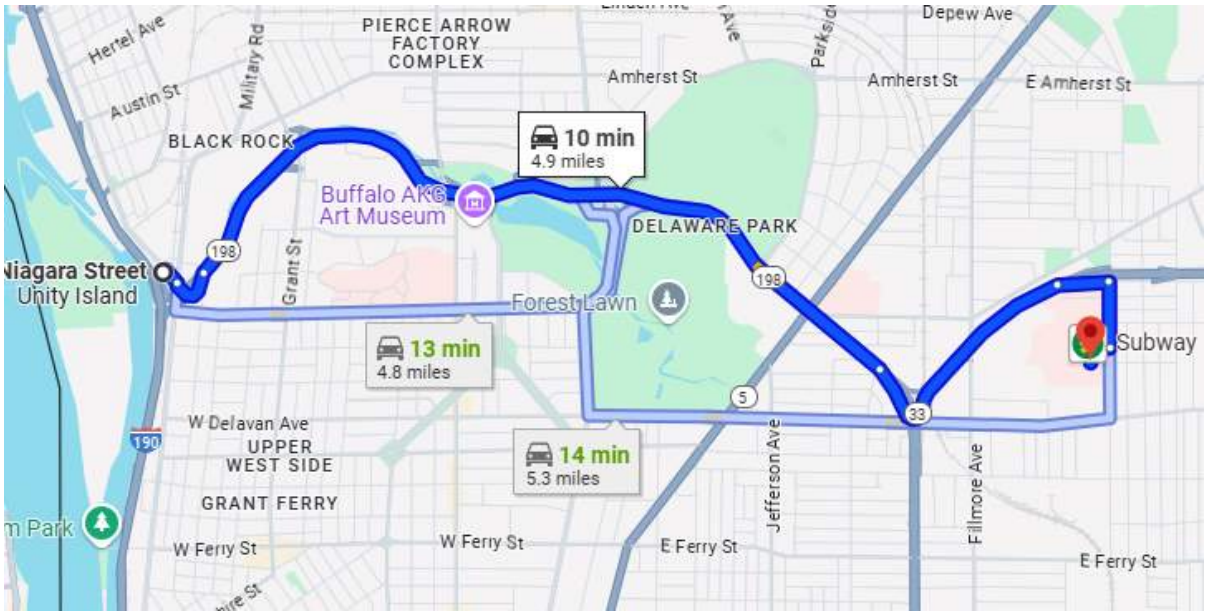
Hospital Directions

Hospital Information	
Erie County Medical Center 462 Grider St Buffalo, NY 14215	(716)898-3000

Scan this QR code or click the link for access to Google Maps to type in the address of your local hospital.

Directions from Site to Erie County Medical Center

Get on NY-198 E
1 min (0.3 mi)
Continue on NY-198 E. Drive to Grider St. Take the Grider St exit from NY-33 E
7 min (4.3 mi)
Continue onto Grider St to your destination
2 min (0.4 mi)



Appendix B

Community Air Monitoring Plan

GEI Consultants

Consulting
Engineers and
Scientists

Health and Safety Plan

Appendix A - Community Air Monitoring Plan

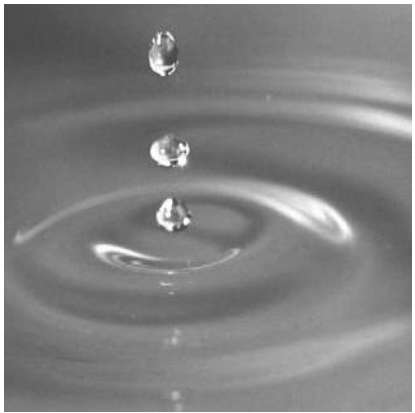
GLLA Program- Lower Scajaquada Creek Operable Unit
Buffalo, New York

NYSDEC Site #915351
Submitted to:

National Fuel Gas Distribution Corporation
6363 Main Street
Williamsville, NY 14221

Submitted by:
GEI Consultants, Inc., P.C.
100 Sylvan Parkway, Suite 400
Amherst, NY 14228

February 2025
GEI Project 2101620



Richard H. Frappa, P.G.
Senior Consultant

Kelly R. McIntosh, P.E., Ph.D.
Senior Consultant

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Abbreviations and Acronyms

CAMP	Community Air Monitoring Plan
COC	Compounds of Concern
GEI	GEI Consultants, Inc., P.C.
HASP	Health and Safety Plan
MGP	Manufactured Gas Plant
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
PAH	Polycyclic Aromatic Hydrocarbon
PID	Photo-ionization Detector
ppm	Parts per Million
SCWP	Site Characterization Work Plan
SVOC	Semi-Volatile Organic Compound
VOC	Volatile Organic Compound
$\mu g/m^3$	Micrograms per cubic meter

1. Introduction

This document presents the Community Air Monitoring Plan (CAMP) that will be implemented during the Remedial Investigation/ Feasibility Study (RI/FS) at the Lower Scajaquada Creek Operable Unit in Buffalo, New York. A CAMP is required by the New York State Department of Environmental Conservation (NYSDEC) and the New York State Department of Health (NYSDOH) at sites where ground-intrusive activities may result in airborne release of compounds of concern (COCs). This CAMP has been developed to monitor for total volatile organic compound (VOC) vapors, and for particulates (dust) which are the principal COCs resulting from intrusive activities performed during implementation of the Site Characterization Plan prepared for the Site.

The Site Characterization will consist of the following elements:

- Installation of soil borings and monitoring wells on the land portion of the Site,
- Sampling of monitoring wells,
- Completions of sediment borings in Scajaquada Creek.

The objectives of this CAMP are to:

- Ensure that the airborne concentrations of COCs are minimized to protect the community.
- Provide an early warning system so that potential emissions can be controlled on site at the source.
- Measure and document the concentrations of airborne COCs to confirm compliance with the specified limits.

This CAMP is a companion document to GEI's site-specific Health and Safety Plan (HASP) which is directed primarily toward protection of on-site workers within the designated work zones. Implementation of this CAMP will be required during any intrusive activities performed on the land portion of the Site, including the installation of soil borings, monitoring wells Sediment borings in Scajaquada Creek are proposed to be completed "in-the-wet" and are not anticipated to generate dust. Therefore, only VOC monitoring will be required in the work zone and upwind and downwind areas.

2. Air Monitoring Equipment, Methods, and Action Levels

This section provides instructions for conducting the CAMP. Discussed are the COCs to be monitored, the equipment to be used, and the action limits. Community air monitoring will be performed for total VOCs and particulates (dust) during the excavation and backfilling activities performed at the Site.

In addition to the community air monitoring, work/exclusion zone monitoring will be performed during work activities where impacted soil may be encountered. The exclusion zone air monitoring requirements, equipment, and action levels are described in the site-specific HASP for this project. Note, however, that the work zone air monitoring and the community air monitoring are conducted as part of the overall site control program. When work zone VOC or particulate readings are found to exceed the downwind CAMP limits, the field staff will check the upwind and downwind air monitoring instruments to assess whether control measures will be required.

2.1 Monitoring Locations

Air monitoring locations will be established at the start of each workday. A minimum of one station located upwind of the work area, and one located downwind of the work area/exclusion zone will be placed at the Site perimeter as determined by the prevailing wind direction at the start of the day. The purpose of the upwind station will be to determine the background concentration of VOCs and particulates at the worksite. The downwind monitoring station will be used to assess compliance with the NYSDEC/NYSDOH specified action limits for VOCs and particulates. The upwind VOC and dust measurements will be subtracted from the downwind measurements in order to compare the downwind instrument readings to the CAMP action levels.

The location of each monitoring station will be noted in the Daily Field Report along with the start and ending time for monitoring at each location. The locations of the instruments may be changed during the day to adapt to changing wind directions. Field personnel will be prepared to move the equipment to multiple locations in the event that there is little wind, if the wind direction changes frequently, or if there is a change to the location of the most sensitive downwind receptor location.

It is not anticipated that any work area will be within 20’ of any occupied structure during implementation of the RI/FS Work Plan. However, if any work area is less than 20 feet from the nearest occupied building, a downwind air monitoring station will be positioned at the air intake for the building or at the most sensitive exposure point for the downwind receptors. Background measurements inside the building will be made prior to the start of work. If

exceedances of the action levels are measured at the outside wall of the building, additional measurements will be made inside the building using portable meters.

If necessary, precautions to minimize the release of VOCs and particulates will be taken at the work zone, and engineering or work controls used to protect the downwind receptor. These controls for minimizing releases from the work zone are discussed in Section 3.

2.2 Air Monitoring Equipment

The monitoring instruments will be calibrated at the start of each workday, and again during the day if the performance of an instrument is in question. The time and method of calibration will be noted on the Daily Field Report. Both the photo-ionization detectors (PIDs) and particulate meters will be mounted on a tripod in a vented protective case, and programmed to record 15-minute averages. A monitoring technician will check the instrumentation at each of these locations regularly during the workday to ensure that they are operating properly.

2.2.1 VOC Monitoring Equipment

VOC monitoring will be performed using PIDs (RAE Systems MiniRAE™ or equivalent) equipped with a 10.2 or 10.6 eV bulb. The instruments will be set to record 15-minute running average concentrations. The PIDs will be equipped with an audible alarm to indicate an exceedance of the action level of 5 ppm total VOCs.

2.2.2 Particulate (Dust) Monitoring Equipment

Particulate monitoring will be performed using meters set to measure 10 micron and finer particulates (PM-10). Particulates will be monitored using an MIE DataRAM DR-2000l, TSI DustTrak™, or equivalent, which is capable of measuring PM-10. The equipment used will be set to record 15-minute running average concentrations, for comparison to the action levels.

In addition to the instrument readings, fugitive dust migration will be visually assessed during all work activities, and the observations recorded. Per NYSDEC requirements, visible dust migration will not be allowed. If visible dust is observed to be migrating from the work zone, the work will be stopped and dust control measures implemented.

2.3 Monitoring Action Levels and Responses

The action levels and responses for VOCs and particulates are presented in Table 1.

Table 1. Air Monitoring Response Levels and Actions

VOCs	
Response Level	**Actions**
>1 ppm at the wall of an occupied structure or at an air intake	▪ Check the indoor air concentration and compare with background measurements taken previously
>5 ppm above background for 15-minute average	▪ Temporarily halt work activities ▪ Continue monitoring, especially inside of occupied structures ▪ Perform work during cold weather ▪ If VOC levels decrease (per instantaneous readings) below 5 ppm over background, work activities can resume
Persistent levels >5 ppm over background but <25 ppm	▪ Halt work activities ▪ Identify source of vapors ▪ Corrective action to abate emissions ▪ Continue monitoring ▪ Resume work activities if VOC levels 200 feet downwind of the property boundary or half the distance to the nearest potential receptor is <5 ppm for a 15-minute average
>25 ppm at the perimeter of the work area	▪ Shut down work

Particulates	
Response Level	**Actions**
>100 µg/m^3 above background for 15-minute average or visual dust observed leaving the site	▪ Apply dust suppression ▪ Continue monitoring ▪ Continue work if downwind PM-10 particulate levels are <150 µg/m^3 above upwind levels and no visual dust leaving site
>150 µg/m^3 above background for 15-minute average	▪ Stop work ▪ Re-evaluate activities ▪ Continue monitoring ▪ Continue work if downwind PM-10 particulate levels are <150 µg/m^3 above upwind levels and no visual dust leaving site

Sources:

- NYSDOH Generic Community Air Monitoring Plan, December 2009, as published in NYSDEC DER-10, Appendix 1A, 2010.
- Fugitive Dust and Particulate Monitoring, NYSDEC DER-10, Appendix 1B, 2010.
- Special Requirements for Work Within 20 Feet of Potentially Exposed Individuals or Structures, NYSDOH.

All data will be downloaded to a computer on a daily basis and saved for review. The data will be provided to the NYSDEC and/or the NYSDOH upon request at any stage of the project.

If VOC or particulate action levels are observed to be exceeded during the workday, the event, the source, and corrective actions taken will be recorded on the Daily Field Report and reported to the on-site NYSDEC representative, if present. If an on-site representative is not

present, exceedances will be noted in the daily report to the NYSDEC project manager within one business day.

Table 2. Emergency Contacts and Telephone Numbers

Fire, Police, Ambulance		911
NYSDEC Contact	Matt King – Project Manager	716-851-7258
National Fuel Contact	Tanya Alexander – Project Manager	(716) 857-7410 office (716) 864-5318 cell
GEI Project Manager	Richard Frappa	(716) 984-5958 cell

2.4 Odor Monitoring

The field personnel will record observations of odors generated during the implementation of the RI/FS Work Plan. Odors will be recorded as weak, moderate, or strong, based on olfactory responses. When odors attributable to the exposing of impacted media are generated in the work area during excavation, observations will also be made at the downwind limit of the Site. The observations will be made to assess the potential for significant odors reaching on-site receptors or being transmitted off site. The downwind odor monitoring will be performed in conjunction with the PID and dust monitoring program described in this CAMP.

Upon detection of strong odors at the site perimeter, site controls, starting in the work area, will be implemented and will include encapsulation of the impacted media in 55-gallon drums or temporarily wrapping in polyethylene sheeting until more permanent mitigation can be accomplished. Note that the goal of the Odor Management Plan is to minimize and to prevent, where practicable, the off-site migration of odors. Due to the short distances between some work areas at the site and the property lines of on-site receptors, site controls may be implemented proactively when odors are detected in the breathing zone in some work areas.

There are no action levels specified for odors. In the event that odors persist at the downwind receptors or property line after control measures are carried-out, the odor conditions will be discussed with the National Fuel and NYSDEC project managers.

3. Control Procedures

A dust, odor, and organic vapor control and monitoring plan (DOOVC Plan) will be developed for on-site remedial construction work activity that will meet CAMP requirements and OSHA standards for construction (29 CFR 1926) after selection of the final remedy. The DOOVC Plan will specifically address work practices to minimize dust, odors and vapors during all remediation construction activities.

4. Documentation and Reporting

A Daily Data Sheet will be filled-out each day to record all of the details of the CAMP work. The form will be used to record the following information:

- Date and weather, with significant changes noted which may affect the positioning of the meters or recording of the data.
- Calibration results for the instruments.
- Locations of the upwind and downwind monitoring stations, and any changes made to the locations during the day to adjust for changing work locations or wind directions.
- Any significant readings made during the day, such as exceedances which occur and their causes.

Additional information will be noted in the project fieldbook(s), as necessary.

The electronic measurements from the PIDs and dust meters will be downloaded each day, reviewed, and archived. Exceedances of the action levels, if any, and the actions to be taken to mitigate the situations, will be discussed immediately with the on-site representatives, or reported within one business day to the NYSDEC project manager (if on-site NYSDEC oversight is not provided). The results of the daily CAMP monitoring will also be discussed in the daily written report to the NYSDEC project manager. Summaries of all air monitoring data will be provided to the NYSDEC or the NYSDOH upon request.

CAMP odor monitoring results will be recorded in the field log book and/or the Daily Data Sheet, and will also be available for review by the state agencies.

Appendix C

Job Hazard Analyses

Job Hazard Analysis

Task	**Barge Safety (Passenger)**		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	**Client Name**	National Fuel Gas
GEI Project Manager	Richard Frappa	**PM Review Date**	
Tools/Equipment Used			
Task Specific Training	GEI Annual Safety Training (Field or HAZWOPER), CPR/First Aid		
Personal Protective Equipment (PPE)	☐ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☒Hearing Protection ☒ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves Leather, work gloves ☐Respirator ☐Other		

JHA Prepared By: Josh Prygon	**Date:**	**Approved by: *RSM to complete***	**Date:**

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Equipment Setup	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries • Wear appropriate footwear • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions • Take breaks when carrying items frequently and/or for long distances • Do not overreach when picking up or placing items. • Use the buddy system when necessary

[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (Isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
2. Pre-boarding Safety Presentation	Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Hazard Identification HS-026 Ergonomic Program	• All passengers should be given a presentation of the barge's safety equipment, emergency procedures, hazards, and general passenger instructions/ expectations before boarding. • Passengers, along with the operator, are responsible for knowing the plan and emergency procedures. • Confirm all appropriate operational and safety equipment is on board and in working condition. • Attend a tailgate meeting and review the float plan in detail. • Determine your method of emergency communication (radios, cell phones, air horns, etc.) and obtain all equipment or information needed (radio, cell phone charger, key phone numbers, etc.). • The operator should review all emergency procedures before launching.
3. Boarding	Slips/ Trips/ falls, Drowning, Pinch points, Cuts and Lacerations	Water Safety HS-017 Hazard Identification HS-026 GEI's Ergonomics Program	• Do not board before you have operator approval. • Confirm the barge is operational before boarding. • All passengers should be given a tour/ presentation of the safety equipment, emergency procedures, hazards, and general passenger instructions. • When boarding, use 3 points of contact. Do not jump, run, board in inappropriate locations, or board without captain/ operator permission. The barge should be stationary and secured. • The operator and all passengers are responsible for housekeeping on the on the working platform. All objects, tools, and equipment should be secured and put away before the passengers board. After boarding, take time to find a place for everyone's belongings before leaving the launch. Do not take unnecessary items. • Know your path of travel and keep hands free. • Take orders from and direct communication to operator of vessel. • Hand off packages, backpacks, and carry-ons before attempting a transfer. • Keep self and others clear of suspended or carried loads. • Be aware of and stay clear of overhead hazards or equipment.
4. Barge Passenger	Struck By, Cuts and lacerations, pinch points, fire/ explosion, remote work areas, ergonomics,	Inclement Weather HS-010 Noise Exposure HS-012	• Take orders from and direct communication to operator of vessel. • Do not interfere with the operator while they are maneuvering • Make sure all personnel on board have PFDs on and/or that railing

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
	drowning	Water Safety HS-017 Hazard Identification HS-026	systems are OSHA-compliant. • All life jackets or PFDs should be fastened and well fitting. • Prior to leaving the shore or dock, the operator should look around to determine if there are any oncoming boats or other hazards. Passengers should assist in looking for hazards. • Boat/barge safety practices will be conducted in general accordance with guidance provided in the U.S. Army Corps of Engineers (USACE) Safety and Health Requirements Manual (EM) 385-1-1 and the U.S. Coast Guard. • Keep limbs within the perimeter of the barge platform while it is in motion. • Passengers avoid excessive movement and pay attention to the navigation while the barge is in motion. • The consumption of alcoholic beverages and the use of illegal drugs will not be permitted aboard boats/barges on which GEI employees operating or conducting company business. This includes boats/barges that may belong to someone else (subcontractor, client, etc.). • Employees working on a barge will monitor the weather, incorporating, as appropriate, National Oceanic and Atmospheric Administration (NOAA) marine weather broadcasts and other local commercial weather forecasting services as may be available. • Beware of carbon monoxide poisoning when the engine is running in closed or poorly ventilated areas. • Do not attempt to brace the barge or push off against objects or hazards to assist the operator without specific directions to do so. • Report any near miss or incident situations. • Dress for the weather. • The operator shall maintain good weight distribution to avoid capsizing. • Follow the operator's directions in an emergency situation. Do not panic. • If equipment is on board, confirm it is well secured to prevent it from shifting during transit.
5. Docking (to other boats/barges, shorelines, launches, docks, etc.)	Cuts, scrapes, Struck-by, Eye Injury, Slips/Trips/Falls,	Noise Exposure HS-012 Water Safety HS-017	• Everyone should keep hands, feet, and equipment inside the vessel before docking to avoid pinch points.

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
	Ergonomics, Biological Hazards, Drowning	Hazard Identification HS-026	• If docking to another boat or barge, clearly communicate your plans before coming up alongside their vessel. • If docking on a bank or shoreline, pay attention and watch for overhead branches, poisonous plant materials, and other hazards. • Avoid overreaching when transferring tools or equipment from one boat to another. Confirm both boats are stationary and prepared for the transfer.
6. De-boarding	Slips/ Trips/ falls, Drowning, Pinch points, Cuts and Lacerations, Drowning	Water Safety HS-017 Hazard Identification HS-026 GEI's Ergonomics Program	• When de-boarding, use three points of contact. Do not jump, run, board in inappropriate locations, or de-board without captain/ operator permission. The barge should be stationary and secured. If deboarding to land, check the consistency of the soil before stepping off the boat. • Only exit the barge in approved location • Maintain good housekeeping on the vessel. • Know your path of travel and keep hands free. • Take orders from and direct communication to operator of vessel. • Hand off packages, backpacks, and carry-ons before attempting a transfer. • Keep self and others clear of suspended or carried loads. • Do not leave any trash behind and take your belongings with you. • Once de-boarded, clear the area so that the barge to be trailered or craned out.
7. Equipment De-Mob	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries • Wear appropriate footwear • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions • Take breaks when carrying items frequently and/or for long distances • Do not overreach when picking up or placing items. • Use the buddy system when necessary • Properly secure all cargo before traveling

Job Hazard Analysis

Task	**Boat Safety- Bathymetric Survey (Operator and Passenger)**		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	**Client Name**	
GEI Project Manager	Richard Frappa	**PM Review Date**	
Tools/Equipment Used	Inflatable Boat, Transducer, ODOM Hydrotrak II Fathometer, Laptop with Hypack Software		
Task Specific Training	GEI Annual Safety Training (Field or HAZWOPER), CPR/First Aid, State Required Boater Safety License (Minimum Requirement)		
Personal Protective Equipment (PPE)	☐ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☐Hearing Protection ☒ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves Leather, work gloves ☐Respirator ☐Other		

JHA Prepared By: Josh Prygon	**Date:**	**Approved by: *Insert Preparer's Name***	**Date:**

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Trailering	Vehicle Accidents, Crush/pinch points, Struck by	Driver Safety HS-004 Hazard Identification HS-026 Safe Trailer Use HS-028	• Secure the load completely before traveling. Make stops to check the load. • Only those trained in and comfortable with trailer maneuvering will be allowed to drive with a trailer. • Complete a pre-trip inspection of the vehicle, trailer, and load. • Drive at moderate speeds. This will place less strain on your tow vehicle and trailer. Trailer instability (sway) is more likely to occur as speed increases. • Avoid sudden starts and stops that can cause skidding, sliding, or jackknifing. • Make wider turns at curves and corners. • Check routes and restrictions on bridges and tunnels prior to departure. • Allow considerably more distance for stopping. • When passing a slower vehicle or changing lanes, signal well in advance and make sure you allow extra distance to clear the vehicle before you pull back into the lane. • Avoid passing on steep upgrades or downgrades. • When unhitching a trailer, place blocks at the front and rear of the trailer to keep the trailer from moving while people work, PPE (gloves) when coupling is released.

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Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
2. Equipment Setup	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.
3. Boat Safety Check	Cuts/Scrapes, Slips/Trips/Falls, Pinch points, Overreaching	Hazard Identification HS-026 Ergonomic Program	• Confirm all appropriate operational and safety equipment is on board and in working condition. • Complete appropriate paperwork to document the safety check procedure. • Inspect the vessel for damage. If damage is found, the vessel should be taken out of service until it can be repaired. • Attend a tailgate meeting and review the float plan in detail. • Determine your method of emergency communication (radios, cell phones, air horns, etc.) and obtain all equipment or information needed (radio, cell phone charger, key phone numbers, etc.). • The operator should review all emergency procedures before launching. • Check with your local and state agency to verify their boat law/regulations before launching.
4. Handling Batteries (Batteries are used to power a small motor and the survey equipment)	Spill, Explosion, Skin Contact	Hazardous Substance Management HS-009 Hazard Identification HS-026 Hazard Communication (HAZCOM) Program	• Avoid bringing metal into contact with the battery terminals. • Never allow both terminals to make contact with an item simultaneously especially you • Do not use damaged batteries due to the risk of battery acid contact • Wear safety gloves when handling the batteries

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
5. Launching the Vessel	Pinch Points, Struck By, cuts and lacerations, Slips/Trips/Falls, Ergonomics	Driver Safety HS-004 Water Safety HS-017 Hazard Identification HS-026 Safe Trailer Use HS-028	• Inspect the launch site before backing down the ramp. Look for people, debris, water body conditions, ruts, and other hazards. • Wear proper footwear. Maintain three points of contact. • Stay clear of moving vehicles and equipment. Keep hands clear of moving parts. • Disconnect trailer lights prior to boat entering the water. • Complete inspection of boat prior to launch. Start boat when in water, while hooked to the trailer. Do not unhook from trailer until boat is fully operational. • PFDs are to be worn within 10' of the water. • Utilize a spotter when backing up. • Hearing Protection must be worn around all loud motors. • All unnecessary personnel should stay out of the launch area while vehicles and boats are in motion. • Confirm secure tie off, anchor hold, or spud pole stick before deboarding the vessel.
6. Passenger Boarding	Slips/ Trips/ falls, Drowning, Pinch points, Cuts and Lacerations, drowning	Water Safety HS-017 Hazard Identification HS-026 GEI's Ergonomics Program	• Confirm the motor is working properly before anyone but the operator boards. • All passengers should be given a tour/ presentation of the boat's safety equipment, emergency procedures, hazards, and general passenger instructions. • When boarding, use three points of contact. Do not jump, run, board in inappropriate locations, or board without captain/ operator permission. The boat should be stationary and secured. • The operator should confirm housekeeping is maintained on the boat. All objects, tools, and equipment should be secured and put away before the passengers board. Take time to find a place for the passenger's belongings before leaving the launch. • Know your path of travel and keep hands free. • Take orders from and direct communication to operator of vessel. • Hand off packages, backpacks, and carry-ons before attempting a transfer. • Keep self and others clear of suspended or carried loads.
7. Boat Operation	Struck By, Cuts and lacerations, pinch points, fire/ explosion, remote work areas, ergonomics, drowning	Inclement Weather HS-010 Noise Exposure HS-012 Water Safety HS-017 Hazard Identification HS-026	• Allow engine to warm up idle speed before leaving the dock or shore. • Make sure all personnel on board are wearing PFDs. • Prior to leaving the shore or dock, the operator should look around to determine if there are any oncoming boats or other

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
			hazards. • Boat safety practices will be conducted in general accordance with guidance provided in the U.S. Army Corps of Engineers (USACE) Safety and Health Requirements Manual (EM) 385-1-1 and the U.S. Coast Guard. • Keep limbs inside the boat while it is in Operation. • Confirm all passengers stay in a sitting position and pay attention to the navigation while the boat is in motion. • Any boat operator, whether GEI or a subcontractor, is not to exceed 12 hours of duty time in a 24-hour period. • The consumption of alcoholic beverages and the use of illegal drugs will not be permitted aboard boats/barges on which GEI employees operating or conducting company business. This includes boats/barges that may belong to someone else (subcontractor, client, etc.). • Employees operating or working on a boat will monitor the weather, incorporating, as appropriate, National Oceanic and Atmospheric Administration (NOAA) marine weather broadcasts and other local commercial weather forecasting services as may be available. • Beware of carbon monoxide poisoning when the boat engine is running in closed or poorly ventilated areas. • Confirm safe proximity to stationary objects. • Report any near miss or incident situations. • Use proper rules of the river when approaching other vessels. • Confirm vessel is out of the water before dark. Operate to the light conditions. • Dress for the weather. • Operators must keep vessels away from turbulent waters caused by discharges, rapids/large riffles, and stationary hazards. • Stay away from shallow or vegetated areas to avoid motor clogging or damage. • The operator shall maintain good weight distribution to avoid capsizing. • Do not stand in the boat. Avoid sudden movements. • Stay away from the edge of the boat/barge. • Have an extra set of clothing in case of drenching/soaking to prevent hypothermia.

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
8. Docking (to other boats/barges, shorelines, launches, docks, etc.)	Cuts, scrapes, Struck-by, Eye Injury, Slips/Trips/Falls, Ergonomics, Biological Hazards	Noise Exposure HS-012 Water Safety HS-017 Hazard Identification HS-026	• Confirm all passengers have hands, feet, and equipment inside the vessel before docking to avoid pinch points. • If docking to another boat or barge, clearly communicate your plans before coming up alongside their vessel. • If docking on a bank or shoreline, watch for overhead branches, poisonous plant materials, and other hazards. • Avoid overreaching when transferring tools or equipment from one boat to another. Confirm both boats are stationary and prepared for the transfer.
9. De-boarding Passengers	Slips/ Trips/ falls, Drowning, Pinch points, Cuts and Lacerations	Water Safety HS-017 Hazard Identification HS-026 GEI's Ergonomics Program	• When de-boarding, use three points of contact. Do not jump, run, board in inappropriate locations, or de-board without captain/ operator permission. The boat should be stationary and secured. If deboarding to land, check the consistency of the soil before stepping off the boat. • Maintain good housekeeping on the vessel. • Know your path of travel and keep hands free. • Take orders from and direct communication to operator of vessel. • Hand off packages, backpacks, and carry-ons before attempting a transfer. • Keep self and others clear of suspended or carried loads. • Do not leave any trash behind and take your belongings with you.
10. Loading Boat on Trailer	Slips/ Trips/ falls, Drowning, Pinch points, Cuts and Lacerations	Driver Safety HS-004 Water Safety HS-017 Hazard Identification HS-026 Safe Trailer Use HS-028	• Confirm the boat launch area is clear before backing the boat trailer down the ramp. • Back up slowly and work with a spotter. • Line the boat up properly on the trailer before pulling it. Tilted or improperly loaded boats will not be secured properly for road travel. • Do not stand between the boat, trailer, and vehicle when loading. • Maintain a high level of communication. • Only use properly maintained winches and equipment. • Securely fasten tie-downs before traveling on roads.
11. Vessel Maintenance	Cuts, Lacerations, Eye Injury, Burns, Pinch points, motorized equipment, ergonomics	Non-Powered Hand Tools HS-008a Hazard Identification HS-026 Manual Lifting HS-025 Ergonomic Program	• Vessels should be maintained according to the operator's manual and as frequently as necessary to keep the boat in good working condition. • Only perform maintenance to your level of comfort and knowledge. Use professionals when necessary. • Use the right equipment for the job and avoid working in

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
			ergonomically harsh positions. • Team lift or use equipment when necessary. • Clean the vessel to prevent the spread of invasive species. • All damage or wear should be addressed prior to further use.
12. Equipment Demobilization	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary. • Properly secure all cargo before traveling.

Job Hazard Analysis

Task	Drilling Oversight		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	Client Name	National Fuel Gas
GEI Project Manager	Richard Frappa	PM Review Date	
Tools/Equipment Used			
Task Specific Training	GEI Annual Safety Training (Field or HAZWOPER), First Aid/CPR		
Personal Protective Equipment (PPE)	☒ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☒Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves Nitrile ☐Respirator ☐Other		

JHA Prepared By: Josh Prygon	Date:	Approved by: *RSM to complete*	Date:

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Mobilize on site	Slips/trips/falls, stuck by, crushing hazards	Hazard Identification HS-026	• Wear PPE that properly fits, is in good condition, and is appropriate for the activities and hazards. • Maintain good visibility of the work area. • Avoid walking on uneven, steeply sloped, or debris ridden ground surfaces. • Plan tasks prior to preforming them including an activity hazard analysis. • Keep trafficked areas free from slip/trip/fall hazards. • Wear shoes with traction. • Avoid traversing steep areas in slippery conditions. • Do not carry heavy objects to work areas, on steeply sloped areas, or where steep areas must be traversed to reach work areas.

[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (Isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
2. Utility survey/clearance	Shock, Explosion, Fire	Utility Mark-Out HS-014	• Confirm that all utilities have been properly marked prior to beginning work. • If a utility strike occurs, call the appropriate authorities immediately. Clear the area and do not delay. • Confirm adequate clearance from overhead utilities. • Do not stand or sit under suspended loads or near any pressurized equipment lines.
3. Observe contractor activities	Struck by/crushing hazards	Working Around Heavy Equipment HS-018	• Identify yourself and your work location to heavy equipment operators, so they may incorporate you into their operations. • Coordinate hand signals with operators. • Confirm equipment inspection has been completed. • Stay Alert! Pay attention to equipment backup alarms and swing radii. • Position yourself in a safe location. • Notify the contractor immediately if any problems arise. • Do not operate cellular telephones in the vicinity of heavy equipment operation.
4. Demobilize from site	Personal/material security, slips/trips/falls	Hazard Identification HS-026	• Confirm all materials are secured/labeled, including drums and any equipment left on site. • Confirm that any open trenches/pits are properly marked or barricaded. • Clean all equipment/tools prior to leaving. • Secure any gates/locks. • Notify project manager (or designee) you are leaving the site.

Job Hazard Analysis

Task	Driving – Site Mobilization		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	Client Name	National Fuel Gas
GEI Project Manager	Richard Frappa	PM Review Date	
Tools/Equipment Used	GEI fleet vehicle, rental vehicle, or personal vehicle		
Task Specific Training	GEI Annual Safety Training (Field or HAZWOPER), First Aid/CPR		
Personal Protective Equipment (PPE)	☐ Hard Hat ☐Safety Glasses ☐Safety-Toed Boots ☐ Reflective Vest ☐Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☐ Gloves Nitrile ☐Respirator ☐Other		

JHA Prepared By: Josh Prygon	Date:	Approved by: *RSM to complete*	Date:

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Inspect Vehicle	Slips/trips/falls	Hazard Identification HS-026 Driver Safety HS-004	• Walk around vehicle to inspect for any vehicle safety issues or hazards that could be within the travel path. • Adjust mirrors and seats prior to driving. • Becoming familiar with dashboard, center console, and steering controls. • Locate the turn signals, windshield wipers, lights, emergency flashers, and the heating, air conditioning, and defrost controls. • Use a spotter if needed when backing up.
2. Driving	Struck by/crushing hazards	Driver Safety SOP HS-004	• Employees must wear their safety belt while in a moving vehicle. • Employees will follow safe driving behaviors, which include limiting distractions such as manipulating radios or other equipment that may cause a distraction. Employees will not exceed the posted speed limit and will maintain a safe distance between other vehicles. • Use defensive driving techniques. • Avoid driving during hazardous weather conditions. • Driving distance and time after a 12-hour shift will not exceed 30 miles or 30 minutes (whichever is greater). • Vehicle accidents will be reported in accordance with GEI's Accident Reporting procedures.

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Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
3. Parking Vehicle	Struck by, crushing hazards, security	Driver Safety SOP HS-004	• Be aware of surrounding conditions. Park in designated areas or a safe area away from heavy equipment. • Position the vehicle in a manner which reduces or eliminates the need to operate the vehicle in reverse. Choose an easy-exit parking space, like pull-through or where no one else is parked. • Don't crowd neighboring vehicles; be sure to park your vehicle in the middle of your space. • Secure equipment and supplies in the trunk or where they cannot be seen. Or take items with you if they cannot be hidden. • If at night, park in well-lit areas.
4. Site Entry	Slips/trips/falls, struck by, crushing	Working Around Heavy Equipment HS-018 Hazard Identification HS-026	• Don appropriate PPE prior to walking on site. • Identify yourself and your work location to heavy equipment operators, so they may incorporate you into their operations. • Stay Alert! Pay attention to equipment backup alarms and swing radii. • Avoid distractions like using cell phones while traversing the site.
5. Backing Up Vehicle	Struck by, crushing	Driver Safety HS-004	• Before entering your vehicle do a walk-around. Check for fences, poles, drop-offs, buildings, etc. • Know your clearances. While performing your walk-around also check for obstructions, low hanging eaves and tree limbs, wires, and any other potential clearance-related obstacles. • Use a spotter. Do not allow the spotter to be positioned directly behind your vehicle or walk backwards behind you while giving instructions. They should be off to the driver's side where you can see them in your side mirror.

Job Hazard Analysis

Task	Drum Handling/Staging		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	Client Name	National Fuel Gas
GEI Project Manager	Richard Frappa	PM Review Date	
Tools/Equipment Used	Drum opening tools (bung wrench/lid tool/drum dolly)		
Task Specific Training	GEI Annual Safety Training (HAZWOPER), First Aid/CPR		
Personal Protective Equipment (PPE)	☐ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☐Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves Nitrile, Cut resistant ☒Respirator if action levels are exceeded ☐Other		

JHA Prepared By: Josh Prygon	Date:	Approved by: *RSM to complete*	Date:

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Mobilize On Site/Equipment Setup	Slips/trips/falls, stuck by, crushing hazards	Hazard Identification HS-026	• Wear PPE that properly fits, is in good condition, and is appropriate for the activities and hazards. • Maintain good visibility of the work area. • Avoid walking on uneven, steeply sloped, or debris ridden ground surfaces. • Plan tasks prior to preforming them including an activity hazard analysis. • Keep trafficked areas free from slip/trip/fall hazards. • Wear shoes with traction. • Avoid traversing steep areas in slippery conditions. • Do not carry heavy objects to work areas, on steeply sloped areas, or where steep areas must be traversed to reach work areas.
2. Opening/Closing Drums	Contaminant Contact, Cuts or Abrasions Heavy Lifting , Slips/Trips/Falls	Container Management HS–003 Ergonomic Program	• Wear proper PPE during sampling including work gloves/nitrile gloves, safety glasses and face shield as appropriate. • Confirm the drum is upright and stable to avoid tipping while opening the drum. • Inspect the drum prior to opening. Do not handle drums with bulging sides. When a drum or container exhibits signs of over pressurization (such as swelling or bulging), the person at the is not to be moved until the cause of the over-pressurization has

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Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
			been determined and proper containment procedures have been implemented. • Inspect drum tools before use. If equipment is damaged, tag it, and do not use. • If the drum has been sealed for a long time, or has been exposed to changes in temperature, there may be pressure built up inside. Gently tap the bung holes with a rubber mallet, and remove the caps with a drum wrench or drum opening tool/pair of pliers to release pressure. • Use drum wrench to loosen the ring clamp around the outside of the drum. • Once the ring is removed, if the lid cannot be removed easily, tap around the entire edge of the lid with a hammer, then use a drum opening tool/wrench to pry the lid away from your body. • Do not use picks or chisels to open drums. • Do not lean over open drums or stand on closed drums. • Dispose of gloves after use and wash hands. • Wear work gloves over nitrile gloves.
3. Moving/Staging Drums	Contaminant Contact, Cuts or Abrasions Heavy Lifting , Slips/Trips/Falls	Container Management HS–003 Manual Lifting HS–025 Ergonomic Program	• Wear proper PPE during sampling including nitrile gloves and safety glasses and face shield as appropriate. • Before moving, check the drum for damage and confirm the bung/lid is securely fastened. Use proper tools to secure. • Confirm the pathway you plan to take is clear and free from debris. • Confirm the drum dolly being used is in a good condition and is designed to handle the specific drum-type being handled; some drum dollies cannot handle plastic open-top drums. • Do not attempt to move drums up and down steep slopes with manual equipment not intended for the application. • Operate drum hand trucks with a straight back using two hands and your leg if required by the design of the trolley; confirm no one is standing close to the load during manipulation. • Do not handle drums with bulging sides. • Dispose of gloves after use and wash hands. • Wear work gloves over nitrile gloves. • Use proper lifting techniques. • Ask fellow worker for help.

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
4. Demobilize from site	Personal/material security, slips/trips/falls	Hazard Identification HS-026	• Confirm all materials are secured/labeled, including drums and any equipment left on site. • Clean all equipment/tools prior to leaving. • Secure any gates/locks. • Notify project manager (or designee) you are leaving the site.

Job Hazard Analysis

Task	GPR Utility Scanning		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	Client Name	National Fuel Gas
GEI Project Manager	Richard Frappa	PM Review Date	
Tools/Equipment Used	Ground Penetrating Radar Unit, Marking paint		
Task Specific Training	GEI Annual Safety Training (Field or HAZWOPER), First Aid/CPR, Manufacturer Specific Training on GPR Equipment		
Personal Protective Equipment (PPE)	☐ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☐Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves leather or other mechanics gloves ☐Respirator ☐Other		

JHA Prepared By: Josh Prygon	Date:	Approved by: *RSM to complete*	Date:

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Mobilize on site	Slips/trips/falls, stuck by, crushing hazards	Hazard Identification HS-026	• Wear PPE that properly fits, is in good condition, and is appropriate for the activities and hazards. • Maintain good visibility of the work area. • Avoid walking on uneven, steeply sloped, or debris ridden ground surfaces. • Plan tasks prior to preforming them including an activity hazard analysis. • Keep trafficked areas free from slip/trip/fall hazards. • Wear shoes with traction. • Avoid traversing steep areas in slippery conditions. • Do not carry heavy objects to work areas, on steeply sloped areas, or where steep areas must be traversed to reach work areas.
2. Carry Equipment and Set-up	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions.

[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (Isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
			• Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.
3. GPR Equipment Utility Scanning	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Powered Hand Tools HS-008b Manual Lifting HS-025 Ergonomic Program	• Visually scan route before making passes with GPR. • Look for indicators of utility presence. • Keep GPR equipment under constant surveillance or secured against unauthorized use or removal.
4. Demobilize from site	Personal/material security, slips/trips/falls	Hazard Identification HS-026	• Confirm all materials/equipment are secured. • Clean all equipment/tools prior to leaving. • Secure any gates/locks. • Notify project manager (or designee) you are leaving the site.

Job Hazard Analysis

Task	**Groundwater Sampling – Bailer**		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	**Client Name**	NFG
GEI Project Manager	Richard Frappa	**PM Review Date**	
Tools/Equipment Used	Bailer, string, scissors, sample containers, cooler, ice		
Task Specific Training	GEI Annual Safety Training (Field or HAZWOPER), First Aid/CPR		
Personal Protective Equipment (PPE)	☐ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☐Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves Nitrile/Cut resistant ☐Respirator ☐Other		
JHA Prepared By: Josh Prygon	**Date:**	**Approved by: *RSM to complete***	**Date:**

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Carry Equipment and Set-up	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.
2. Cut Tubing/String	Cuts/Scrapes, pinch points	Non-Powered Hand Tools HS-008a	• Wear cut resistant gloves. • Use safe cutting tools such as safety scissors is a best practice. • Keep free hand out of the way. • Secure the item being cut. • Use only sharp blades; dull blades require more force that results in less control. • Do not put the sharp cutting tool in your pocket.

[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (Isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
3. Secure string to bailer and lower bailer down well to collect water	Contaminant Contact, Awkward position, Slips/Trips/Falls, Heavy lifting	Ergonomic Program	• Wear proper PPE during sampling including nitrile gloves and safety glasses and face shield as appropriate. • Keep trafficked areas free from slip/trip/fall hazards. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.
4. Collect GW Reading/Samples	Awkward position, contaminant exposure	Hazard Identification HS–026 Benzene Awareness Program Ergonomic Program	• Wear Nitrile/neoprene gloves and dispose of gloves after use and wash hands. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
5. Drum Purge Water	Contaminant Contact, Cuts or Abrasions Heavy Lifting , Slips/Trips/Falls	Container Management HS–003 Manual Lifting HS–025 Ergonomic Program	• Wear proper PPE during sampling including nitrile gloves and safety glasses and face shield as appropriate. • Use proper dollies or drum moving tools. • Use applicable tools to open/close drum lids. • Do not handle drums with bulging sides. • Dispose of gloves after use and wash hands. • Wear work gloves over nitrile gloves. • Use proper lifting techniques. • Ask fellow worker for help. • Keep trafficked areas free from slip/trip/fall hazards.
6. Pack Samples	Heavy lifting, cuts, or abrasions	Manual Lifting HS–025 Ergonomic Program	• Confirm that the lids on all bottles are tight (will not leak). • Place cushioning/absorbent material in the bottom of the cooler and then place the containers in the cooler with sufficient space to allow for the addition of cushioning between the containers. • Use proper dollies or other lifting tools. • Use proper lifting techniques. • Take regular breaks and do not work in unusual positions for long periods of time. • If glassware breaks, dispose of in puncture-resistant containers. Use cut resistant gloves to handle any broken glass.

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
7. Equipment Decontamination	Contaminant Contact, Cuts or Abrasions, Slips/Trips/Falls	Hazard Identification HS–026 Ergonomic Program	• Wear nitrile gloves and glasses to provide eye protection from splashing. • Wash hands immediately after use. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
8. Equipment Demobilization	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.

Job Hazard Analysis

Task	**Groundwater Sampling – (Low Flow)**		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	**Client Name**	National Fuel Gas
GEI Project Manager	Richard Frappa	**PM Review Date**	
Tools/Equipment Used	Hand tools, pump, water level meter, battery		
Task Specific Training	GEI Annual Safety Training (Field or HAZWOPER), First Aid/CPR		
Personal Protective Equipment (PPE)	☐ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☐Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves Nitrile/Cut resistant ☐Respirator ☐Other		
JHA Prepared By: Josh Prygon	**Date:**	**Approved by: *RSM to complete***	**Date:**

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Carry Equipment and Set-up	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.
2. Open Monitoring Well	Cuts/Scrapes, Insect Bites	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Use proper tools to open monitoring wells. • Open casings slowly in case insects inhabit the area. • If insect nest is inside casing, open protective casing and see if insects will leave the nest. Once free of insects, proceed with work. If not, relocate well if possible. • If severely allergic to insects, have appropriate epinephrine on

[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (Isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
			hand. • Remain calm and still if a single stinging insect is flying around. (Swatting at an insect may cause it to sting.) • If you are attacked by several stinging insects at once, run to get away from them. (Bees release a chemical when they sting, which may attract other bees.) • Follow incident reporting procedures if first aid is needed.
3. Cut Tubing/String	Cuts/Scrapes, pinch points	Non-Powered Hand Tools HS-008a	• Wear cut resistant gloves. • Using safe cutting tools such as safety scissors or snips are best practices. • Keep free hand out of the way. • Secure the item being cut. • Use only sharp blades; dull blades require more force that results in less control. • Do not put the sharp cutting tool in your pocket.
4. Deploy Water Level Meter	Awkward position, contaminant exposure	Hazard Identification HS–026 Ergonomic Program	• Wear Nitrile/neoprene gloves and dispose of gloves after use and wash hands. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep control of the reel when deploying the probe. Avoid letting the probe drop quickly into the well. • Reel the probe up slowly to avoid splashes from contaminated groundwater or sediment. • Keep trafficked areas free from slip/trip/fall hazards.
5. Collect GW Reading/Samples	Awkward position, contaminant exposure	Hazard Identification HS–026 Ergonomic Program	• Wear Nitrile/neoprene gloves and dispose of gloves after use and wash hands. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
6. Pack Groundwater Samples	Heavy lifting, cuts, or abrasions	Manual Lifting HS–025 Ergonomic Program	• Confirm that the lids on all bottles are tight (will not leak). • Place cushioning/absorbent material in the bottom of the cooler and then place the containers in the cooler with sufficient space to allow for the addition of cushioning between the containers. • Use proper dollies or other lifting tools. • Use proper lifting techniques. • Take regular breaks and do not work in unusual positions for long periods of time. • If glassware breaks, dispose of in puncture-resistant containers.

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
			Use cut resistant gloves to handle any broken glass.
7. Drum Purge Water	Contaminant Contact, Cuts or Abrasions Heavy Lifting , Slips/Trips/Falls	Container Management HS–003 Manual Lifting HS–025 Ergonomic Program	• Wear proper PPE during sampling including nitrile gloves and safety glasses and face shield as appropriate. • Use proper dollies or drum moving tools. • Use applicable tools to open/close drum lids. • Do not handle drums with bulging sides. • Dispose of gloves after use and wash hands. • Wear work gloves over nitrile gloves. • Use proper lifting techniques. • Ask fellow worker for help. • Keep trafficked areas free from slip/trip/fall hazards.
8. Equipment Decontamination	Contaminant Contact, Cuts or Abrasions, Slips/Trips/Falls	Hazard Identification HS–026 Ergonomic Program	• Wear nitrile gloves and glasses to provide eye protection from splashing. • Wash hands immediately after use. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
9. Equipment Demobilization	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.

Job Hazard Analysis

Task	Monitoring Well Installation and Development		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	Client Name	National Fuel Gas
GEI Project Manager	Richard Frappa	PM Review Date	
Tools/Equipment Used	Pump, tubing, bucket, hand tools		
Task Specific Training	GEI Annual Safety Training (Field or HAZWOPER), First Aid/CPR		
Minimum PPE Required	☒ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☒Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves Nitrile/Cut resistant ☐Respirator ☐Other		

JHA Prepared By: Josh Prygon	Date:	Approved by: *RSM to complete*	Date:

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Mobilize on-site/Carry Equipment and Set-up	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• Wear PPE that properly fits, is in good condition, and is appropriate for the activities and hazards. • Maintain good visibility of the work area. • Avoid walking on uneven, steeply sloped, or debris ridden ground surfaces. • Plan tasks prior to preforming them including an activity hazard analysis. • User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary. • Do not carry heavy objects to work areas, on steeply sloped areas, or where steep areas must be traversed to reach work areas.

[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
2. Utility survey	Shock, Explosion, Fire	Utility Mark Out HS-014	• Confirm that all utilities have been properly marked prior to beginning work. • If a utility strike occurs, call the appropriate authorities immediately. Clear the area and do not delay.
3. Observe contractor activities	Struck by/crushing hazards	Working Around Heavy Equipment HS-018	• Identify yourself and your work location to heavy equipment operators, so they may incorporate you into their operations. • Coordinate hand signals with operators. • Confirm equipment inspection has been completed. • Stay Alert! Pay attention to equipment backup alarms and swing radii. • Position yourself in a safe location. • Confirm adequate clearance from overhead utilities. • Notify the contractor immediately if any problems arise. • Do not stand or sit under suspended loads or near any pressurized equipment lines. • Do not operate cellular telephones in the vicinity of heavy equipment operation.
4. Open Monitoring Well	Cuts/Scrapes, Insect Bites	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Use proper tools to open monitoring wells. • Open casings slowly in case insects inhabit the area. • If insect nest is inside casing, open protective casing and see if insects will leave the nest. Once free of insects, proceed with work. If not, contact project manager if problem persists. • If severely allergic to insects, have appropriate epinephrine on hand. • Remain calm and still if a single stinging insect is flying around. (Swatting at an insect may cause it to sting.) • If you are attacked by several stinging insects at once, run to get away from them. (Bees release a chemical when they sting, which may attract other bees.) • Follow incident reporting procedures if first aid is needed.
5. Cut Tubing	Cuts/Scrapes, pinch points	Non-Powered Hand Tools HS-008a	• Wear cut resistant gloves. • Using safe cutting tools such as safety scissors or snips are best practices. • Keep free hand out of the way. • Secure the item being cut. • Use only sharp blades; dull blades require more force that results in

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
			less control. • Do not put the sharp cutting tool in your pocket.
6. Purge Well	Contaminant exposure	Hazard Identification HS-026 Ergonomic Program	• Wear Nitrile/neoprene gloves and dispose of gloves after use and wash hands. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
7. Drum Purge Water and Soil Cuttings	Contaminant Contact, Cuts or Abrasions Heavy Lifting , Slips/Trips/Falls	Container Management HS–003 Manual Lifting HS–025 Ergonomic Program	• Wear proper PPE during sampling including nitrile gloves and safety glasses and face shield as appropriate. • Use proper dollies or drum moving tools. • Use applicable tools to open/close drum lids. • Do not handle drums with bulging sides. • Dispose of gloves after use and wash hands. • Wear work gloves over nitrile gloves. • Use proper lifting techniques. • Ask fellow worker for help. • Keep trafficked areas free from slip/trip/fall hazards.
8. Equipment Decontamination	Contaminant Contact, Cuts or Abrasions, Slips/Trips/Falls	Hazard Identification HS-026 Ergonomic Program	• Wear nitrile gloves and glasses to provide eye protection from splashing. • Wash hands immediately after use. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
9. Equipment Demobilization	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.

Job Hazard Analysis

Task	**NAPL Gauging and Recovery**		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	**Client Name**	National Fuel Gas
GEI Project Manager	Richard Frappa	**PM Review Date**	
Tools/Equipment Used	Pump, generator, compressor, interface probe, tubing cutter, bailer, string, scissors		
Task Specific Training	GEI Annual Safety Training (HAZWOPER), First Aid/CPR		
Personal Protective Equipment (PPE)	☐ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☒Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☒ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves Nitrile/Cut resistant ☐Respirator ☒Other face shield		
JHA Prepared By: Josh Prygon	**Date:**	**Approved by: *RSM to complete***	**Date:**

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Carry Equipment and Set-up	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.
2. Cut Tubing/String	Cuts/Scrapes, pinch points	Non-Powered Hand Tools HS-008a	• Wear cut resistant gloves. • Using safe cutting tools such as safety scissors are snips are best practices. • Keep free hand out of the way. • Secure the item being cut. • Use only sharp blades; dull blades require more force that results in less control. • Do not put the sharp cutting tool in your pocket.

[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (Isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
3. Set up pump, generator and compressor, if needed.	Heavy Lifting, Slips/Trips/Falls	Manual Lifting HS–025 Ergonomic Program	• Check air compressor hoses before use. • Position the generator downwind of the work area. • Keep trafficked areas free from slip/trip/fall hazards.
4. Fuel generator	Heavy Lifting, Slips/Trips/Falls	Manual Lifting HS–025 Ergonomic Program	• Turn off the generator and allow to cool at least 15-minutes before refueling. • Gasoline used to refill the generator will be stored in an OSHA-approved portable fuel container (safety can).
5. Secure string to bailer and lower bailer down well to collect NAPL	Contaminant Contact, Awkward position, Slips/Trips/Falls, Heavy lifting	Benzene Awareness Program Ergonomic Program	• Wear proper PPE during sampling including nitrile gloves, Tyvek or Tyvek apron with sleeves, and safety glasses and face shield as appropriate. • Keep trafficked areas free from slip/trip/fall hazards. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.
6. Drum Purged NAPL	Contaminant Contact, Cuts or Abrasions Heavy Lifting , Slips/Trips/Falls	Container Management HS–003 Manual Lifting HS–025 Ergonomic Program Benzene Awareness Program	• Wear proper PPE during sampling including nitrile gloves, Tyvek or Tyvek apron with sleeves , and safety glasses and face shield as appropriate. • Use proper dollies or drum moving tools. • Use applicable tools to open/close drum lids. • Do not handle drums with bulging sides. • Dispose of gloves after use and wash hands. • Wear work gloves over nitrile gloves. • Use proper lifting techniques. • Ask fellow worker for help. • Keep trafficked areas free from slip/trip/fall hazards.
7. Equipment Decontamination	Contaminant Contact, Cuts or Abrasions, Slips/Trips/Falls	Hazard Identification HS–026 Benzene Awareness Program Ergonomic Program	• Wear nitrile gloves and glasses to provide eye protection from splashing. • Wash hands immediately after use. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
8. Equipment Demobilization	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.

Job Hazard Analysis

Task	**Sediment Sampling – Grab Sampler**		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	**Client Name**	National Fuel Gas
GEI Project Manager	Richard Frappa	**PM Review Date**	
Tools/Equipment Used	Sediment grab sampler, rope		
Task Specific Training	Annual Safety Training (Field or HAZWOPER), First Aid/CPR		
Personal Protective Equipment (PPE)	☐ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☐Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves Nitrile ☐Respirator ☐Other		

JHA Prepared By: Josh Prygon	**Date:**	**Approved by: *RSM to complete***	**Date:**

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Carry Equipment and Set-up	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.
2. Grab Sampler Use	Cuts and lacerations/pinch points	Non-Powered Hand Tools HS-008a	• Use proper lifting techniques. • Do not remove equipment guards on equipment. • Take regular breaks and do not work in unusual positions for long periods of time. • Inspect equipment or tools prior to use. Tag and remove from service if tool is damaged.
3. Soil Sampling	Contaminant Exposure, Cuts/Scrapes, Repetition,	Hazard Identification HS-026	• Dispose of gloves after use and wash hands. • Wear work gloves over nitrile gloves. • Excavation entry will be allowed only with proper sloping or

[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (Isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
	Slips/Trips/Falls		shoring. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
4. Drum Soil Cutting	Contaminant Contact, Cuts or Abrasions Heavy Lifting , Slips/Trips/Falls	Container Management HS–003 Manual Lifting HS–025 Ergonomic Program	• Wear proper PPE during sampling including nitrile gloves and safety glasses and face shield as appropriate. • Use proper dollies or drum moving tools. • Use applicable tools to open/close drum lids. • Do not handle drums with bulging sides. • Dispose of gloves after use and wash hands. • Wear work gloves over nitrile gloves. • Use proper lifting techniques. • Ask fellow worker for help. • Keep trafficked areas free from slip/trip/fall hazards.
5. Pack Soil Samples	Heavy lifting, cuts, or abrasions	Manual Lifting HS–025 Ergonomic Program	• Confirm that the lids on all bottles are tight (will not leak). • Place cushioning/absorbent material in the bottom of the cooler and then place the containers in the cooler with sufficient space to allow for the addition of cushioning between the containers. • Use proper dollies or other lifting tools. • Use proper lifting techniques. • Take regular breaks and do not work in unusual positions for long periods of time. • If glassware breaks, dispose of in puncture-resistant containers. Use cut resistant gloves to handle any broken glass.
6. Equipment Decontamination	Contaminant Contact, Cuts or Abrasions, Slips/Trips/Falls	Hazard Identification HS–026 Ergonomic Program	• Wear nitrile gloves and glasses to provide eye protection from splashing. • Wash hands immediately after use. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
7. Demobilize from site	Personal/material security, slips/trips/falls	Hazard Identification HS-026	• Confirm all materials are secured/labeled, including drums and any equipment left on site. • Confirm that any open trenches/pits are properly marked or barricaded. • Clean all equipment/tools prior to leaving. • Secure any gates/locks. • Notify project manager (or designee) you are leaving the site.

Job Hazard Analysis

Task	**Sediment Sampling - Shovel**		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	**Client Name**	National Fuel Gas
GEI Project Manager	Richard Frappa	**PM Review Date**	
Tools/Equipment Used	Shovel, sample jars		
Task Specific Training	Annual Safety Training (Field or HAZWOPER), First Aid/CPR		
Personal Protective Equipment (PPE)	☐ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☐Hearing Protection ☒ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves Nitrile ☐Respirator ☐Other		

JHA Prepared By: Josh Prygon	**Date:**	**Approved by: *RSM to complete***	**Date:**

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Carry Equipment and Set-up	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.
2. Working in Water	Drowning, hypothermia	Water Safety HS-017	• Buddy system shall be in use. • Become familiar and comfortable when walking in water with uneven and slippery surfaces. • Be aware of your surroundings. • Avoid going in water deeper than mid-thigh (some waders are chest waders). • Wear PPE, including PFD, gloves, wading boots, and waders. • Avoid going in water deeper than waders. • Avoid areas with a combination of deep water and fast currents.

[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (Isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
			• Become familiar and comfortable when walking in streams.
3. Shovel Use	Cuts and lacerations/pinch points	Non-Powered Hand Tools HS-008a	• Use proper lifting techniques. • Do not remove equipment guards on equipment. • Take regular breaks and do not work in unusual positions for long periods of time. • Inspect equipment or tools prior to use. Tag and remove from service if tool is damaged.
4. Soil Sampling	Cuts/Scrapes, Repetition, Slips/Trips/Falls	Hazard Identification HS-026	• Dispose of gloves after use and wash hands. • Wear work gloves over nitrile gloves. • Excavation entry will be allowed only with proper sloping or shoring. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
5. Pack Soil Samples	Heavy lifting, cuts, or abrasions	Manual Lifting HS–025 Ergonomic Program	• Confirm that the lids on all bottles are tight (will not leak). • Place cushioning/absorbent material in the bottom of the cooler and then place the containers in the cooler with sufficient space to allow for the addition of cushioning between the containers. • Use proper dollies or other lifting tools. • Use proper lifting techniques. • Take regular breaks and do not work in unusual positions for long periods of time. • If glassware breaks, dispose of in puncture-resistant containers. Use cut resistant gloves to handle any broken glass.
6. Equipment Cleanup	Cuts or Abrasions, Slips/Trips/Falls	Hazard Identification HS-026 Ergonomic Program	• Wear nitrile gloves and glasses to provide eye protection from splashing. • Wash hands immediately after use. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
7. Demobilize from site	Personal/material security, slips/trips/falls	Hazard Identification HS-026	• Confirm all materials are secured/labeled, including drums and any equipment left on site. • Confirm that any open trenches/pits are properly marked or barricaded. • Clean all equipment/tools prior to leaving.

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
			• Secure any gates/locks. • Notify project manager (or designee) you are leaving the site.

Job Hazard Analysis

Task	**Site Mobilization - Walking**		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	**Client Name**	National Fuel Gas
GEI Project Manager	Richard Frappa	**PM Review Date**	
Tools/Equipment Used			
Task Specific Training	Annual Safety Training (Field), First Aid/CPR		
Personal Protective Equipment (PPE)	☐ Hard Hat ☐Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☐Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☐ Gloves Enter Type(s) - e.g., Nitrile, Cut Resistant, Latex ☐Respirator Enter Type/Cartridges - (e.g., Half-face, P-100) ☐Other		

JHA Prepared By: Josh Prygon	**Date:**	**Approved by: *RSM to complete***	**Date:**

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Walk to Site	Slip/trips/falls, repetition, heat stress, cold stress, biological hazards	Biological Hazards HS-001 Inclement Weather HS-010 Manual Lifting HS-025 Hazard Identification HS-026 GEI Cold Stress Program GEI Heat Stress Program GEI Ergonomic Program	• Follow the rules of the road and obey signs and signals. • Walk on sidewalks whenever they are available. • If there is no sidewalk, walk facing traffic and as far from traffic as possible. • Cross streets at crosswalks or intersections. Look for cars in all directions, including those turning left or right. • If a crosswalk or intersection is not available, locate a well-lit area where you have the best view of traffic. Wait for a gap in traffic that allows enough time to cross safely; continue watching for traffic as you cross. • Watch for cars entering or exiting driveways, or backing up in parking lots. • Avoid walking on uneven, steeply sloped or debris ridden ground surfaces. • Discuss action plan prior to severe weather.

[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (Isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
2. Carrying Equipment/Site Setup	Heavy lifting, strains/sprains, slip/trips/falls, pinch points	Non-Powered Hand Tools HAS-008a Manual Lifting HS-025 GEI Ergonomic Program	• Use proper lifting techniques. • Check route for clearance of trip hazards. • Wear appropriate footwear and gloves to protect hands against sharp edges and soft tissue injuries. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.
3. Demobilize from Site	Personal/material security, slips/trips/falls	Hazard Identification HS-026	• Confirm all materials are secured/labeled, including drums and any equipment left on site. • Confirm that any open trenches/pits are properly marked or barricaded. • Clean all equipment/tools prior to leaving. • Secure any gates/locks. • Notify project manager (or designee) you are leaving the site.

Job Hazard Analysis

Task	**Site Surveying**		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	**Client Name**	National Fuel Gas
GEI Project Manager	Richard Frappa	**PM Review Date**	
Tools/Equipment Used	GPS receiver, level, rod, measuring tape, total station, notepad/camera/tablet		
Task Specific Training	Annual Safety Training (Field or Hazwoper), First Aid/CPR		
Personal Protective Equipment (PPE)	☐ Hard Hat ☐Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☐Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☐ Gloves Enter Type(s) - e.g., Nitrile, Cut Resistant, Latex ☐Respirator Enter Type/Cartridges - (e.g., Half-face, P-100) ☐Other		
JHA Prepared By: Josh Prygon	**Date:**	**Approved by: *RSM to complete***	**Date:**

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Clothing Treatment - Permethrin	Chemical Exposure	Biological Hazards HS-001 (Permethrin Guidance)	• Read application instructions thoroughly and apply according to the directions. Do not over-treat products. • Permethrin treatments are for meant only for fabrics, do not apply directly to the skin. • Apply product to clothing and gear outdoors in a well-ventilated area protected from wind. • Hang treated clothing outdoors and allow for them to dry completely before wearing. • Do not spray directly on clothing that you are wearing. • Do not wear clothing or use gear that is not completely dry. • Wash treated clothing separate from other garments. • Retreat clothing and gear after six washings or six weeks of exposure to sun and air.
2. Carrying Equipment/Site Setup	Heavy lifting, strains/sprains, slip/trips/falls, pinch points	Non-Powered Hand Tools HAS-008a Manual Lifting HS-025 GEI Ergonomic Program	• Use proper lifting techniques. • Check route for clearance of trip hazards. • Wear appropriate footwear and gloves to protect hands against sharp edges and soft tissue injuries. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances.

[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (Isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
			• Do not overreach when picking up or placing items. • Use the buddy system when necessary.
3. Site Walk/Survey	Slip/trips/falls, repetition, heat stress, cold stress, biological hazards	Biological Hazards HS-001 Inclement Weather HS-010 Manual Lifting HS-025 Hazard Identification HS-026 GEI Cold Stress Program GEI Heat Stress Program GEI Ergonomic Program	• Wear steel-toed boots and high visibility reflective vest when traversing work areas. • Avoid walking on uneven, steeply sloped or debris ridden ground surfaces. • Avoid areas infested with poisonous plants. Use a barrier cream to provide some protection against poisonous plants. If exposed to a poisonous plant, wash with soap and water or a product such as Technu. Use a tool or grab stick to move branches instead of touching them directly. • Discuss action plan prior to severe weather. • Apply insect repellent and poisonous plant barrier cream prior to the start of any field work and as needed throughout the workday. • Use the buddy system and have a check in plan with project team and/or project manager. • Bring adequate hydration for the workday (water, fluids with electrolytes). • Apply sunscreen regularly or wear a hat and long sleeve to reduce sun exposure. • Plan an area to take breaks with natural shade or provided by pop up structure. • Bring backpack to carry your fluids, snacks, first aid kits, etc. for larger project sites. • Some project sites may have mammals such as stray or domesticated dogs and cats, raccoons, rats, snakes, etc. All employees should use discretion and attempt to remove avoid any contact with these animals.
4. Demobilize from Site	Personal/material security, slips/trips/falls	Hazard Identification HS-026	• Confirm all materials are secured/labeled, including drums and any equipment left on site. • Confirm that any open trenches/pits are properly marked or barricaded. • Clean all equipment/tools prior to leaving. • Secure any gates/locks. • Notify project manager (or designee) you are leaving the site.

Job Hazard Analysis

Task	**Soil Sampling – Drill Rig**		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	**Client Name**	National Fuel Gas
GEI Project Manager	Richard Frappa	**PM Review Date**	
Tools/Equipment Used	Geoprobe Drill Rig (operated by subcontractor), PID, measuring tape, metal spoon/spatula		
Task Specific Training	GEI Annual Safety Training (Field or HAZWOPER), First Aid/CPR		
Personal Protective Equipment (PPE)	☒ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☒Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves Nitrile, Cut resistant ☒Respirator if action levels are exceeded ☐Other		

JHA Prepared By: Josh Prygon	**Date:**	**Approved by: *RSM to complete***	**Date:**

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Mobilize On Site/Equipment Setup	Slips/trips/falls, stuck by, crushing hazards	Hazard Identification HS-026	• Wear PPE that properly fits, is in good condition, and is appropriate for the activities and hazards. • Maintain good visibility of the work area. • Avoid walking on uneven, steeply sloped, or debris ridden ground surfaces. • Plan tasks prior to preforming them including an activity hazard analysis. • Keep trafficked areas free from slip/trip/fall hazards. • Wear shoes with traction. • Avoid traversing steep areas in slippery conditions. • Do not carry heavy objects to work areas, on steeply sloped areas, or where steep areas must be traversed to reach work areas.

[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (Isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
2. Utility survey	Shock, Explosion, Fire	Utility Mark Out HS-014	• Confirm that all utilities have been properly marked prior to beginning work. • If a utility strike occurs, call the appropriate authorities. immediately. Clear the area and do not delay.
3. Observe contractor activities	Struck by/crushing hazards	Working Around Heavy Equipment HS-018	• Identify yourself and your work location to heavy equipment operators, so they may incorporate you into their operations. • Coordinate hand signals with operators. • Confirm equipment inspection has been completed. • Stay Alert! Pay attention to equipment backup alarms and swing radii. • Position yourself in a safe location. • Confirm adequate clearance from overhead utilities. • Notify the contractor immediately if any problems arise. • Do not stand or sit under suspended loads or near any pressurized equipment lines. • Do not operate cellular telephones in the vicinity of heavy equipment operation.
4. Cutting Soil Core Liners	Cuts/lacerations, contaminant exposure	Non-Powered Hand Tools HS-008a Ergonomic Program	• Eliminate the hazard by having the drillers open the cores/liners for you. o Keep a safe distance from the core while they are cutting the liner (hands, face, and body). o Do not stand on either side of the core in the direction they are cutting. o Make sure your safety glasses are on, in case of material splatter. • If you are cutting the liner, use a liner cutting tool, safety shears, or at a minimum hooked blades on a self-retracting knife. • When using cutting tools, follow these safety precautions: o Wear cut resistant gloves. o Keep free hand out of the way. o Secure the liner if cutting through thick material. o Use only sharp blades; dull blades require more force that results in less control. o Do not put the sharp cutting tool in your pocket. • Use care when evaluating cores, as the plastic that has been cut can be sharp.
5. Sample Soil	Contaminant Exposure, Cuts/Scrapes, Repetition,	Hazard Identification HS-026/Container Management HS-003	• Dispose of gloves after use and wash hands. • Wear work gloves over nitrile gloves. • Excavation entry will be allowed only with proper sloping or

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
	Slips/Trips/Falls		shoring. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
6. Drum Soil Cuttings	Contaminant Contact, Cuts or Abrasions Heavy Lifting , Slips/Trips/Falls	Container Management HS–003 Manual Lifting HS–025 Ergonomic Program	• Wear proper PPE during sampling including nitrile gloves and safety glasses and face shield as appropriate. • Use proper dollies or drum moving tools. • Use applicable tools to open/close drum lids. • Do not handle drums with bulging sides. • Dispose of gloves after use and wash hands. • Wear work gloves over nitrile gloves. • Use proper lifting techniques. • Ask fellow worker for help. • Keep trafficked areas free from slip/trip/fall hazards.
7. Pack Soil Samples	Heavy lifting, cuts, or abrasions	Manual Lifting HS–025 Ergonomic Program	• Confirm that the lids on all bottles are tight (will not leak). • Place cushioning/absorbent material in the bottom of the cooler and then place the containers in the cooler with sufficient space to allow for the addition of cushioning between the containers. • Use proper dollies or other lifting tools. • Use proper lifting techniques. • Take regular breaks and do not work in unusual positions for long periods of time. • If glassware breaks, dispose of in puncture-resistant containers. Use cut resistant gloves to handle any broken glass.
8. Equipment Decontamination	Contaminant Contact, Cuts or Abrasions, Slips/Trips/Falls	Hazard Identification HS–026 Ergonomic Program	• Wear nitrile gloves and glasses to provide eye protection from splashing. • Wash hands immediately after use. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
9. Demobilize from site	Personal/material security, slips/trips/falls	Hazard Identification HS-026	• Confirm all materials are secured/labeled, including drums and any equipment left on site. • Confirm that any open trenches/pits are properly marked or barricaded. • Clean all equipment/tools prior to leaving. • Secure any gates/locks. • Notify project manager (or designee) you are leaving the site.

Job Hazard Analysis

Task	Soil Sampling – Hand Auger		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	Client Name	National Fuel Gas
GEI Project Manager	Richard Frappa	PM Review Date	
Tools/Equipment Used	Hand auger, hand tools, sampling supplies, decon equipment		
Task Specific Training	Annual Safety Training (Field or HAZWOPER), First Aid/CPR		
Personal Protective Equipment (PPE)	☐ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☐Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves Nitrile ☐Respirator ☐Other		

JHA Prepared By: Josh Prygon	Date:	Approved by: *RSM to complete*	Date:

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Carry Equipment and Set-up	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.
2. Hand Auger Use	Cuts and lacerations/pinch points	Non-Powered Hand Tools HS-008a	• Use proper lifting techniques. • Do not remove equipment guards on equipment. • Take regular breaks and do not work in unusual positions for long periods of time. • Inspect equipment or tools prior to use. Tag and remove from service if tool is damaged.
3. Soil Sampling	Contaminant Exposure, Cuts/Scrapes, Repetition,	Hazard Identification HS-026/Container Management HS-003	• Dispose of gloves after use and wash hands. • Wear work gloves over nitrile gloves. • Excavation entry will be allowed only with proper sloping or

[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (Isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
	Slips/Trips/Falls		shoring. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
4. Drum Soil Cutting	Contaminant Contact, Cuts or Abrasions Heavy Lifting , Slips/Trips/Falls	Container Management HS–003 Manual Lifting HS–025 Ergonomic Program	• Wear proper PPE during sampling including nitrile gloves and safety glasses and face shield as appropriate. • Use proper dollies or drum moving tools. • Use applicable tools to open/close drum lids. • Do not handle drums with bulging sides. • Dispose of gloves after use and wash hands. • Wear work gloves over nitrile gloves. • Use proper lifting techniques. • Ask fellow worker for help. • Keep trafficked areas free from slip/trip/fall hazards.
5. Pack Soil Samples	Heavy lifting, cuts, or abrasions	Manual Lifting HS–025 Ergonomic Program	• Confirm that the lids on all bottles are tight (will not leak). • Place cushioning/absorbent material in the bottom of the cooler and then place the containers in the cooler with sufficient space to allow for the addition of cushioning between the containers. • Use proper dollies or other lifting tools. • Use proper lifting techniques. • Take regular breaks and do not work in unusual positions for long periods of time. • If glassware breaks, dispose of in puncture-resistant containers. Use cut resistant gloves to handle any broken glass.
6. Equipment Decontamination	Contaminant Contact, Cuts or Abrasions, Slips/Trips/Falls	Hazard Identification HS–026 Ergonomic Program	• Wear nitrile gloves and glasses to provide eye protection from splashing. • Wash hands immediately after use. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.
7. Demobilize from site	Personal/material security, slips/trips/falls	Hazard Identification HS-026	• Confirm all materials are secured/labeled, including drums and any equipment left on site. • Confirm that any open trenches/pits are properly marked or barricaded. • Clean all equipment/tools prior to leaving. • Secure any gates/locks. • Notify project manager (or designee) you are leaving the site.

Job Hazard Analysis

Task	**Surface Water Sampling**		
Project Name/Number	2406810 - National Fuel GLLA Lower Scajaquada Ck RI/FS OU	**Client Name**	National Fuel Gas
GEI Project Manager	Richard Frappa	**PM Review Date**	
Tools/Equipment Used	Sample jars and cooler		
Task Specific Training	GEI Annual Safety Training (Field or HAZWOPER), First Aid/CPR		
Personal Protective Equipment (PPE)	☐ Hard Hat ☒Safety Glasses ☒Safety-Toed Boots ☒ Reflective Vest ☐Hearing Protection ☐ Personal Flotation Device (PFD) ☐ Face Shield ☐ Chaps ☐ Tyvek clothing/boots ☐ Flame Resistant Clothing ☒ Gloves Nitrile/Cut resistant ☐Respirator ☒Other Waders (as needed)		

JHA Prepared By: Josh Prygon	**Date:**	**Approved by: *RSM to complete***	**Date:**

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
1. Carry Equipment and Set-up	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions. • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.

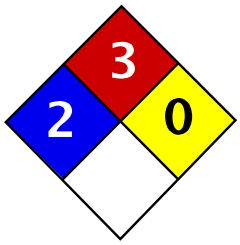
[1] Use the hierarchy of controls to determine applicable hazard controls for the task in order of most effective to least effective: Elimination (physically remove the hazard), Substitution (replace the hazard), Engineering Controls (Isolate the team from the hazard), Administrative Controls (change the way people work), PPE (personal protective equipment).

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
2. Sampling Mobilization	Cuts/Scrapes, Insect Bites, Slips/trips/falls, repetition, heat stress, cold stress, biological hazards	Biological Hazards HS-001 Inclement Weather HS-010 Manual Lifting HS-025 Hazard Identification HS-026 GEI Ergonomic Program	• Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Open cover slowly in case insects inhabit the area. • Wear steel-toed boots and high visibility reflective vest when traversing work areas. • Avoid walking on uneven, steeply sloped or debris ridden ground surfaces. • Discuss action plan prior to severe weather. • Apply insect repellent and poisonous plant barrier cream prior to the start of any field work and as needed throughout the workday. • Use the buddy system and have a check in plan with project team and/or project manager. • Bring adequate hydration for the workday (water, fluids with electrolytes). • Apply sunscreen regularly or wear a hat and long sleeve to reduce sun exposure. • Plan an area to take breaks with natural shade or provided by pop up structure. • Bring backpack to carry your fluids, snacks, first aid kits, etc. for larger project sites. • Some project sites may have mammals such as stray or domesticated dogs and cats, raccoons, rats, snakes, etc. All employees should use discretion and attempt to remove avoid any contact with these animals. • Wear proper footwear and PPE prior to start of project work • Become familiar and comfortable when walking in water with uneven and slippery surfaces. • Be aware of your surroundings. • Avoid going in water deeper than mid-thigh (some waders are chest waders). • If wading is necessary to collect surface water samples, see Wading JHA.
3. Collect Samples	Awkward position, contaminant exposure	Hazard Identification HS-026 Ergonomic Program	• Wear Nitrile/neoprene gloves and dispose of gloves after use and wash hands. • Take regular breaks and do not work in unusual positions for long periods of time. • Keep trafficked areas free from slip/trip/fall hazards.

Task Steps	Potential Hazards	GEI SOP or Program	Hazard Controls[1]
4. Pack Samples	Heavy lifting, cuts, or abrasions	Manual Lifting HS–025 Ergonomic Program	• Confirm that the lids on all bottles are tight (will not leak). • Place cushioning/absorbent material in the bottom of the cooler and then place the containers in the cooler with sufficient space to allow for the addition of cushioning between the containers. • Use proper dollies or other lifting tools. • Use proper lifting techniques. • Take regular breaks and do not work in unusual positions for long periods of time. • If glassware breaks, dispose of in puncture-resistant containers. Use cut resistant gloves to handle any broken glass.
5. Equipment Demobilization	Heavy lifting, Cuts/Scrapes, Slips/Trips/Falls, Pinch points	Non-Powered Hand Tools HS-008a Manual Lifting HS-025 Ergonomic Program	• User proper lifting techniques. • Keep trafficked areas free from slip/trip/fall hazards. • Wear the proper type of glove to protect hands against sharp edges and skin/soft tissue injuries. • Wear appropriate footwear. • Be aware of hard to grip and hold items that may force your hand or wrist into awkward, stressful positions . • Take breaks when carrying items frequently and/or for long distances. • Do not overreach when picking up or placing items. • Use the buddy system when necessary.

Appendix D

Safety Data Sheets



Health	2
Fire	3
Reactivity	0
Personal Protection	H

Material Safety Data Sheet
Benzene MSDS

Section 1: Chemical Product and Company Identification

Product Name: Benzene

Catalog Codes: SLB1564, SLB3055, SLB2881

CAS#: 71-43-2

RTECS: CY1400000

TSCA: TSCA 8(b) inventory: Benzene

CI#: Not available.

Synonym: Benzol; Benzine

Chemical Name: Benzene

Chemical Formula: C6-H6

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call:
1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Benzene	71-43-2	100

Toxicological Data on Ingredients: Benzene: ORAL (LD50): Acute: 930 mg/kg [Rat]. 4700 mg/kg [Mouse]. DERMAL (LD50): Acute: >9400 mg/kg [Rabbit]. VAPOR (LC50): Acute: 10000 ppm 7 hours [Rat].

Section 3: Hazards Identification

Potential Acute Health Effects:
Very hazardous in case of eye contact (irritant), of inhalation. Hazardous in case of skin contact (irritant, permeator), of ingestion. Inflammation of the eye is characterized by redness, watering, and itching.

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: Classified A1 (Confirmed for human.) by ACGIH, 1 (Proven for human.) by IARC.
MUTAGENIC EFFECTS: Classified POSSIBLE for human. Mutagenic for mammalian somatic cells. Mutagenic for bacteria and/or yeast.
TERATOGENIC EFFECTS: Not available.
DEVELOPMENTAL TOXICITY: Classified Reproductive system/toxin/female [POSSIBLE].
The substance is toxic to blood, bone marrow, central nervous system (CNS).
The substance may be toxic to liver, Urinary System.
Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4: First Aid Measures

Eye Contact:
Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. WARM water MUST be used. Get medical attention immediately.

Skin Contact:
In case of contact, immediately flush skin with plenty of water. Cover the irritated skin with an emollient. Remove contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation:
If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention if symptoms appear.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek medical attention.

Ingestion:
Do NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. If large quantities of this material are swallowed, call a physician immediately. Loosen tight clothing such as a collar, tie, belt or waistband.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Flammable.

Auto-Ignition Temperature: 497.78°C (928°F)

Flash Points: CLOSED CUP: -11.1°C (12°F). (Setaflash)

Flammable Limits: LOWER: 1.2% UPPER: 7.8%

Products of Combustion: These products are carbon oxides (CO, CO2).

Fire Hazards in Presence of Various Substances:
Highly flammable in presence of open flames and sparks, of heat.
Slightly flammable to flammable in presence of oxidizing materials.
Non-flammable in presence of shocks.

Explosion Hazards in Presence of Various Substances:
Risks of explosion of the product in presence of mechanical impact: Not available.
Risks of explosion of the product in presence of static discharge: Not available.
Explosive in presence of oxidizing materials, of acids.

Fire Fighting Media and Instructions:
Flammable liquid, soluble or dispersed in water.
SMALL FIRE: Use DRY chemical powder.
LARGE FIRE: Use alcohol foam, water spray or fog.

Special Remarks on Fire Hazards:
Extremely flammable liquid and vapor. Vapor may cause flash fire.
Reacts on contact with iodine heptafluoride gas.

Dioxygenyl tetrafluoroborate is as very powferful oxidant. The addition of a small particle to small samples of benzene, at ambient temperature, causes ignition.
Contact with sodium peroxide with benzene causes ignition.
Benzene ignites in contact with powdered chromic anhydride.
Virgorous or incandescent reaction with hydrogen + Raney nickel (above 210 C) and bromine trifluoride.

Special Remarks on Explosion Hazards:
Benzene vapors + chlorine and light causes explosion.
Reacts explosively with bromine pentafluoride, chlorine, chlorine trifluoride, diborane, nitric acid, nitryl perchlorate, liquid oxygen, ozone, silver perchlorate.
Benzene + pentafluoride and methoxide (from arsenic pentafluoride and potassium methoxide) in trichlorotrifluoroethane causes explosion.
Interaction of nitryl perchlorate with benzene gave a slight explosion and flash.
The solution of permanganic acid (or its explosive anhydride, dimaganese heptoxide) produced by interaction of permanganates and sulfuric acid will explode on contact with benzene.
Peroxodisulfuric acid is a very powferful oxidant. Uncontrolled contact with benzene may cause explosion.
Mixtures of peroxomonsulfuric acid with benzene explodes.

Section 6: Accidental Release Measures

Small Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal.

Large Spill:
Flammable liquid.
Keep away from heat. Keep away from sources of ignition. Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not touch spilled material. Prevent entry into sewers, basements or confined areas; dike if needed. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:
Keep locked up.. Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapor/spray. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as oxidizing agents, acids.

Storage:
Store in a segregated and approved area. Keep container in a cool, well-ventilated area. Keep container tightly closed and sealed until ready for use. Avoid all possible sources of ignition (spark or flame).

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection:
Splash goggles. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:

TWA: 0.5 STEL: 2.5 (ppm) from ACGIH (TLV) [United States]
TWA: 1.6 STEL: 8 (mg/m3) from ACGIH (TLV) [United States]
TWA: 0.1 STEL: 1 from NIOSH
TWA: 1 STEL: 5 (ppm) from OSHA (PEL) [United States]
TWA: 10 (ppm) from OSHA (PEL) [United States]
TWA: 3 (ppm) [United Kingdom (UK)]
TWA: 1.6 (mg/m3) [United Kingdom (UK)]
TWA: 1 (ppm) [Canada]
TWA: 3.2 (mg/m3) [Canada]
TWA: 0.5 (ppm) [Canada]Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor:
Aromatic. Gasoline-like, rather pleasant.
(Strong.)

Taste: Not available.

Molecular Weight: 78.11 g/mole

Color: Clear Colorless. Colorless to light yellow.

pH (1% soln/water): Not available.

Boiling Point: 80.1 (176.2°F)

Melting Point: 5.5°C (41.9°F)

Critical Temperature: 288.9°C (552°F)

Specific Gravity: 0.8787 @ 15 C (Water = 1)

Vapor Pressure: 10 kPa (@ 20°C)

Vapor Density: 2.8 (Air = 1)

Volatility: Not available.

Odor Threshold: 4.68 ppm

Water/Oil Dist. Coeff.: The product is more soluble in oil; log(oil/water) = 2.1

Ionicity (in Water): Not available.

Dispersion Properties: See solubility in water, diethyl ether, acetone.

Solubility:
Miscible in alcohol, chloroform, carbon disulfide oils, carbon tetrachloride, glacial acetic acid, diethyl ether, acetone.
Very slightly soluble in cold water.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Heat, ignition sources, incompatibles.

Incompatibility with various substances: Highly reactive with oxidizing agents, acids.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity:
Benzene vapors + chlorine and light causes explosion.
Reacts explosively with bromine pentafluoride, chlorine, chlorine trifluoride, diborane, nitric acid, nitryl perchlorate, liquid oxygen, ozone, silver perchlorate.
Benzene + pentafluoride and methoxide (from arsenic pentafluoride and potassium methoxide) in trichlorotrifluoroethane causes explosion.
Interaction of nitryl perchlorate with benzene gave a slight explosion and flash.
The solution of permanganic acid (or its explosive anhydride, dimaganese heptoxide) produced by interaction of permanganates and sulfuric acid will explode on contact with benzene.
Peroxodisulfuric acid is a very powferful oxidant. Uncontrolled contact with benzene may cause explosion.
Mixtures of peroxomonsulfuric acid with benzene explodes.

Special Remarks on Corrosivity: Not available.

Polymerization: Will not occur.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation.

Toxicity to Animals:
WARNING: THE LC50 VALUES HEREUNDER ARE ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE.
Acute oral toxicity (LD50): 930 mg/kg [Rat].
Acute dermal toxicity (LD50): >9400 mg/kg [Rabbit].
Acute toxicity of the vapor (LC50): 10000 7 hours [Rat].

Chronic Effects on Humans:
CARCINOGENIC EFFECTS: Classified A1 (Confirmed for human.) by ACGIH, 1 (Proven for human.) by IARC.
MUTAGENIC EFFECTS: Classified POSSIBLE for human. Mutagenic for mammalian somatic cells. Mutagenic for bacteria and/or yeast.
DEVELOPMENTAL TOXICITY: Classified Reproductive system/toxin/female [POSSIBLE].
Causes damage to the following organs: blood, bone marrow, central nervous system (CNS).
May cause damage to the following organs: liver, Urinary System.

Other Toxic Effects on Humans:
Very hazardous in case of inhalation.
Hazardous in case of skin contact (irritant, permeator), of ingestion.

Special Remarks on Toxicity to Animals: Not available.

Special Remarks on Chronic Effects on Humans:
May cause adverse reproductive effects (female fertility, Embryotoxic and/or foetotoxic in animal) and birth defects.
May affect genetic material (mutagenic).
May cause cancer (tumorigenic, leukemia))
Human: passes the placental barrier, detected in maternal milk.

Special Remarks on other Toxic Effects on Humans:
Acute Potential Health Effects:
Skin: Causes skin irritation. It can be absorbed through intact skin and affect the liver, blood, metabolism,and urinary system.
Eyes: Causes eye irritation.
Inhalation: Causes respiratory tract and mucous membrane irritation. Can be absorbed through the lungs. May affect behavior/Central and Peripheral nervous systems (somnolence, muscle weakness, general anesthetic, and

other symptoms similar to ingestion), gastrointestinal tract (nausea), blood metabolism, urinary system.
Ingestion: May be harmful if swallowed. May cause gastrointestinal tract irritation including vomiting. May affect behavior/Central and Peripheral nervous systems (convulsions, seizures, tremor, irritability, initial CNS stimulation followed by depression, loss of coordination, dizziness, headache, weakness, pallor, flushing), respiration (breathlessness and chest constriction), cardiovascular system, (shallow/rapid pulse), and blood.

Section 12: Ecological Information

Ecotoxicity: Not available.

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are less toxic than the product itself.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:
Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14: Transport Information

DOT Classification: CLASS 3: Flammable liquid.

Identification: : Benzene UNNA: 1114 PG: II

Special Provisions for Transport: Not available.

Section 15: Other Regulatory Information

Federal and State Regulations:
California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer, birth defects or other reproductive harm, which would require a warning under the statute: Benzene
California prop. 65 (no significant risk level): Benzene: 0.007 mg/day (value)
California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer which would require a warning under the statute: Benzene
Connecticut carcinogen reporting list.: Benzene
Connecticut hazardous material survey.: Benzene
Illinois toxic substances disclosure to employee act: Benzene
Illinois chemical safety act: Benzene
New York release reporting list: Benzene
Rhode Island RTK hazardous substances: Benzene
Pennsylvania RTK: Benzene
Minnesota: Benzene
Michigan critical material: Benzene
Massachusetts RTK: Benzene
Massachusetts spill list: Benzene
New Jersey: Benzene
New Jersey spill list: Benzene
Louisiana spill reporting: Benzene
California Director's list of Hazardous Substances: Benzene

TSCA 8(b) inventory: Benzene
SARA 313 toxic chemical notification and release reporting: Benzene
CERCLA: Hazardous substances.: Benzene: 10 lbs. (4.536 kg)

Other Regulations:
OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).
EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances.

Other Classifications:

WHMIS (Canada):
CLASS B-2: Flammable liquid with a flash point lower than 37.8°C (100°F).
CLASS D-2A: Material causing other toxic effects (VERY TOXIC).

DSCL (EEC):
R11- Highly flammable.
R22- Harmful if swallowed.
R38- Irritating to skin.
R41- Risk of serious damage to eyes.
R45- May cause cancer.
R62- Possible risk of impaired fertility.
S2- Keep out of the reach of children.
S26- In case of contact with eyes, rinse immediately with plenty of water and seek medical advice.
S39- Wear eye/face protection.
S46- If swallowed, seek medical advice immediately and show this container or label.
S53- Avoid exposure - obtain special instructions before use.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 3

Reactivity: 0

Personal Protection: h

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 3

Reactivity: 0

Specific hazard:

Protective Equipment:
Gloves.
Lab coat.
Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate.
Splash goggles.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

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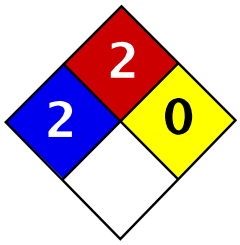

Health	2
Fire	2
Reactivity	0
Personal Protection	E

Material Safety Data Sheet
Naphthalene MSDS

Section 1: Chemical Product and Company Identification

Product Name: Naphthalene

Catalog Codes: SLN1789, SLN2401

CAS#: 91-20-3

RTECS: QJ0525000

TSCA: TSCA 8(b) inventory: Naphthalene

CI#: Not available.

Synonym:

Chemical Name: Not available.

Chemical Formula: C10H8

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call:
1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Naphthalene	91-20-3	100

Toxicological Data on Ingredients: Naphthalene: ORAL (LD50): Acute: 490 mg/kg [Rat]. 533 mg/kg [Mouse]. 1200 mg/kg [Guinea pig]. DERMAL (LD50): Acute: 20001 mg/kg [Rabbit]. VAPOR (LC50): Acute: 170 ppm 4 hour(s) [Rat].

Section 3: Hazards Identification

Potential Acute Health Effects:
Very hazardous in case of ingestion. Hazardous in case of eye contact (irritant), of inhalation. Slightly hazardous in case of skin contact (irritant, permeator). Severe over-exposure can result in death.

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: A4 (Not classifiable for human or animal.) by ACGIH.
MUTAGENIC EFFECTS: Not available.
TERATOGENIC EFFECTS: Not available.
DEVELOPMENTAL TOXICITY: Classified Development toxin [POSSIBLE].
The substance is toxic to blood, kidneys, the nervous system, the reproductive system, liver, mucous membranes, gastrointestinal tract, upper respiratory tract, central nervous system (CNS).
Repeated or prolonged exposure to the substance can produce target organs damage. Repeated exposure to an highly toxic material may produce general deterioration of health by an accumulation in one or many human organs.

Section 4: First Aid Measures

Eye Contact:
Check for and remove any contact lenses. Immediately flush eyes with running water for at least 15 minutes, keeping eyelids open. Cold water may be used. Do not use an eye ointment. Seek medical attention.

Skin Contact:
After contact with skin, wash immediately with plenty of water. Gently and thoroughly wash the contaminated skin with running water and non-abrasive soap. Be particularly careful to clean folds, crevices, creases and groin. Cover the irritated skin with an emollient. If irritation persists, seek medical attention. Wash contaminated clothing before reusing.

Serious Skin Contact: Not available.

Inhalation: Allow the victim to rest in a well ventilated area. Seek immediate medical attention.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek immediate medical attention.

Ingestion:
Do not induce vomiting. Examine the lips and mouth to ascertain whether the tissues are damaged, a possible indication that the toxic material was ingested; the absence of such signs, however, is not conclusive. Loosen tight clothing such as a collar, tie, belt or waistband. If the victim is not breathing, perform mouth-to-mouth resuscitation. Seek immediate medical attention.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Flammable.

Auto-Ignition Temperature: 567°C (1052.6°F)

Flash Points: CLOSED CUP: 88°C (190.4°F). OPEN CUP: 79°C (174.2°F).

Flammable Limits: LOWER: 0.9% UPPER: 5.9%

Products of Combustion: These products are carbon oxides (CO, CO2).

Fire Hazards in Presence of Various Substances: Not available.

Explosion Hazards in Presence of Various Substances:
Risks of explosion of the product in presence of mechanical impact: Not available.
Risks of explosion of the product in presence of static discharge: Not available.

Fire Fighting Media and Instructions:
Flammable solid.
SMALL FIRE: Use DRY chemical powder.
LARGE FIRE: Use water spray or fog. Cool containing vessels with water jet in order to prevent pressure build-up, autoignition or explosion.

Special Remarks on Fire Hazards: Not available.

Special Remarks on Explosion Hazards: Not available.

Section 6: Accidental Release Measures

Small Spill: Use appropriate tools to put the spilled solid in a convenient waste disposal container.

Large Spill:
Flammable solid.
Stop leak if without risk. Do not touch spilled material. Use water spray curtain to divert vapor drift. Prevent entry into sewers, basements or confined areas; dike if needed. Eliminate all ignition sources. Call for assistance on disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:
Keep locked up Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe dust. Avoid contact with eyes Wear suitable protective clothing In case of insufficient ventilation, wear suitable respiratory equipment If ingested, seek medical advice immediately and show the container or the label. Keep away from incompatibles such as oxidizing agents.

Storage:
Flammable materials should be stored in a separate safety storage cabinet or room. Keep away from heat. Keep away from sources of ignition. Keep container tightly closed. Keep in a cool, well-ventilated place. Ground all equipment containing material. Keep container dry. Keep in a cool place.

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Use process enclosures, local exhaust ventilation, or other engineering controls to keep airborne levels below recommended exposure limits. If user operations generate dust, fume or mist, use ventilation to keep exposure to airborne contaminants below the exposure limit.

Personal Protection:
Splash goggles. Lab coat. Dust respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Dust respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:
Israel: TWA: 10 (ppm)
TWA: 10 STEL: 15 (ppm) from ACGIH (TLV) [1995]
TWA: 52 STEL: 79 (mg/m3) from ACGIH [1995]
Australia: STEL: 15 (ppm)
Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Solid. (Crystalline solid.)

Odor: Aromatic.

Taste: Not available.

Molecular Weight: 128.19 g/mole

Color: White.

pH (1% soln/water): Not available.

Boiling Point: 218°C (424.4°F)

Melting Point: 80.2°C (176.4°F)

Critical Temperature: Not available.

Specific Gravity: 1.162 (Water = 1)

Vapor Pressure: Not applicable.

Vapor Density: 4.4 (Air = 1)

Volatility: Not available.

Odor Threshold: 0.038 ppm

Water/Oil Dist. Coeff.: Not available.

Ionicity (in Water): Not available.

Dispersion Properties:
Partially dispersed in hot water, methanol, n-octanol.
Very slightly dispersed in cold water.
See solubility in methanol, n-octanol.

Solubility:
Partially soluble in methanol, n-octanol.
Very slightly soluble in cold water, hot water.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Not available.

Incompatibility with various substances: Highly reactive with oxidizing agents.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity: Not available.

Special Remarks on Corrosivity: May attack some forms of rubber and plastic

Polymerization: No.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation. Ingestion.

Toxicity to Animals:
WARNING: THE LC50 VALUES HEREUNDER ARE ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE.
Acute oral toxicity (LD50): 490 mg/kg [Rat].
Acute dermal toxicity (LD50): 20001 mg/kg [Rabbit].
Acute toxicity of the vapor (LC50): 170 ppm 4 hour(s) [Rat].

Chronic Effects on Humans:
CARCINOGENIC EFFECTS: A4 (Not classifiable for human or animal.) by ACGIH.

DEVELOPMENTAL TOXICITY: Classified Development toxin [POSSIBLE].
The substance is toxic to blood, kidneys, the nervous system, the reproductive system, liver, mucous membranes, gastrointestinal tract, upper respiratory tract, central nervous system (CNS).

Other Toxic Effects on Humans:
Very hazardous in case of ingestion.
Hazardous in case of inhalation.
Slightly hazardous in case of skin contact (irritant, permeator).

Special Remarks on Toxicity to Animals: Not available.

Special Remarks on Chronic Effects on Humans: Not available.

Special Remarks on other Toxic Effects on Humans: Not available.

Section 12: Ecological Information

Ecotoxicity: Ecotoxicity in water (LC50): 305.2 ppm 96 hour(s) [Trout].

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may arise.

Toxicity of the Products of Biodegradation: The products of degradation are more toxic.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:

Section 14: Transport Information

DOT Classification: CLASS 4.1: Flammable solid.

Identification: : Naphthalene, refined : UN1334 PG: III

Special Provisions for Transport: Marine Pollutant

Section 15: Other Regulatory Information

Federal and State Regulations:
Rhode Island RTK hazardous substances: Naphthalene
Pennsylvania RTK: Naphthalene
Florida: Naphthalene
Minnesota: Naphthalene
Massachusetts RTK: Naphthalene
TSCA 8(b) inventory: Naphthalene
TSCA 8(a) PAIR: Naphthalene
TSCA 8(d) H and S data reporting: Naphthalene: 06/01/87
SARA 313 toxic chemical notification and release reporting: Naphthalene: 1%
CERCLA: Hazardous substances.: Naphthalene: 100 lbs. (45.36 kg)

Other Regulations:
OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).

EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances.

Other Classifications:

WHMIS (Canada):
CLASS B-4: Flammable solid.
CLASS D-1B: Material causing immediate and serious toxic effects (TOXIC).
CLASS D-2B: Material causing other toxic effects (TOXIC).

DSCL (EEC):
R36- Irritating to eyes.
R40- Possible risks of irreversible effects.
R48/22- Harmful: danger of serious damage to health by prolonged exposure if swallowed.
R48/23- Toxic: danger of serious damage to health by prolonged exposure through inhalation.
R63- Possible risk of harm to the unborn child.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 2

Reactivity: 0

Personal Protection: E

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 2

Reactivity: 0

Specific hazard:

Protective Equipment:
Gloves.
Lab coat.
Dust respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate.
Splash goggles.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

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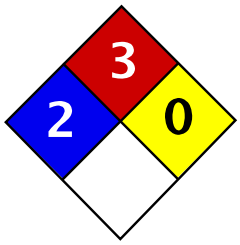

Health	2
Fire	3
Reactivity	0
Personal Protection	H

Material Safety Data Sheet
Toluene MSDS

Section 1: Chemical Product and Company Identification

Product Name: Toluene

Catalog Codes: SLT2857, SLT3277

CAS#: 108-88-3

RTECS: XS5250000

TSCA: TSCA 8(b) inventory: Toluene

CI#: Not available.

Synonym: Toluol, Tolu-Sol; Methylbenzene; Methacide; Phenylmethane; Methylbenzol

Chemical Name: Toluene

Chemical Formula: C6-H5-CH3 or C7-H8

Contact Information:

Sciencelab.com, Inc.
14025 Smith Rd.
Houston, Texas 77396

US Sales: **1-800-901-7247**
International Sales: **1-281-441-4400**

Order Online: ScienceLab.com

CHEMTREC (24HR Emergency Telephone), call: 1-800-424-9300

International CHEMTREC, call: 1-703-527-3887

For non-emergency assistance, call: 1-281-441-4400

Section 2: Composition and Information on Ingredients

Composition:

Name	CAS #	% by Weight
Toluene	108-88-3	100

Toxicological Data on Ingredients: Toluene: ORAL (LD50): Acute: 636 mg/kg [Rat]. DERMAL (LD50): Acute: 14100 mg/kg [Rabbit]. VAPOR (LC50): Acute: 49000 mg/m 4 hours [Rat]. 440 ppm 24 hours [Mouse].

Section 3: Hazards Identification

Potential Acute Health Effects:
Hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion, of inhalation. Slightly hazardous in case of skin contact (permeator).

Potential Chronic Health Effects:
CARCINOGENIC EFFECTS: A4 (Not classifiable for human or animal.) by ACGIH, 3 (Not classifiable for human.) by IARC.
MUTAGENIC EFFECTS: Not available.
TERATOGENIC EFFECTS: Not available.
DEVELOPMENTAL TOXICITY: Not available.
The substance may be toxic to blood, kidneys, the nervous system, liver, brain, central nervous system (CNS). Repeated or prolonged exposure to the substance can produce target organs damage.

Section 4: First Aid Measures

Eye Contact:
Check for and remove any contact lenses. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Get medical attention.

Skin Contact:
In case of contact, immediately flush skin with plenty of water. Cover the irritated skin with an emollient. Remove contaminated clothing and shoes. Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention.

Serious Skin Contact:
Wash with a disinfectant soap and cover the contaminated skin with an anti-bacterial cream. Seek immediate medical attention.

Inhalation:
If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Get medical attention.

Serious Inhalation:
Evacuate the victim to a safe area as soon as possible. Loosen tight clothing such as a collar, tie, belt or waistband. If breathing is difficult, administer oxygen. If the victim is not breathing, perform mouth-to-mouth resuscitation. WARNING: It may be hazardous to the person providing aid to give mouth-to-mouth resuscitation when the inhaled material is toxic, infectious or corrosive. Seek medical attention.

Ingestion:
Do NOT induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. If large quantities of this material are swallowed, call a physician immediately. Loosen tight clothing such as a collar, tie, belt or waistband.

Serious Ingestion: Not available.

Section 5: Fire and Explosion Data

Flammability of the Product: Flammable.

Auto-Ignition Temperature: 480°C (896°F)

Flash Points: CLOSED CUP: 4.4444°C (40°F). (Setaflash) OPEN CUP: 16°C (60.8°F).

Flammable Limits: LOWER: 1.1% UPPER: 7.1%

Products of Combustion: These products are carbon oxides (CO, CO2).

Fire Hazards in Presence of Various Substances:
Flammable in presence of open flames and sparks, of heat.
Non-flammable in presence of shocks.

Explosion Hazards in Presence of Various Substances:
Risks of explosion of the product in presence of mechanical impact: Not available.
Risks of explosion of the product in presence of static discharge: Not available.

Fire Fighting Media and Instructions:
Flammable liquid, insoluble in water.
SMALL FIRE: Use DRY chemical powder.
LARGE FIRE: Use water spray or fog.

Special Remarks on Fire Hazards: Not available.

Special Remarks on Explosion Hazards:
Toluene forms explosive reaction with 1,3-dichloro-5,5-dimethyl-2,4-imidazolididione; dinitrogen tetraoxide;

concentrated nitric acid, sulfuric acid + nitric acid; N_2O_4; $AgClO_4$; BrF_3; Uranium hexafluoride; sulfur dichloride. Also forms an explosive mixture with tetranitromethane.

Section 6: Accidental Release Measures

Small Spill: Absorb with an inert material and put the spilled material in an appropriate waste disposal.

Large Spill:
Toxic flammable liquid, insoluble or very slightly soluble in water.
Keep away from heat. Keep away from sources of ignition. Stop leak if without risk. Absorb with DRY earth, sand or other non-combustible material. Do not get water inside container. Do not touch spilled material. Prevent entry into sewers, basements or confined areas; dike if needed. Call for assistance on disposal. Be careful that the product is not present at a concentration level above TLV. Check TLV on the MSDS and with local authorities.

Section 7: Handling and Storage

Precautions:
Keep away from heat. Keep away from sources of ignition. Ground all equipment containing material. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Wear suitable protective clothing. In case of insufficient ventilation, wear suitable respiratory equipment. If ingested, seek medical advice immediately and show the container or the label. Avoid contact with skin and eyes. Keep away from incompatibles such as oxidizing agents.

Storage:
Store in a segregated and approved area. Keep container in a cool, well-ventilated area. Keep container tightly closed and sealed until ready for use. Avoid all possible sources of ignition (spark or flame).

Section 8: Exposure Controls/Personal Protection

Engineering Controls:
Provide exhaust ventilation or other engineering controls to keep the airborne concentrations of vapors below their respective threshold limit value. Ensure that eyewash stations and safety showers are proximal to the work-station location.

Personal Protection:
Splash goggles. Lab coat. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves.

Personal Protection in Case of a Large Spill:
Splash goggles. Full suit. Vapor respirator. Boots. Gloves. A self contained breathing apparatus should be used to avoid inhalation of the product. Suggested protective clothing might not be sufficient; consult a specialist BEFORE handling this product.

Exposure Limits:
TWA: 200 STEL: 500 CEIL: 300 (ppm) from OSHA (PEL) [United States]
TWA: 50 (ppm) from ACGIH (TLV) [United States] SKIN
TWA: 100 STEL: 150 from NIOSH [United States]
TWA: 375 STEL: 560 (mg/m^3) from NIOSH [United States]
Consult local authorities for acceptable exposure limits.

Section 9: Physical and Chemical Properties

Physical state and appearance: Liquid.

Odor: Sweet, pungent, Benzene-like.

Taste: Not available.

Molecular Weight: 92.14 g/mole

Color: Colorless.

pH (1% soln/water): Not applicable.

Boiling Point: 110.6°C (231.1°F)

Melting Point: -95°C (-139°F)

Critical Temperature: 318.6°C (605.5°F)

Specific Gravity: 0.8636 (Water = 1)

Vapor Pressure: 3.8 kPa (@ 25°C)

Vapor Density: 3.1 (Air = 1)

Volatility: Not available.

Odor Threshold: 1.6 ppm

Water/Oil Dist. Coeff.: The product is more soluble in oil; log(oil/water) = 2.7

Ionicity (in Water): Not available.

Dispersion Properties: See solubility in water, diethyl ether, acetone.

Solubility:
Soluble in diethyl ether, acetone.
Practically insoluble in cold water.
Soluble in ethanol, benzene, chloroform, glacial acetic acid, carbon disulfide.
Solubility in water: 0.561 g/l @ 25 deg. C.

Section 10: Stability and Reactivity Data

Stability: The product is stable.

Instability Temperature: Not available.

Conditions of Instability: Heat, ignition sources (flames, sparks, static), incompatible materials

Incompatibility with various substances: Reactive with oxidizing agents.

Corrosivity: Non-corrosive in presence of glass.

Special Remarks on Reactivity:
Incompatible with strong oxidizers, silver perchlorate, sodium difluoride, Tetranitromethane, Uranium Hexafluoride.
Frozen Bromine Trifluoride reacts violently with Toluene at -80 deg. C.
Reacts chemically with nitrogen oxides, or halogens to form nitrotoluene, nitrobenzene, and nitrophenol and halogenated products, respectively.

Special Remarks on Corrosivity: Not available.

Polymerization: Will not occur.

Section 11: Toxicological Information

Routes of Entry: Absorbed through skin. Dermal contact. Eye contact. Inhalation. Ingestion.

Toxicity to Animals:
WARNING: THE LC50 VALUES HEREUNDER ARE ESTIMATED ON THE BASIS OF A 4-HOUR EXPOSURE.
Acute oral toxicity (LD50): 636 mg/kg [Rat].
Acute dermal toxicity (LD50): 14100 mg/kg [Rabbit].
Acute toxicity of the vapor (LC50): 440 24 hours [Mouse].

Chronic Effects on Humans:
CARCINOGENIC EFFECTS: A4 (Not classifiable for human or animal.) by ACGIH, 3 (Not classifiable for human.) by IARC.
May cause damage to the following organs: blood, kidneys, the nervous system, liver, brain, central nervous system (CNS).

Other Toxic Effects on Humans:
Hazardous in case of skin contact (irritant), of ingestion, of inhalation.
Slightly hazardous in case of skin contact (permeator).

Special Remarks on Toxicity to Animals:
Lowest Published Lethal Dose:
LDL [Human] - Route: Oral; Dose: 50 mg/kg
LCL [Rabbit] - Route: Inhalation; Dose: 55000 ppm/40min

Special Remarks on Chronic Effects on Humans:
Detected in maternal milk in human. Passes through the placental barrier in human. Embryotoxic and/or foetotoxic in animal. May cause adverse reproductive effects and birth defects (teratogenic). May affect genetic material (mutagenic)

Special Remarks on other Toxic Effects on Humans:
Acute Potential Health Effects:
Skin: Causes mild to moderate skin irritation. It can be absorbed to some extent through the skin.
Eyes: Cauess mild to moderate eye irritation with a burning sensation. Splash contact with eyes also causes conjunctivitis, blepharospasm, corneal edema, corneal abraisons. This usually resolves in 2 days.
Inhalation: Inhalation of vapor may cause respiratory tract irritation causing coughing and wheezing, and nasal discharge. Inhalation of high concentrations may affect behavior and cause central nervous system effects characterized by nausea, headache, dizziness, tremors, restlessness, lightheadedness, exhilaration, memory loss, insomnia, impaired reaction time, drowsiness, ataxia, hallucinations, somnolence, muscle contraction or spasticity, unconsciousness and coma. Inhalation of high concentration of vapor may also affect the cardiovascular system (rapid heart beat, heart palpitations, increased or decreased blood pressure, dysrhythmia,), respiration (acute pulmonary edema, respiratory depression, apnea, asphyxia), cause vision disturbances and dilated pupils, and cause loss of appetite.
Ingestion: Aspiration hazard. Aspiration of Toluene into the lungs may cause chemical pneumonitis. May cause irritation of the digestive tract with nausea, vomiting, pain. May have effects similar to that of acute inhalation.
Chronic Potential Health Effects:
Inhalation and Ingestion: Prolonged or repeated exposure via inhalation may cause central nervous system and cardiovascular symptoms similar to that of acute inhalation and ingestion as well liver damage/failure, kidney damage/failure (with hematuria, proteinuria, oliguria, renal tubular acidosis), brain damage, weight loss, blood (pigmented or nucleated red blood cells, changes in white blood cell count), bone marrow changes, electrolyte imbalances (Hypokalemia, Hypophostatemia), severe, muscle weakness and Rhabdomyolysis.
Skin: Repeated or prolonged skin contact may cause defatting dermatitis.

Section 12: Ecological Information

Ecotoxicity:
Ecotoxicity in water (LC50): 313 mg/l 48 hours [Daphnia (daphnia)]. 17 mg/l 24 hours [Fish (Blue Gill)]. 13 mg/l 96 hours [Fish (Blue Gill)]. 56 mg/l 24 hours [Fish (Fathead minnow)]. 34 mg/l 96 hours [Fish (Fathead minnow)]. 56.8 ppm any hours [Fish (Goldfish)].

BOD5 and COD: Not available.

Products of Biodegradation:
Possibly hazardous short term degradation products are not likely. However, long term degradation products may

arise.

Toxicity of the Products of Biodegradation: The products of degradation are less toxic than the product itself.

Special Remarks on the Products of Biodegradation: Not available.

Section 13: Disposal Considerations

Waste Disposal:
Waste must be disposed of in accordance with federal, state and local environmental control regulations.

Section 14: Transport Information

DOT Classification: CLASS 3: Flammable liquid.

Identification: : Toluene UNNA: 1294 PG: II

Special Provisions for Transport: Not available.

Section 15: Other Regulatory Information

Federal and State Regulations:
California prop. 65: This product contains the following ingredients for which the State of California has found to cause cancer, birth defects or other reproductive harm, which would require a warning under the statute: Toluene
California prop. 65 (no significant risk level): Toluene: 7 mg/day (value)
California prop. 65 (acceptable daily intake level): Toluene: 7 mg/day (value)
California prop. 65: This product contains the following ingredients for which the State of California has found to cause birth defects which would require a warning under the statute: Toluene
Connecticut hazardous material survey.: Toluene
Illinois toxic substances disclosure to employee act: Toluene
Illinois chemical safety act: Toluene
New York release reporting list: Toluene
Rhode Island RTK hazardous substances: Toluene
Pennsylvania RTK: Toluene
Florida: Toluene
Minnesota: Toluene
Michigan critical material: Toluene
Massachusetts RTK: Toluene
Massachusetts spill list: Toluene
New Jersey: Toluene
New Jersey spill list: Toluene
Louisiana spill reporting: Toluene
California Director's List of Hazardous Substances.: Toluene
TSCA 8(b) inventory: Toluene
TSCA 8(d) H and S data reporting: Toluene: Effective date: 10/04/82; Sunset Date: 10/0/92
SARA 313 toxic chemical notification and release reporting: Toluene
CERCLA: Hazardous substances.: Toluene: 1000 lbs. (453.6 kg)

Other Regulations:
OSHA: Hazardous by definition of Hazard Communication Standard (29 CFR 1910.1200).
EINECS: This product is on the European Inventory of Existing Commercial Chemical Substances.

Other Classifications:

WHMIS (Canada):
CLASS B-2: Flammable liquid with a flash point lower than 37.8°C (100°F).
CLASS D-2A: Material causing other toxic effects (VERY TOXIC).

DSCL (EEC):
R11- Highly flammable.
R20- Harmful by inhalation.
S16- Keep away from sources of ignition - No smoking.
S25- Avoid contact with eyes.
S29- Do not empty into drains.
S33- Take precautionary measures against static discharges.

HMIS (U.S.A.):

Health Hazard: 2

Fire Hazard: 3

Reactivity: 0

Personal Protection: h

National Fire Protection Association (U.S.A.):

Health: 2

Flammability: 3

Reactivity: 0

Specific hazard:

Protective Equipment:
Gloves.
Lab coat.
Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate.
Splash goggles.

Section 16: Other Information

References: Not available.

Other Special Considerations: Not available.

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Occupational Health Guideline for Coal Tar Pitch Volatiles

INTRODUCTION

This guideline is intended as a source of information for employees, employers, physicians, industrial hygienists, and other occupational health professionals who may have a need for such information. It does not attempt to present all data; rather, it presents pertinent information and data in summary form.

SUBSTANCE IDENTIFICATION

Anthracene

- Formula: $C_{14}H_{10}$
- Synonyms: None
- Appearance and odor: Pale green solid with a faint aromatic odor.

Phenanthrene

Formula: $C_{14}H_{10}$
Synonyms: None
Appearance and odor: Colorless solid with a faint aromatic odor.

Pyrene

- Formula: $C_{16}H_{10}$
- Synonyms: None
- Appearance: Bright yellow solid

Carbazole

- Formula: $C_{12}H_9N$
- Synonyms: None
- Appearance and odor: Colorless solid with a faint aromatic odor.

Benzo(a)pyrene

- Formula: $C_{20}H_{12}$
- Synonyms: BaP, 3,4-benzopyrene
- Appearance and odor: Colorless solid with a faint aromatic odor.

PERMISSIBLE EXPOSURE LIMIT (PEL)

The current OSHA standard for coal tar pitch volatiles is 0.2 milligram of coal tar pitch volatiles per cubic meter of air (mg/m^3) averaged over an eight-hour work shift. NIOSH has recommended that the permissible exposure limit for coal tar products be reduced to 0.1 mg/m^3 (cyclohexane-extractable fraction) averaged over a work shift of up to 10 hours per day, 40 hours per week, and that coal tar products be regulated as occupational carcinogens. The NIOSH Criteria Document for Coal Tar Products and NIOSH Criteria Document for Coke Oven Emissions should be consulted for more detailed information.

HEALTH HAZARD INFORMATION

- **Routes of exposure**

Coal tar pitch volatiles can affect the body if they are inhaled or if they come in contact with the eyes or skin.

- **Effects of overexposure**

Repeated exposure to coal tar pitch volatiles has been associated with an increased risk of developing bronchitis and cancer of the lungs, skin, bladder, and kidneys. Pregnant women may be especially susceptible to exposure effects associated with coal tar pitch volatiles. Repeated exposure to these materials may also cause sunlight to have a more severe effect on a person's skin. In addition, this type of exposure may cause an allergic skin rash.

- **Reporting signs and symptoms**

A physician should be contacted if anyone develops any signs or symptoms and suspects that they are caused by exposure to coal tar pitch volatiles.

- **Recommended medical surveillance**

The following medical procedures should be made available to each employee who is exposed to coal tar pitch volatiles at potentially hazardous levels:

These recommendations reflect good industrial hygiene and medical surveillance practices and their implementation will assist in achieving an effective occupational health program. However, they may not be sufficient to achieve compliance with all requirements of OSHA regulations.

U.S. DEPARTMENT OF HEALTH AND HUMAN SERVICES
Public Health Service Centers for Disease Control
National Institute for Occupational Safety and Health

U.S. DEPARTMENT OF LABOR
Occupational Safety and Health Administration

September 1978

1. Initial Medical Examination:

—A complete history and physical examination: The purpose is to detect pre-existing conditions that might place the exposed employee at increased risk, and to establish a baseline for future health monitoring. Examination of the oral cavity, respiratory tract, bladder, and kidneys should be stressed. The skin should be examined for evidence of chronic disorders, for premalignant and malignant lesions, and evidence of hyperpigmentation or photosensitivity.

—Urinalysis: Coal tar pitch volatiles are associated with an excess of kidney and bladder cancer. A urinalysis should be obtained to include at a minimum specific gravity, albumin, glucose, and a microscopic on centrifuged sediment, as well as a test for red blood cells.

—Urinary cytology: Coal tar pitch volatiles are associated with an excess of kidney and bladder cancer. Employees having 5 or more years of exposure or who are 45 years of age or older should have a urinary cytology examination.

—Sputum cytology: Coal tar pitch volatiles are associated with an excess of lung cancer. Employees having 10 or more years of exposure or who are 45 years of age or older should have a sputum cytology examination.

—14" x 17" chest roentgenogram: Coal tar pitch volatiles are associated with an excess of lung cancer. Surveillance of the lungs is indicated.

—FVC and FEV (1 sec): Coal tar pitch volatiles are reported to cause an excess of bronchitis. Periodic surveillance is indicated.

—A complete blood count: Due to the possibility of benzene exposure associated with coal tar pitch volatiles, a complete blood count is considered necessary to search for leukemia and aplastic anemia.

—Skin disease: Coal tar pitch volatiles are defatting agents and can cause dermatitis on prolonged exposure. Persons with pre-existing skin disorders may be more susceptible to the effects of these agents.

2. Periodic Medical Examination: The aforementioned medical examinations should be repeated on an annual basis, and semi-annually for employees 45 years of age or older or with 10 or more years' exposure to coal tar pitch volatiles.

• **Summary of toxicology**

Coal tar pitch volatiles (CTPV) are products of the destructive distillation of bituminous coal and contain polynuclear aromatic hydrocarbons (PNA's). These hydrocarbons sublime readily, thereby increasing the amounts of carcinogenic compounds in working areas. Epidemiologic evidence suggests that workers intimately exposed to the products of combustion or distillation of bituminous coal are at increased risk of cancer at many sites. These include cancer of the respiratory tract, kidney, bladder, and skin. In a study of coke oven workers, the level of exposure to CTPV and the length of time exposed were related to the development of cancer. Coke oven workers with the highest risk of cancer were those employed exclusively at topside jobs for 5 or more years, for whom the increased risk of dying from lung cancer was 10-fold; all coke oven workers had a 7-½-fold increase in risk of dying from kidney cancer. Although the causative agent or agents of the cancer in coke oven workers is unidentified, it is suspected that several PNA's in the CTPV generated during the coking process are involved. Certain industrial populations exposed to coal tar products have a demonstrated risk of skin cancer. Substances containing PNA's which may produce skin cancer also produce contact dermatitis; examples are coal tar, pitch, and cutting oils. Although allergic dermatitis is readily induced by PNA's in guinea pigs, it is only rarely reported in humans from occupational contact with PNA's; these have resulted largely from the therapeutic use of coal tar preparations. Components of pitch and coal tar produce cutaneous photosensitization; skin eruptions are usually limited to areas exposed to the sun or ultraviolet light. Most of the phototoxic agents will induce hypermelanosis of the skin; if chronic photodermatitis is severe and prolonged, leukoderma may occur. Some oils containing PNA's have been associated with changes of follicular and sebaceous glands which commonly take the form of acne. There is evidence that exposures to emissions at coke ovens and gas retorts may be associated with an increased occurrence of chronic bronchitis. Coal tar pitch volatiles may be associated with benzene, an agent suspected of causing leukemia and known to cause aplastic anemia.

CHEMICAL AND PHYSICAL PROPERTIES

• **Physical data—Anthracene**

1. Molecular weight: 178.2
2. Boiling point (760 mm Hg): 340 C (644 F)
3. Specific gravity (water = 1): 1.24
4. Vapor density (air = 1 at boiling point of anthracene): 6.15
5. Melting point: 217 C (423 F)
6. Vapor pressure at 20 C (68 F): Less than 1 mm Hg
7. Solubility in water, g/100 g water at 20 C (68 F): Insoluble
8. Evaporation rate (butyl acetate = 1): Not applicable

• **Physical data—Phenanthrene**

1. Molecular weight: 178.2
2. Boiling point (760 mm Hg): 340 C (644 F)
3. Specific gravity (water = 1): 1.18
4. Vapor density (air = 1 at boiling point of phenanthrene): 6.15
5. Melting point: 100.5 C (213 F)
6. Vapor pressure at 20 C (68 F): Less than 1 mm Hg
7. Solubility in water, g/100 g water at 20 C (68 F): Insoluble
8. Evaporation rate (butyl acetate = 1): Not applicable

• **Physical data—Pyrene**

1. Molecular weight: 202.3
2. Boiling point (760 mm Hg): Greater than 360 C (greater than 680 F)

3. Specific gravity (water = 1): 1.28
4. Vapor density (air = 1 at boiling point of pyrene): 6.9
5. Melting point: 150.4 C (303 F)
6. Vapor pressure at 20 C (68 F): Less than 1 mm Hg
7. Solubility in water, g/100 g water at 20 C (68 F): Insoluble
8. Evaporation rate (butyl acetate = 1): Not applicable

• **Physical data—Carbazole**

1. Molecular weight: 167.2
2. Boiling point (760 mm Hg): 355 C (671 F)
3. Specific gravity (water = 1): Greater than 1
4. Vapor density (air = 1 at boiling point of carbazole): 5.8
5. Melting point: 246 C (475 F)
6. Vapor pressure at 20 C (68 F): Less than 1 mm Hg
7. Solubility in water, g/100 g water at 20 C (68 F): Insoluble
8. Evaporation rate (butyl acetate = 1): Not applicable

• **Physical data—Benzo(a)pyrene**

1. Molecular weight: 252.3
2. Boiling point (760 mm Hg): Greater than 360 C (greater than 680 F)
3. Specific gravity (water = 1): Greater than 1
4. Vapor density (air = 1 at boiling point of benzo(a)pyrene): 8.7
5. Melting point: 179 C (354 F)
6. Vapor pressure at 20 C (68 F): Less than 1 mm Hg
7. Solubility in water, g/100 g water at 20 C (68 F): Insoluble
8. Evaporation rate (butyl acetate = 1): Not applicable

• **Reactivity**

1. Conditions contributing to instability: None hazardous
2. Incompatibilities: Contact with strong oxidizers may cause fires and explosions.
3. Hazardous decomposition products: None
4. Special precautions: None

• **Flammability**

1. Flash point: Anthracene: 121 C (250 F) (closed cup); Others: Data not available
2. Autoignition temperature: Anthracene: 540 C (1004 F); Others: Data not available
3. Flammable limits in air, % by volume: Anthracene: Lower: 0.6; Others: Data not available
4. Extinguishant: Foam, dry chemical, and carbon dioxide

• **Warning properties**

Grant states that "coal tar and its various crude fractions appear principally to cause reddening and squamous eczema of the lid margins, with only small erosions of the corneal epithelium and superficial changes in the stroma, which disappear in a month following exposure. Chronic exposure of workmen to tar fumes and dust has been reported to cause conjunctivitis and discoloration of the cornea in the palpebral fissure, either near the limbus or, in extreme cases, across the whole cornea. Occasionally, epithelioma of the lid margin has been attributed to contact with coal tar."

MONITORING AND MEASUREMENT PROCEDURES

• **General**

Measurements to determine employee exposure are best taken so that the average eight-hour exposure is based on a single eight-hour sample or on two four-hour samples. Several short-time interval samples (up to 30 minutes) may also be used to determine the average exposure level. Air samples should be taken in the employee's breathing zone (air that would most nearly represent that inhaled by the employee).

• **Method**

Coal tar products may be sampled by collection on a glass fiber filter with subsequent ultrasonic extraction and weighing. An analytical method for coal tar pitch volatiles is in the *NIOSH Manual of Analytical Methods*, 2nd Ed., Vol. 1, 1977, available from the Government Printing Office, Washington, D.C. 20402 (GPO No. 017-033-00267-3).

RESPIRATORS

• Good industrial hygiene practices recommend that engineering controls be used to reduce environmental concentrations to the permissible exposure level. However, there are some exceptions where respirators may be used to control exposure. Respirators may be used when engineering and work practice controls are not technically feasible, when such controls are in the process of being installed, or when they fail and need to be supplemented. Respirators may also be used for operations which require entry into tanks or closed vessels, and in emergency situations. If the use of respirators is necessary, the only respirators permitted are those that have been approved by the Mine Safety and Health Administration (formerly Mining Enforcement and Safety Administration) or by the National Institute for Occupational Safety and Health.

• In addition to respirator selection, a complete respiratory protection program should be instituted which includes regular training, maintenance, inspection, cleaning, and evaluation.

PERSONAL PROTECTIVE EQUIPMENT

• Employees should be provided with and required to use impervious clothing, gloves, face shields (eight-inch minimum), and other appropriate protective clothing necessary to prevent skin contact with condensed coal tar pitch volatiles, where skin contact may occur.

• If employees' clothing may have become contaminated with coal tar pitch volatiles, employees should change into uncontaminated clothing before leaving the work premises.

• Clothing contaminated with coal tar pitch volatiles

should be placed in closed containers for storage until it can be discarded or until provision is made for the removal of coal tar pitch volatiles from the clothing. If the clothing is to be laundered or otherwise cleaned to remove the coal tar pitch volatiles, the person performing the operation should be informed of coal tar pitch volatiles's hazardous properties.

- Employees should be provided with and required to use splash-proof safety goggles where condensed coal tar pitch volatiles may contact the eyes.

SANITATION

- Workers subject to skin contact with coal tar pitch volatiles should wash with soap or mild detergent and water any areas of the body which may have contacted coal tar pitch volatiles at the end of each work day.
- Employees who handle coal tar pitch volatiles should wash their hands thoroughly with soap or mild detergent and water before eating, smoking, or using toilet facilities.
- Areas in which exposure to coal tar pitch volatiles may occur should be identified by signs or other appropriate means, and access to these areas should be limited to authorized persons.

COMMON OPERATIONS AND CONTROLS

The following list includes some common operations in which exposure to coal tar pitch volatiles may occur and control methods which may be effective in each case:

Operation	Controls
Liberation from extraction and packaging from coal tar fraction of coking	Process enclosure; local exhaust ventilation; general dilution ventilation; personal protective equipment
Use as a binding agent in manufacture of coal briquettes used for fuel; use as a dielectric in the manufacture of battery electrodes, electric-arc furnace electrodes, and electrodes for alumina reduction	Process enclosure; local exhaust ventilation; general dilution ventilation; personal protective equipment
Use in manufacture of roofing felts and papers and roofing	Process enclosure; local exhaust ventilation; general dilution ventilation; personal protective equipment
Use for protective coatings for pipes for underground conduits and drainage; use as a coating on concrete as waterproofing and corrosion-resistant material; use in road paving and sealing	Process enclosure; local exhaust ventilation; general dilution ventilation; personal protective equipment
Use in manufacture and repair of refractory brick; use in production of foundry cores; use in manufacture of carbon ceramic items	Process enclosure; local exhaust ventilation; general dilution ventilation; personal protective equipment

EMERGENCY FIRST AID PROCEDURES

In the event of an emergency, institute first aid procedures and send for first aid or medical assistance.

- **Eye Exposure**

If condensed coal tar pitch volatiles get into the eyes, wash eyes immediately with large amounts of water, lifting the lower and upper lids occasionally. If irritation is present after washing, get medical attention. Contact lenses should not be worn when working with these chemicals.

- **Skin Exposure**

If condensed coal tar pitch volatiles get on the skin, wash the contaminated skin using soap or mild detergent and water. Be sure to wash the hands before eating or smoking and to wash thoroughly at the close of work.

- **Breathing**

If a person breathes in large amounts of coal tar pitch volatiles, move the exposed person to fresh air at once. If breathing has stopped, perform artificial respiration. Keep the affected person warm and at rest. Get medical attention as soon as possible.

- **Rescue**

Move the affected person from the hazardous exposure. If the exposed person has been overcome, notify someone else and put into effect the established emergency rescue procedures. Do not become a casualty. Understand the facility's emergency rescue procedures and know the locations of rescue equipment before the need arises.

SPILL AND DISPOSAL PROCEDURES

- Persons not wearing protective equipment and clothing should be restricted from areas of releases until cleanup has been completed.
- If coal tar pitch volatiles are released in hazardous concentrations, the following steps should be taken:

1. Ventilate area of spill.

2. Collect released material in the most convenient and safe manner for reclamation or for disposal in sealed containers in a secured sanitary landfill.

• Waste disposal method:

Coal tar pitch volatiles may be disposed of in sealed containers in a secured sanitary landfill.

REFERENCES

• American Conference of Governmental Industrial Hygienists: "Coal Tar Pitch Volatiles," *Documentation of the Threshold Limit Values for Substances in Workroom Air* (3rd ed., 2nd printing), Cincinnati, 1974.

• Bingham, E.: "Environmental Carcinogens," *Archives of Environmental Health*, 19:779-85, DES 1969.

• Bingham, E.: "Thresholds in Cancer Inductions," *Archives of Environmental Health*, 22:692-95, June 1971.

• "Coke Oven Emissions," *Federal Register*, 40:32268-32282, July 31, 1975.

• Committee on Biologic Effects of Atmospheric Pollutants, Division of Medical Sciences, National Research Council: *Particulate Polycyclic Organic Matter*, National Academy of Sciences, Washington, D.C., 1972.

• Fannick, N., et al.: "Exposure to Coal Tar Pitch Volatiles at Coke Ovens," *American Industrial Hygiene Association Journal*, 33:461-468, 1972.

• Grant, W. M.: *Toxicology of the Eye* (2nd ed.), C. C. Thomas, Springfield, Illinois, 1974.

• Hittle, D. C., and Stukel, J. J.: "Particle Size Distribution and Chemical Composition of Coal-Tar Fumes," *American Industrial Hygiene Association Journal*, 37:199-204, 1976.

• *Hygienic Information Guide No. 89 – Coal Tar Pitch Volatiles*, Commonwealth of Pennsylvania, Department of Environmental Resources, Bureau of Occupational Health, 1972.

• International Labour Office: *Encyclopedia of Occupational Health and Safety*, McGraw-Hill, New York, 1971.

• Lloyd, J. W.: "Long-Term Mortality Study of Steelworkers. V. Respiratory Cancer in Coke Plant Workers," *Journal of Occupational Medicine*, 13:53-68, 1971.

• Mazumdar, S., et al.: "An Epidemiological Study of Exposure to Coal Tar Pitch Volatiles among Coke Oven Workers," *Journal of the Air Pollution Control Association*, 25:382-389, 1975.

• National Institute for Occupational Safety and Health, U.S. Department of Health, Education, and Welfare: *Criteria for a Recommended Standard . . . . Occupational Exposure to Coal Tar Products*, HEW Publication No. (NIOSH) 78-107, U.S. Government Printing Office, Washington, D.C., 1977.

• National Institute for Occupational Safety and Health, U.S. Department of Health, Education, and Welfare: *Criteria for a Recommended Standard . . . . Occupational Exposure to Coke Oven Emissions*, HEW Publication No. HSM 73-11016, GPO No. 017-033-00015, U.S. Government Printing Office, Washington, D.C., 1973.

• Redmond, C. K., et al.: "Long-Term Mortality Study of Steelworkers. VI. Mortality from Malignant Neoplasms Among Coke Oven Workers," *Journal of Occupational Medicine*, 14:621-629, 1972.

• Scala, R. A.: "Toxicology of PPOM," *Journal of Occupational Medicine*, 17:784-788, 1975.

• Tye, R., and Stemmer, K. L.: "Experimental Carcinogenesis of the Lung. II. Influence of Phenols in the Production of Carcinoma," *Journal of the National Cancer Institute*, 39:175-179, 1967.

RESPIRATORY PROTECTION FOR COAL TAR PITCH VOLATILES

Condition	Minimum Respiratory Protection* Required Above 0.2 mg/m³
Particulate and Vapor Concentration	
2 mg/m³ or less	A chemical cartridge respirator with an organic vapor cartridge(s) and with a fume or high-efficiency filter. Any supplied-air respirator. Any self-contained breathing apparatus.
10 mg/m³ or less	A chemical cartridge respirator with a full facepiece and an organic vapor cartridge(s) and with a fume or high-efficiency filter. A gas mask with a chin-style or a front- or back-mounted organic vapor canister and with a full facepiece and a fume or high-efficiency filter. Any supplied-air respirator with a full facepiece, helmet, or hood. Any self-contained breathing apparatus with a full facepiece.
200 mg/m³ or less	A Type C supplied-air respirator operated in pressure-demand or other positive pressure or continuous-flow mode. A powered air-purifying respirator with an organic vapor cartridge and a high-efficiency particulate filter.
400 mg/m³ or less	A Type C supplied-air respirator with a full facepiece operated in pressure-demand or other positive pressure mode or with a full facepiece, helmet, or hood operated in continuous-flow mode.
Greater than 400 mg/m³ or entry and escape from unknown concentrations	Self-contained breathing apparatus with a full facepiece operated in pressure-demand or other positive pressure mode. A combination respirator which includes a Type C supplied-air respirator with a full facepiece operated in pressure-demand or other positive pressure or continuous-flow mode and an auxiliary self-contained breathing apparatus operated in pressure-demand or other positive pressure mode.
Fire Fighting	Self-contained breathing apparatus with a full facepiece operated in pressure-demand or other positive pressure mode.
Escape	Any gas mask providing protection against organic vapors and particulates, including pesticide respirators which meet the requirements of this class. Any escape self-contained breathing apparatus.

*Only NIOSH-approved or MSHA-approved equipment should be used.

BOC GASES

MATERIAL SAFETY DATA SHEET

PRODUCT NAME: ISOBUTYLENE

1. Chemical Product and Company Identification

BOC Gases,
Division of
The BOC Group, Inc.
575 Mountain Avenue
Murray Hill, NJ 07974

TELEPHONE NUMBER: (908) 464-8100
24-HOUR EMERGENCY TELEPHONE NUMBER:
CHEMTREC (800) 424-9300

BOC Gases
Division of
BOC Canada Limited
5975 Falbourne Street, Unit 2
Mississauga, Ontario L5R 3W6

TELEPHONE NUMBER: (905) 501-1700
24-HOUR EMERGENCY TELEPHONE NUMBER:
(905) 501-0802
EMERGENCY RESPONSE PLAN NO: 20101

PRODUCT NAME: ISOBUTYLENE
CHEMICAL NAME: Isobutylene
COMMON NAMES/SYNONYMS: 2-Methylpropene, Isobutene
TDG (Canada) CLASSIFICATION: 2.1
WHMIS CLASSIFICATION: A, B1, D2B

PREPARED BY: Loss Control (908)464-8100/(905)501-1700
PREPARATION DATE: 6/1/95
REVIEW DATES: 6/7/96

2. Composition, Information on Ingredients

INGREDIENT	% VOLUME	PEL-OSHA[1]	TLV-ACGIH[2]	LD_{50} or LC_{50} Route/Species
Isobutylene FORMULA: C4H8 CAS: 115-11-7 RTECS #: UD0890000	99.0 to 99.8	Simple Asphyxiant	Simple Asphyxiant	LC_{50} 620 mg/m^3/3H (rat)

[1] As stated in 29 CFR 1910, Subpart Z (revised July 1, 1993)
[2] As stated in the ACGIH 1994-95 Threshold Limit Values for Chemical Substances and Physical Agents

3. Hazards Identification

EMERGENCY OVERVIEW

This product does not contain oxygen and may cause asphyxia if released in a confined area. Simple hydrocarbons can cause irritation and central nervous system depression at high concentrations. flammable.

ROUTE OF ENTRY:

Skin Contact	Skin Absorption	Eye Contact	Inhalation	Ingestion
Yes	No	Yes	Yes	No

HEALTH EFFECTS:

Exposure Limits	Irritant	Sensitization
No	Yes	No
Teratogen	Reproductive Hazard	Mutagen
No	No	No
Synergistic Effects		
None Reported		

Carcinogenicity: -- NTP: No IARC: No OSHA: No

EYE EFFECTS:
Irritation may occur.

SKIN EFFECTS:
None anticipated as product is a gas at room temperature.

INGESTION EFFECTS:
Ingestion is unlikely.

INHALATION EFFECTS:
Product is relatively nontoxic. Simple hydrocarbons can irritate the eyes, mucous membranes and respiratory system at high concentrations.

Inhalation of high concentrations may cause dizziness, disorientation, incoordination, narcosis, nausea or narcotic effects.

This product may displace oxygen if released in a confined space. Maintain oxygen levels above 19.5% at sea level to prevent asphyxiation.

Effects of oxygen deficiency resulting from simple asphyxiants may include: rapid breathing, diminished mental alertness, impaired muscular coordination, faulty judgement, depression of all sensations, emotional instability, and fatigue. As asphyxiation progresses, nausea, vomiting, prostration, and loss of consciousness may result, eventually leading to convulsions, coma, and death.

Oxygen deficiency during pregnancy has produced developmental abnormalities in humans and experimental animals.

NFPA HAZARD CODES	HMIS HAZARD CODES	RATINGS SYSTEM
Health: 1	Health: 1	0 = No Hazard
Flammability: 4	Flammability: 4	1 = Slight Hazard
Reactivity: 0	Reactivity: 0	2 = Moderate Hazard
		3 = Serious Hazard
		4 = Severe Hazard

4. First Aid Measures

EYES:
Never introduce oil or ointment into the eyes without medical advice! If pain is present, refer the victim to an ophthalmologist for further treatment and follow up.

SKIN:

Remove contaminated clothing and wash affected area with soap and water. If irritation persists, seek medical attention.

INGESTION:
Not normally required. Seek immediate medical attention.

INHALATION:
PROMPT MEDICAL ATTENTION IS MANDATORY IN ALL CASES OF OVEREXPOSURE TO PRODUCT. RESCUE PERSONNEL SHOULD BE EQUIPPED WITH SELF-CONTAINED BREATHING APPARATUS. Conscious persons should be assisted to an uncontaminated area and inhale fresh air. Quick removal from the contaminated area is most important. Unconscious persons should be moved to an uncontaminated area, given assisted (artificial) respiration and supplemental oxygen. Further treatment should be symptomatic and supportive.

5. Fire Fighting Measures

Conditions of Flammability: Flammable liquid and vapor		
Flash point: -105 °F (-76 °C)	Method: Closed Cup	Autoignition Temperature: 869 °F (465 °C)
LEL(%): 1.8	UEL(%): 9.6	
Hazardous combustion products: Carbon monoxide, Carbon dioxide		
Sensitivity to mechanical shock: None		
Sensitivity to static discharge: Not Available		

FIRE AND EXPLOSION HAZARDS:
Isobutylene is heavier than air and may travel a considerable distance to an ignition source. Isobutylene is a flammable gas! Keep away from open flame and other sources of ignition. Do not allow smoking in storage areas or when handling.

EXTINGUISHING MEDIA:
Water, carbon dioxide, dry chemical.

FIRE FIGHTING INSTRUCTIONS:
If possible, stop the flow of gas with a remote valve. Use water spray to cool fire exposed containers. If fire is extinguished and flow of gas is continued, increase ventilation to prevent a build up of a flammable/ explosive atmosphere. Extinguish sources of ignition.

Be cautious of a Boiling Liquid Evaporating Vapor Explosion, BLEVE, if flame is impinging on surrounding containers. Direct 500 GPM water stream onto containers above the liquid level with remote monitors. Limit the number of personnel in proximity to the fire. Evacuate surrounding areas to at least 3000 feet in all directions.

6. Accidental Release Measures

Evacuate all personnel from affected area. Use appropriate protective equipment. Increase ventilation to prevent build up of a flammable/explosive atmosphere. Extinguish all sources of ignition! If leak is in user's equipment, be certain to purge piping with inert gas prior to attempting repairs. If leak is in container or container valve, contact the appropriate emergency telephone number listed in Section 1 or call your closest BOC location

7. Handling and Storage

Earth bond and ground all lines and equipment associated with the product system. Electrical equipment should be non-sparking and explosion proof.

Use only in well-ventilated areas. Valve protection caps must remain in place unless container is secured with valve outlet piped to use point. Do not drag, slide or roll cylinders. Use a suitable hand truck for cylinder movement. Use a pressure regulator when connecting cylinder to lower pressure (<250 psig) piping or systems. Do not heat cylinder by any means to increase the discharge rate of product from the cylinder. Use a check valve or trap in the discharge line to prevent hazardous back flow into the cylinder.

Protect cylinders from physical damage. Store in cool, dry, well-ventilated area away from heavily trafficked areas and emergency exits. Do not allow the temperature where cylinders are stored to exceed 130°F (54°C). Cylinders should be stored upright and firmly secured to prevent falling or being knocked over. Full and empty cylinders should be segregated. Use a "first in-first out" inventory system to prevent full cylinders from being stored for excessive periods of time.

Post "No Smoking" signs in storage or use areas.

For additional recommendations consult Compressed Gas Association Pamphlet P-1.

Never carry a compressed gas cylinder or a container of a gas in cryogenic liquid form in an enclosed space such as a car trunk, van or station wagon. A leak can result in a fire, explosion, asphyxiation or a toxic exposure.

8. Exposure Controls, Personal Protection

EXPOSURE LIMITS[1]:

INGREDIENT	% VOLUME	PEL-OSHA[2]	TLV-ACGIH[3]	LD_{50} or LC_{50} Route/Species
Isobuylene FORMULA: C4H8 CAS: 115-11-7 RTECS #: UD0890000	99.0 to 99.8	Simple Asphyxiant	Simple Asphyxiant	LC_{50} 620 mg/m^3/3H (rat)

[1] Refer to individual state of provincial regulations, as applicable, for limits which may be more stringent than those listed here.
[2] As stated in 29 CFR 1910, Subpart Z (revised July 1, 1993)
[3] As stated in the ACGIH 1994-1995 Threshold Limit Values for Chemical Substances and Physical Agents.

ENGINEERING CONTROLS:
Use local exhaust to prevent accumulation. Use general ventilation to prevent build up of flammable concentrations. May use hood with forced ventilation when handling small quantities. If product is handled routinely where the potential for leaks exists, all electrical equipment must be rated for use in potentially flammable atmospheres. Consult the National Electrical Code for details.

EYE/FACE PROTECTION:
Safety goggles or glasses.

SKIN PROTECTION:
Protective gloves made of plastic or rubber.

RESPIRATORY PROTECTION:
Positive pressure air line with full-face mask and escape bottle or self-contained breathing apparatus should be available for emergency use.

OTHER/GENERAL PROTECTION:
Safety shoes, safety shower, eyewash.

9. Physical and Chemical Properties

PARAMETER	VALUE	UNITS
Physical state (gas, liquid, solid)	: Gas	
Vapor pressure at 70°F	: 39	psia
Vapor density at STP (Air = 1)	: 1.98	
Evaporation point	: Not Available	
Boiling point	: 19.5	°F
	: -6.9	°C
Freezing point	: -220.6	°F
	: -140.3	°C
pH	: Not Available	
Specific gravity	: Not Available	
Oil/water partition coefficient	: Not Available	
Solubility (H20)	: Insoluble	
Odor threshold	: Not Available	
Odor and appearance	: A colorless gas with an unpleasant odor similar to that of burning coal.	

10. Stability and Reactivity

STABILITY:
Stable

CONDITIONS TO AVOID (STABILITY):
None

INCOMPATIBLE MATERIALS:
Oxidizers

HAZARDOUS DECOMPOSITION PRODUCTS:
Carbon monoxide

11. Toxicological Information

Oxygen deficiency during pregnancy has produced developmental abnormalities in humans and experimental animals.

No chronic effects data given in the Registry of Toxic Effects of Chemical Substances (RTECS) or Sax, Dangerous Properties of Industrial Materials, 7th ed.

12. Ecological Information

No data given.

13. Disposal Considerations

Do not attempt to dispose of residual waste or unused quantities. Return in the shipping container PROPERLY LABELED, WITH ANY VALVE OUTLET PLUGS OR CAPS SECURED AND VALVE PROTECTION CAP IN PLACE to BOC Gases or authorized distributor for proper disposal.

14. Transport Information

PARAMETER	United States DOT	Canada TDG
PROPER SHIPPING NAME:	Isobutylene	Isobutylene
HAZARD CLASS:	2.1	2.1
IDENTIFICATION NUMBER:	UN 1055	UN 1055
SHIPPING LABEL:	FLAMMABLE GAS	FLAMMABLE GAS

15. Regulatory Information

Isoutylene is listed under the accident prevention provisions of section 112(r) of the Clean Air Act (CAA) with a threshold quantity (TQ) of 10,000 pounds.

SARA TITLE III NOTIFICATIONS AND INFORMATION

SARA TITLE III - HAZARD CLASSES:
Acute Health Hazard
Fire Hazard
Sudden Release of Pressure Hazard

16. Other Information

Compressed gas cylinders shall not be refilled without the express written permission of the owner. Shipment of a compressed gas cylinder which has not been filled by the owner or with his/her (written) consent is a violation of transportation regulations.

DISCLAIMER OF EXPRESSED AND IMPLIED WARRANTIES:
Although reasonable care has been taken in the preparation of this document, we extend no warranties and make no representations as to the accuracy or completeness of the information contained herein, and assume no responsibility regarding the suitability of this information for the user's intended purposes or for the consequences of its use. Each individual should make a determination as to the suitability of the information for their particular purpose(s).

Material Safety Data Sheet: **Simple Green® All-Purpose Cleaner**
Simple Green® Scrubbing Pad
Version No. 1300509A Date of Issue: January 2009 *ANSI-Z400.1-2003 Format*

Section 1: PRODUCT & COMPANY IDENTIFICATION

Product Name: Simple Green® All-Purpose Cleaner
Simple Green® Scrubbing Pad
Additional Name: Simple Green® Concentrated Cleaner/Degreaser/Deodorizer

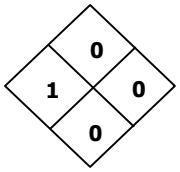
Manufacturer's Product Code Numbers: **Please refer to page 4*

Company: Sunshine Makers, Inc.
15922 Pacific Coast Highway
Huntington Harbour, CA 92649 USA
Telephone: 800-228-0709 ● 562-795-6000 Fax: 562-592-3830
Emergency Phone: Chem-Tel 24-Hour Emergency Service: 800-255-3924

Use of Product: An all purpose cleaner and degreaser used diluted in water for direct, spray and dip tank procedures. Scrubbing pad is used with water for manual scrubbing applications. Both are for cleaning water-safe surfaces.

Section 2: HAZARDS IDENTIFICATION

Emergency Overview: CAUTION. Mild eye irritant.
Simple Green@ is a dark green liquid with a sassafras odor. Scrubbing Pad is a green fibrous rectangle.



HMIS Rating:
Health = 1 = slight
Fire = 0
Reactivity, and Special = 0 = minimal

Eye Contact: Mild Eye Irritant.

Skin Contact: No adverse effects expected under typical use conditions. Prolonged exposure may cause dryness. Under this condition, use of gloves or skin moisturizer after washing may be indicated.

Ingestion: May cause stomach or intestinal upset if swallowed (due to detersive properties.)

Inhalation: No adverse effects expected under typical use conditions. Adequate ventilation should be present when using Simple Green® over a prolonged period of time. Open windows or ventilate via fan or other air-moving equipment if necessary.

Carcinogens: No ingredients are listed by OSHA, IARC, or NTP as known or suspected carcinogens.

Medical Conditions: No medical conditions are known to be aggravated by exposure to Simple Green®. Dermal-sensitive users may experience mild but reversible reactions.

UN Number: Not Required **Dangerous Goods Class:** Non-hazardous

Section 3: COMPOSITION/INFORMATION ON INGREDIENTS

The only ingredient of Simple Green® with established exposure limits is undiluted 2-butoxyethanol (<4%) (Butyl Cellosolve; CAS No. 111-76-2) [*1% for Scrubbing Pad*]: the ACGIH TLV-TWA is 20 ppm (97 mg/m^3). Based upon chemical analysis, Simple Green® contains no known EPA priority pollutants, heavy metals or chemicals listed under RCRA, CERCLA, or CWA. Analysis by TCLP (Toxicity Characteristic Leaching Procedure) according to RCRA revealed no toxic organic or inorganic constituents.

All components of Simple Green® are listed on the TSCA Chemical Substance Inventory.
This product does not contain any ingredients covered by the provisions of 29 CFR 1910.1200.

Section 4: FIRST AID MEASURES

Eye Contact: Reddening may develop. Immediately rinse the eye with large quantities of cool water; continue 10-15 minutes or until the material has been removed; be sure to remove contact lenses, if present, and to lift upper and lower lids during rinsing. Get medical attention if irritation persists.

Skin Contact: Minimal effects, if any; rinse skin with water, rinse shoes and launder clothing before reuse. Reversible reddening may occur in some dermal-sensitive users; thoroughly rinse area and get medical attention if reaction persists.

Swallowing; Essentially non-toxic. Give several glasses of water to dilute; do not induce vomiting. If stomach upset occurs, consult physician.

Inhalation: Non-toxic. Exposure to concentrate may cause mild irritation of nasal passages or throat; remove to fresh air. Get medical attention if irritation persists.

Section 5: FIRE FIGHTING MEASURES

Simple Green® is stable, not flammable, and will not burn. No special procedures required.

Flash Point/Auto-Ignition: Not flammable. Extinguishing Media: Not flammable/non-explosive.
Flammability Limits: Not flammable. Special Fire Fighting Procedures: None required.

Section 6: ACCIDENTAL RELEASE MEASURES

Personal Precautions: Avoid contact with eyes. Do not rub eyes with hands during cleanup. No special precautions for dermal contact are needed. Wash hands thoroughly after cleaning up spill or leak.

Method for cleaning up: Recover usable material by convenient method, residual may be removed by wipe or wet mop. If necessary, unrecoverable material may be washed to drain with large quantities of water.

Section 7: HANDLING AND STORAGE

No Special precautions are required. **This product is non-hazardous for storage and transport according to the U.S. Department of Transportation Regulations.** Simple Green® requires no special labeling or placarding to meet U.S. Department of Transportation requirements.

UN Number: Not Required **Dangerous Goods Class:** Non-hazardous

Section 8: EXPOSURE CONTROLS/PERSONAL PROTECTION

Exposure Limits: The Simple Green® formulation presents no health hazards to the user when used according to label directions for its intended purposes. Mild skin and eye irritation is possible (please see Eye contact and Skin contact in section IV.) No special precautionary measures required under normal use conditions.

Ventilation: No special ventilation, precautions or respiratory protection is required during normal use. Large-scale use indoors should provide an increased rate of air exchange.

Human Health Effects or Risks From Exposure: Adverse effects on human health are not expected from Simple Green®, based on 20 years of use of Simple Green® without reported adverse health incidence in diverse population groups, including extensive use by inmates of U.S. Federal prisons in cleaning operations.

Eye protection: Simple Green® is a mild eye irritant; mucous membranes may become irritated by concentrate. Eye protection not generally required. Wash hands after using wipes.

Skin protection: Simple Green® is not likely to irritate the skin in the majority of users. Repeated daily application to the skin without rinsing, or continuous contact on the skin may lead to temporary, but reversible, irritation. Rinse completely from skin after contact.

Section 8: EXPOSURE CONTROLS/PERSONAL PROTECTION – continued –

General hygiene conditions: There are no known hazards associated with this material when used as recommended. The following general hygiene considerations are recognized as common good industrial hygiene practices:

- Avoid breathing vapor or mist.
- Avoid contact with eyes.
- Wash thoroughly after handling and before eating, drinking, or smoking.

Section 9: PHYSICAL AND CHEMICAL PROPERTIES

Appearance & Odor: Cleaner is a dark green liquid, pad is a fibrous green matrix; both exhibit a sassafras odor.			
Specific Gravity:	1.010 ± 0.010	**Vapor Pressure:**	18 mm Hg @ 20°C; 23.5 mmHg @ 26°C
Evaporation:	>1 (butyl acetate = 1)	**Vapor Density:**	1.3 (air = 1)
Water Solubility:	100%	**Density:**	8.5 lbs/gallon
Boiling Point:	100.6°C (212°F)	**pH:**	9.5 ± 0.3
Ash Content:	At 600°F: 1.86% by weight	**Nutrient Content:** Phosphorus: 0.3% by formula Nitrogen <1.0% by weight (fusion and qualitative test for ammonia) Sulfur: 0.6% by weight (barium chloride precipitation method)	
Freezing Point: Approx -9 °C (16 °F) *If product freezes, it will reconstitute without loss of efficacy when brought back to room temperature and agitated.*			
VOC Composite Partial Pressure: 0.006 mmHg @ 20°C			
Volatile Organic Compounds (VOCs): CARB Method 310 3.8% SCAQMD Method 313 2.8%	Cleaner meets CARB & BAAQMD regulations. Cleaner must be diluted 1:1 with water to Meet SCAQMD Rule 1171 & Rule 1122 VOC requirements for solvent cleaning operations. [*Scrubber VOCs = 3.3% prior to dilution w/water*]		

Section 10: STABILITY AND REACTIVITY

Stability: Stable
Materials to Avoid: None known
Hazardous Decomposition Products: None expected

Section 11: TOXICOLOGICAL INFORMATION

Toxicology information is based on chemical profile of ingredients and extrapolation of data from similar formulas.

Acute Toxicity: Oral LD_{50} (rat) >5 g/kg body weight*
Dermal LD_{50} (rabbit) >2 g/kg body weight

**Calculation from OECD series on testing and assessment number 33, Chapter 3.2*

Eye Irritation: Moderate/Mild reversible eye irritation may occur based on relevant laboratory studies. This potential is reduced by immediate rinsing of eyes in case of eye contact.

Dermal Irritation: Mild, reversible skin irritation may occur based on relevant laboratory studies. A 6-hour exposure to human skin under a patch did not produce irritation

Repeat Exposure Via Skin Contact: Based on relevant laboratory studies, no toxic effects are expected to be associated with daily skin exposures (with up to 2 g/kg/day tested for 13 weeks on rabbits). Skin irritation may, however, occur with repeated or prolonged exposures.

Reproductive Effects Assessment: Based on relevant laboratory studies (CD-1 mouse 18-week fertility assessment continuous breeding), no adverse effects on reproduction, fertility, or health of offspring are expected.

Section 12: ECOLOGICAL INFORMATION

Hazard to wild animals & aquatic organisms: Low, based on toxicological profile.

Biodegradability: Readily biodegradable based on biodegradation profile,
PRO/FT CBT-AC 014-7 "Ready Biodegradability: Closed Bottle Test" OECD, and OECD 302B laboratory tests

Environmental Toxicity Information: It is important not to allow the runoff from cleaning into closed systems such as decorative ponds. Always protect closed systems with tarps or dikes if necessary.

Section 13: DISPOSAL CONSIDERATIONS

Dispose of in accordance with all applicable local, state and federal laws. Dispose of used or unused product, and empty containers in accordance with the local, State, Provincial, and Federal regulations for your location. Never dispose of used degreasing rinsates into lakes, streams, and open bodies of water or storm drains.

Section 14: TRANSPORT INFORMATION

This product is non-hazardous for transport according to the U.S. Department of Transportation Services
UN Number: Not required Dangerous Goods Class: Non-hazardous

Section 15: REGULATORY INFORMATION

*Reportable components:

All components are listed on:	EINECS and TSCA Inventory
No components listed under:	Clean Air Act Section 112
SARA:	This material contains 2-Butoxyethanol, < 4%, (CAS# 111-76-2) which is subject to the reporting requirements of Section 313 of SARA Title III and 49 CFR Part 373.

RCRA Status:	Not a hazardous waste.	CERCLA Status:	No components listed
TSCA TRI Reporting:	Not required / Not listed	CA PROP. 65 Status:	No components listed

Section 16: OTHER INFORMATION

Questions about the information found on this MSDS should be directed to:
SUNSHINE MAKERS, INC. – TECHNICAL DEPARTMENT
15922 Pacific Coast Hwy. Huntington Harbour, CA 92649

Phone: 800/228-0709 [8am-5pm Pacific time, Mon-Fri] *Fax:* 562/592-3830 *Email:* infoweb@simplegreen.com

CAGE CODE 1Z575
GSA/FSS - CONTRACT NO. GS-07F-0065J
National Stock Numbers & Industrial Part Numbers:

	Part Number	NSN	Size
Simple Green	13012	7930-01-342-5315	24 oz spray (12/case)
	13005	7930-01-306-8369	1 Gallon (6/case)
	13006	7930-01-342-5316	5 Gallon
	13016	7930-01-342-5317	15 Gallon
	13008	7930-01-342-4145	55 Gallon
Scrubbing Pad	Part Number	NSN	Size
	10224	7930-01-346-9148	Each (24/case)

Retail Numbers:

Part Number	Size
13002	16 oz Trigger (12/case)
13005	1 Gallon (6/case)
13013	24 oz Trigger (12/case)
13014	67 oz / 2 L (6/case)
13033	32 oz Trigger (12/case)

** part number is for both industrial and retail*

****International Part Numbers May Differ.**

DISCLAIMER: The information provided with this MSDS is furnished in good faith and without warranty of any kind. Personnel handling this material must make independent determinations of the suitability and completeness of information from all sources to assure proper use and disposal of this material and the safety and health of employees and customers. Sunshine Makers, Inc. assumes no additional liability or responsibility resulting from the use of, or reliance on this information.

Appendix E

Forms

Appendix F

GEI's Health and Safety SOPs and Programs

Applicable GEI H&S SOPs *(check all that apply)*		
☒ Biological Hazards – 001	☒ Inclement Weather – 010	☐ Aerial Lift – 020
✔ Bloodborne Pathogens – 002	☐ Ladders -011	☐ Mobile Equipment – 021
☐ Container Management – 003	☒ Noise Exposure -012	☐ Aquatic Ecological Survey & Electrofishing -022
☒ Driver Safety - 004	☐ Nuclear Density Gauge Operation -013	☐ Scaffolding - 023
☒ Electrical Safety - 005a	☒ Utility Markout-014	☐ Wilderness Safety - 024
☒ Lockout Tagout - 005b	☐ Respirator Fit Test Procedure-015	☐ Manual Lifting – 025
☐ Excavation Trenching - 006	☐ Traffic Hazards -016	✔ Hazard Identification - 026
☒ Non-Powered Hand Tools - 008a	☒ Water Safety – 017	☐ Confined Space Entry for Sanitary Sewers – 027
☒ Powered Hand Tools – 008b	☒ Working Around Heavy Equipment – 018	☐ Safe Trailer Use – 028
☒ Hazardous Substances Management -009	☐ Rail Safety -019	

Scan this QR code with your smartphone to access all GEI H&S SOPs

Applicable GEI H&S Programs *(check all that apply)*		
☐ Asbestos Program	☐ DOT Driver Safety	☒ Hydrogen Sulfide
☒ Arsenic Safety	✔ Ergonomic	☐ Injury and Illness Prevention (California Only)
☒ Benzene Awareness	☐ Fall Protection	☐ Respiratory Protection Program
☐ Cadmium Safety	✔ Hazard Communication	☐ Lead Awareness
☐ Cold Stress	☐ Hearing Conservation	✔ Fire Prevention
☐ Confined Space Entry	☐ Heat Illness Prevention	
☐ Crystalline Silica	☐ Hexavalent Chromium	

Scan this QR code with your smartphone to access all GEI Programs

STANDARD OPERATING PROCEDURES

SOP No. HS-001 Biological Hazards

1.1 Objective

The objective of this Standard Operating Procedure (SOP) is to prevent or limit the potential for GEI personnel to encounter biological hazards during field activities.

1.2 General

This SOP is intended for use by employees engaged in work with the potential for contact with biological hazards such as animals, insects, plants, and sewage. The site-specific health and safety plan (HASP) should include a hazard assessment for the project that identifies the potential for encounters with biological hazards and the control methods to be implemented by GEI employees. These hazards must be reviewed in the project safety briefing and documented on the Project Safety Briefing form, found on the Safety page of the GEI intranet.

1.3 Mammals

During some site operations, animals such as stray or domesticated dogs or cats, raccoons, snakes, bears, rats, bats, etc. may be encountered. Employees should use discretion and attempt to avoid contact with animals. If these animals present a problem, efforts will be made to remove these animals from the site by contacting a licensed animal control technician.

1.3.1 Rabies

The rabies virus is transmitted through the bite of an infected animal or contact with saliva or brain/nervous system tissue of an infected animal. The rabies virus infects the central nervous system, causing disease in the brain. The early symptoms of rabies in people are fever, headache, and general weakness or discomfort. As the disease progresses, more specific symptoms appear and may include insomnia, anxiety, confusion, slight or partial paralysis, excitation, hallucinations, agitation, hypersalivation (increase in saliva), difficulty swallowing, and hydrophobia (fear of water). Death usually occurs within days of the onset of these symptoms.

If you are bitten or think you may be exposed, wash any wounds immediately and thoroughly with soap and water. Then go to the hospital emergency room and notify the Project Manager and the People Safety Team. The doctor, possibly in consultation with the state or local health department, will decide if you need a rabies vaccination. Decisions to start series of vaccinations will be based on your type of exposure and the animal you were exposed to, as well as laboratory and surveillance information for the geographic area where the exposure occurred. If possible have someone document what type of animal it was, how it was behaving prior to the bite, what caused it to bite the

employee, and if it's not a domestic animal that would be easy to find again in the future, try to get animal control on site to capture it. An Incident Report Form must be completed and submitted, per GEI's Incident reporting procedures. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

1.4 Insects and Arachnids

Insects, including bees, wasps, hornets, mosquitoes, ticks, spiders, etc., may be present at a job site making the chance of a bite/sting possible. Some individuals may have a severe allergic reaction to an insect bite or sting that can result in a life-threatening condition. Some insect bites can transmit diseases such as Lyme disease or a virus such as West Nile. The following is a list of preventive measures:

- Apply insect repellent prior to performing field work and as often as needed throughout the work shift.
- Wear proper personal protective equipment (PPE), including protective clothing (work boots, socks, and light colored clothing).
- Wear shoes, long pants with bottoms tucked into boots or socks, and a long-sleeved shirt when outdoors for long periods of time, or when many insects are most active (between dawn and dusk).
- When walking in wooded areas, avoid contact with bushes, tall grass, or brush as much as possible.
- Field personnel who have or may have insect allergies must have insect allergy medication onsite and must inform the Site Safety Officer (SSO) and the People and Safety Team of their particular allergy prior to commencing work.
- Field personnel should perform a self-check at the end of the day for ticks.

1.4.1 Tick-borne Diseases

Lyme Disease

Lyme disease is caused by infection from a deer tick that carries a spirochete (a bacterium). During the painless tick bite, the spirochete may be transmitted into the bloodstream, often after feeding on the host for 12 to 24 hours. The ticks that cause the disease are often no bigger than a poppy seed or a comma in newsprint. The peak months for human infection are from May to September.

Symptoms appear in three stages. First symptoms usually appear from 2 days to a few weeks after a person is bitten by an infected tick. Symptoms usually consist of a ring-like red rash on the skin where the tick was attached. The rash is often bulls-eye like with red around the edges and clear in the center. The rash may be warm, itchy, tender, and/or "doughy." This rash appears in only 60 to 80 percent of infected persons. An infected

person also has flu-like symptoms of a stiff neck, chills, fever, sore throat, headache, fatigue, and joint pain. These symptoms often disappear after a few weeks.

The second stage symptoms, which occur weeks to months later include meningitis, severe headache, drooping of the muscles on the face, called Bell's Palsy, encephalitis, numbness, withdrawal, and lethargy. These symptoms may last for several weeks to several months. Third stage symptoms, which occur months or years later include arthritis, heart problems, and loss of memory. The third stage symptoms may mimic multiple sclerosis and Alzheimer's disease.

When in areas that could harbor deer ticks, employees should wear light color clothing, and visually check themselves and check and be checked by another employee when coming from wooded or vegetated areas. If a GEI employee has a tick bite, the People and Safety Team and Project Manager must be contacted immediately. The employee will be offered the option for medical treatment by a physician, which typically involves antibiotics. An Incident Report form must be completed in compliance with the Incident Reporting procedures. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

If personnel feel sick or have signs similar to those mentioned above, the SSO and the People and Safety Team must be notified immediately.

Figure 1: From left to right, the deer tick adult female, adult male, nymph, and larva on a centimeter scale.

How to Remove a Tick

A tick can be removed from the skin by pulling gently at the head with tweezers. If tweezers are not available, use tissue paper or cloth to grasp the tick. It is important to grasp the tick as close to the site of attachment and use a firm steady pull to remove it. Wash hands immediately after with soap and water. The affected area should also be washed with soap and water, then disinfected with an antiseptic wipe, if available. All mouth parts must be removed from the skin. If the tick was removed by breaking off the

mouth parts, an irritation or infection may occur because the organism that is causing the disease can still enter the body through the skin.

Treatment for Lyme Disease

Treatment with antibiotics is effective and recovery is usually complete. For first stage symptoms, antibiotics are usually given orally. However, treatment for second and third stage symptoms is prolonged and recovery may take longer. Antibiotic treatment is usually provided intravenously for second and third stage Lyme disease.

Babesiosis

The deer tick can also cause Babesiosis, an infection of the parasite Babesia Microti. Symptoms of Baesiosis may not be evident, but may also include fever, fatigue and hemolytic anemia lasting from several days to several months. Babesiosis is most commonly diagnosed in the elderly or in individuals whose immune systems are compromised. If there are no signs or symptoms of Babesiosis, usually no treatment it needed. If an employee believes they might have Babesiosis they'll see a physician to be tested. Treatment usually consists of taking prescription medications for 7 to 10 days.

Ehrlichiosis

Ehrlichiosis is a tick-borne disease which can be caused by either of two different organisms. Human monocytic ehrlichiosis (HME) is caused by *Ehrlichia chaffeensis*, which is transmitted by the lone star tick (*Amblyomma americanum*). Human granulocytic anaplasmosis (HGA), previously known as human granulocytic ehrlichiosis (HGE), is caused by *Anaplasma phagocytophilia*, which is transmitted by the deer tick (*Ixodes scapularis*).

Ehrlichiosis is transmitted by the bite of infected ticks, including the deer tick and the lone star tick. The symptoms of HME and HGE are the same and usually include fever, muscle aches, weakness and headache. Patients may also experience confusion, nausea, vomiting and joint pain. Unlike Lyme disease or Rocky Mountain spotted fever, a rash is not common. Infection usually produces mild to moderately severe illness, with high fever and headache, but may occasionally be life-threatening or even fatal. Symptoms appear 1 to 3 weeks after the bite of an infected tick. However, not every exposure results in infection. For those that become infected a drug called Doxcycline will be prescribed.

Rocky Mountain Spotted Fever

Rocky Mountain spotted fever is a tick-borne disease caused by a rickettsia (a microbe that differs somewhat from bacteria and virus). In the eastern United States, children are infected most frequently, while in the western United States, disease incidence is highest among adult males. Disease incidence is directly related to exposure to tick-infested habitats or to infested pets. Rocky Mountain spotted fever is characterized by a sudden onset of moderate to high fever (which can last for 2-3 weeks), severe headache, fatigue, deep muscle pain, chills and rash. The rash begins on the legs or arms, may include the

soles of the feet or palms of the hands and may spread rapidly to the trunk or rest of the body. Symptoms usually appear within 2 weeks of the bite of an infected tick. Like Ehrlichiosis the prescription drug Doxcycline is the first line treatment option.

1.4.2 Mosquito-Borne Disease

West Nile Virus
West Nile Virus is a mosquito-borne infection transmitted through the bite of an infected mosquito. The symptoms of West Nile Virus can be asymptomatic (no symptoms) or in more serious cases can lead to West Nile Fever. West Nile Fever can include fever, headache, tiredness, body ache, an occasional rash on the trunk of the body, and swollen lymph glands, In severe cases, people have developed West Nile Encephalitis or Meningitis which symptoms include fever, headache, neck stiffness, tremors, coma, and in some cases death. The incubation period for the disease is usually 2 to 15 days. The symptoms can range from a few days to several weeks. Most mosquitoes are not infected and the chance of infection from a mosquito bite of an on-site employee is very small.

1.5 Repellants

The following precautions will be used to help reduce the risk of mosquito bites:

Reduce mosquito-breeding areas by making sure wheelbarrows, buckets, and other containers are turned upside down when not used so that they do not collect standing water. According to the Environmental Protection Agency (EPA), many mosquitoes can breed in pooled water that's minimal enough to fill a bottle cap.

Wear shoes, long pants with bottoms tucked into boots or socks, and a long-sleeved shirt when outdoors for long periods of time, or when many mosquitoes are most active (between dawn and dusk).

Use mosquito repellant according to the manufacturer's directions when outdoors for long periods of time and when mosquitoes are most active.

Centers for Disease Control and Prevention (CDC) evaluation of information contained in peer-reviewed scientific literature and data available from the EPA has identified several EPA-registered products that provide repellent activity sufficient to help people avoid the bites of disease carrying mosquitoes. Products containing these active ingredients typically provide reasonably long-lasting protection:

- **DEET** (Chemical Name: N,N-diethyl-m-toluamide or N,N-diethly-3-methyl-benzamide)
- **Picaridin** (KBR 3023, Chemical Name: 2-(2-hydroxyethyl)-1-piperidinecarboxylic acid 1-methylpropyl ester)

- **Oil of Lemon Eucalyptus** or **PMD** (Chemical Name: para-Menthane-3,8-diol) the synthesized version of oil of lemon eucalyptus
- **IR3535** (Chemical Name: 3-[N-Butyl-N-acetyl]-aminopropionic acid, ethyl ester)
- **Permethrin** (3-Phenoxybenzyl (1RS)-cis,trans-3-(2,2-dichlorovinyl) -2,2-dimethylcyclopropanecarboxylate) **–** Permethrin kills ticks and can be used on clothing (but not skin)

The EPA characterizes the active ingredients DEET and Picaridin as "conventional repellents" and Oil of Lemon Eucalyptus, PMD, and IR3535 as "biopesticide repellents", which are derived from natural materials.

In general, higher concentrations of active ingredient provide longer duration of protection, regardless of the active ingredient, although concentrations above approximately 50 percent do not offer a marked increase in protection time. Products with less than 10 percent active ingredient may offer only limited protection, often from 1 to 2 hours. Products that offer sustained release or controlled release (micro-encapsulated) formulations, even with lower active ingredient concentrations, may provide longer protection times. Regardless of what product you use, if you start to get mosquito bites reapply the repellent according to the label instructions or remove yourself from the area with biting insects if possible.

Clothing and other products can be purchased pre-treated, or products can be treated using EPA-registered products. Permethrin is the only pesticide approved by the EPA for these uses. Permethrin binds tightly to the fabrics, resulting in little loss during washing and minimal transfer to the skin. Permethrin is poorly absorbed through the skin, although sunscreens and other products may increase the rate of skin absorption.

If you decide to use permethrin-treated clothing, consider these tips:

- Read the application instructions carefully and apply the product according to the label directions. Do not over-treat products.
- Permethrin treatments are only intended for use on fabrics; do not apply them directly to the skin or other items.
- Do not apply permethrin to clothing while it is being worn.
- Apply the product to clothing outdoors in well ventilated areas that are protected from wind.
- Hang treated fabrics outdoors and allow them to dry completely before wearing them.
- Wash permethrin treated clothing separately from other clothing items.

1.6 Poisonous Plants

The potential for contact with poisonous plants, such as poison ivy, oak, and sumac exists when performing fieldwork in wooded or boggy areas. Urushiol, an oily organic allergen found in plants, can cause an allergic reaction when in contact with the leaves or vines.

Poison ivy can be found as vines on tree trunks or as upright bushes. Poison ivy consists of three leaflets with notched edges. Two leaflets form a pair on opposite sides of the stalk, and the third leaflet stands by itself at the tip. Poison ivy is red in the early spring and turns shiny green later in the spring. Poison ivy grows throughout much of North America, including all states east of the Rocky Mountains. It is normally found in wooded areas, especially along edge areas where the tree line breaks and allows sunshine to filter through. It also grows in exposed rocky areas, open fields, and disturbed areas.

Poison oak can be present as a sparsely-branched shrub. Poison oak can grow anywhere in the United States with the exception of Hawaii, Alaska, and some southwest areas that have desert climates. Poison oak is similar to poison ivy in that it has the same leaflet configuration; however, the leaves have slightly deeper notches.

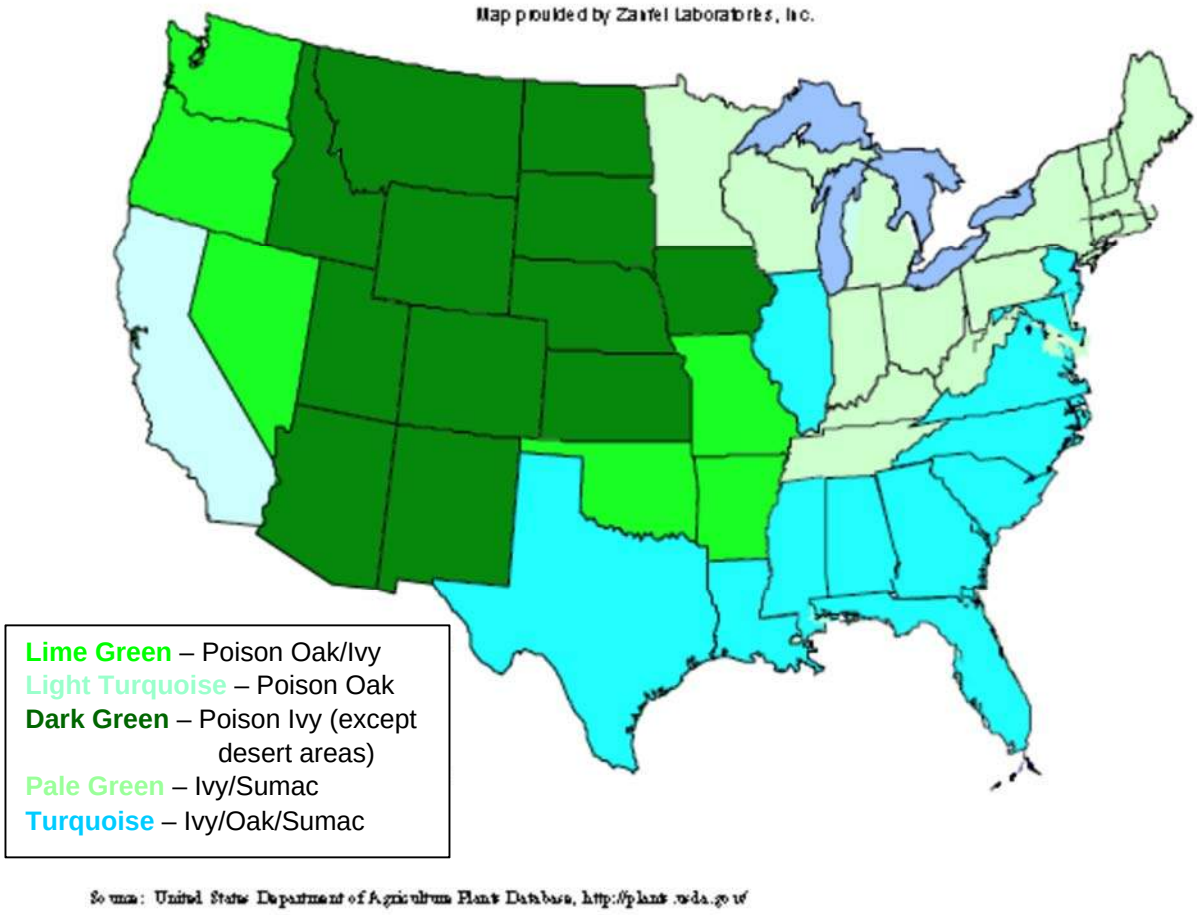
Poison sumac can be present in the form of a flat-topped shrub or tree. It has fern-like leaves, which are velvety dark green on top and pale underneath. The branches of immature trees have a velvety “down.” Poison sumac has white, “hairy” berry clusters. Poison sumac grows exclusively in very wet or flooded soils, usually in swamps and peat bogs, in the eastern United States.

Poison Ivy

Poison Oak

Poison Sumac

U.S. Prevalence of Poison Ivy, Oak & Sumac



Source: United State Department of Agriculture Plant Database, http://plants.usda.gov/

To prevent exposure to these poisonous plants:

- Wear proper PPE, including long sleeves, long pants, boots, and gloves.
- Barrier skin creams, such as lotion containing bentoquatum (Tecnu®), may offer some protection prevent the occurrence of exposure symptoms.
- Contact with poison ivy, sumac, or oak may lead to a skin rash, characterized by reddened, itchy, blistering skin which needs first aid treatment. Employees with known allergies should identify themselves to the SSO or Project Manager prior to starting field work as a precautionary measure. If you believe you have contacted one of these plants:
    - Immediately wash skin thoroughly with soap and water, taking care not to touch your face or other body parts.
    - Contact the People and Safety Team and Project Manager immediately after caring for affected skin.

- Wash exposed clothing separately in hot water with detergent.

- After use, clean tools, and soles of boots with rubbing alcohol or soap and lots of water. Urushiol can remain active on the surface of objects for up to 5 years.
- If a rash occurs, contact the People and Safety Team and complete and submit an Incident Report Form. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

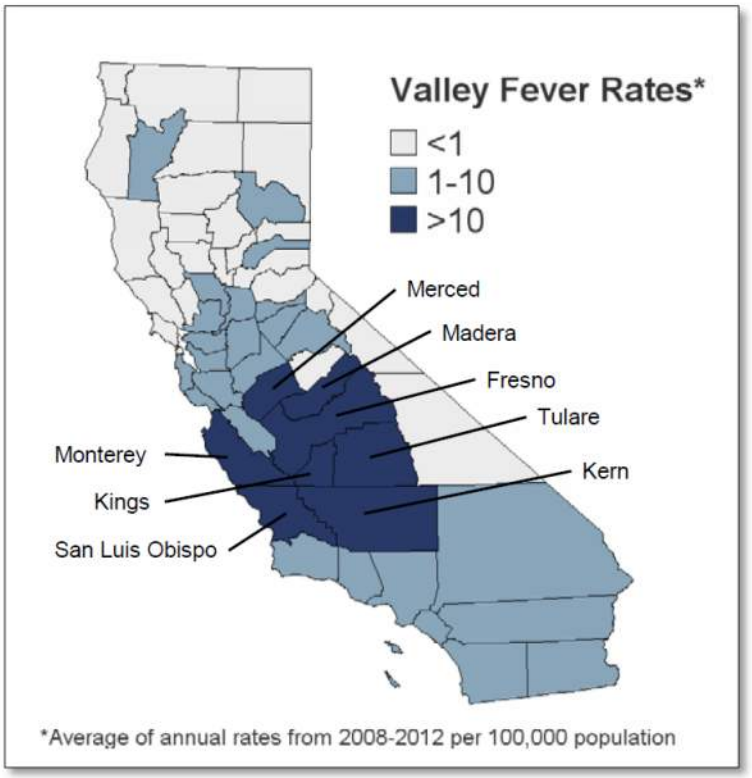
1.7 Sewage and Bacterial Impacted Sediments

Some project work may be conducted at sites that serve or have served as a combined sewer overflow and consequently may have received untreated sanitary sewage from numerous sources. Decomposed sewage can potentially be encountered within sites and their sediments. Sediments could contain soil and marine microorganisms, and bacterium associated with sewage. Many of these bacterium can cause illness through ingestion, direct contact, or the inhalation of a bio-aerosol possibly in the form of dust. Potential respiratory exposure to biological agents can also occur through the inhalation of aerosols produced during sediment handling activities. PPE as identified in the site-specific HASP will be worn to minimize potential exposures. Employees will follow the decontamination or disposal procedures identified in the HASP.

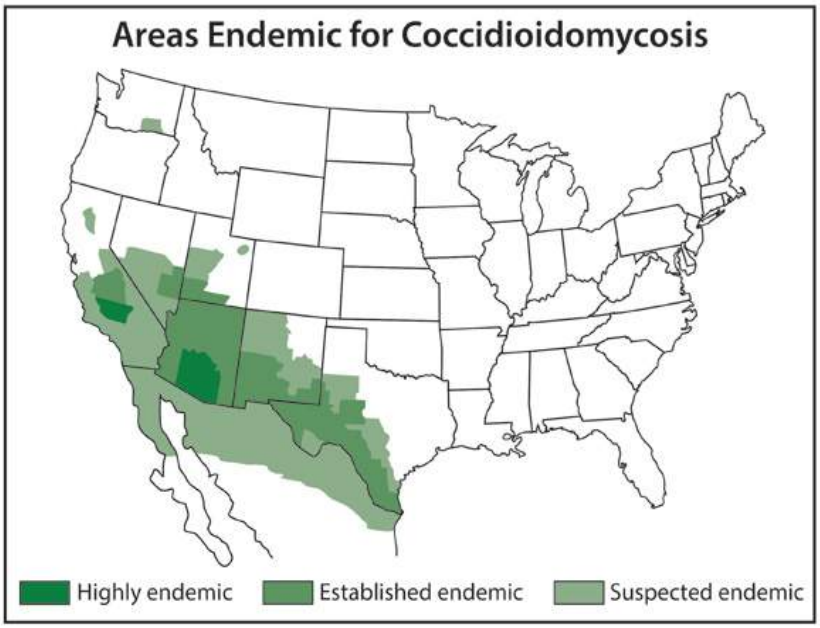
1.7.1 Fungal Spores in Soil – Valley Fever

Valley Fever is an illness that usually affects the lungs. It is caused by the fungus *Coccidioides immitis* that lives in the top 2 to 12 inches of soil in many parts of California. When fungal spores are present, any work activity that disturbs the soil, such as digging, grading, or other earth moving operations, or vehicle operation on dirt roads, can cause the spores to become airborne, therefore increasing the risk of Valley Fever. All employees on sites where the fungus is present, and who are exposed to dusty conditions and wind-blown dusts are at increased risk of becoming infected.

Valley Fever fungal spores are too small to be seen, and there is no reliable way to test the soil for spores before working in a particular place. Valley Fever can be found throughout the southwestern United States, parts of Mexico, and South America. Some California counties consistently have Valley Fever fungus present in the soil. In these regions Valley Fever is considered endemic. Health departments track the number of cases of Valley Fever illness that occur. This information is used to map illness rates as seen on the figures below from the Center of Disease Control Valley Fever Awareness website.



Rates of reported Valley Fever cases in California counties from 2008–2012. Darkest colored counties had the highest rates of Valley Fever.



When present, symptoms usually occur between 7 to 21 days after breathing in spores, and can include:

- Cough
- Fever
- Chest pain
- Headache
- Muscle aches
- Rash on upper trunk or extremities
- Joint pain in the knees or ankles
- Fatigue

Symptoms of Valley Fever can be mistaken for other diseases such as the flu (influenza) and TB (tuberculosis), so it is important for employees to obtain medical care for an accurate diagnosis and possible treatment.

While there is no vaccine to prevent Valley Fever, the following important steps must be taken in order to limit risk:

- Determine if the worksite is in an endemic area. Contact the local health department for more information about the risk in the county GEI is performing work that may disturb soils.
- Prepare work plans and work practices that reduce employee's exposure, which may include:
    - Provide air conditioned cabs with properly maintained dust filters for vehicles that generate heavy dust and make sure employees keep windows and vents closed.
    - Suspend work during heavy winds.
- When exposure to dust is unavoidable, National Institute for Occupational Safety and Health (NIOSH)-approved respiratory protection with particulate filters rated as N95, N99, N100, P100, or High Efficiency Particulate Air (HEPA) must be provided. The Project Manager must work with the Safety Team to develop and implement a respiratory protection program in accordance with California's Occupational Safety and Health Administration (Cal/OSHA's) Respiratory Protection standard (8 CCR 5144) for the project.
- Take measures to reduce transporting spores offsite, such as:
    - Clean tools, equipment, PPE, and vehicles before transporting offsite.
    - If employee's clothing is likely to be heavily contaminated with dust, provide coveralls and change rooms, and showers where possible.

1.8 Injury Reporting

If a GEI employee suffers an injury, bite, or sting on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Safety Officer.

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or Supervisor/Project Manager and submitted to the People & Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, Human Resources, and/or the receipt of the Incident Report Form, the Regional Health & Safety Officer (RHSO) will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health and Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.9 Limitations

Follow safety procedures as defined in the site-specific HASP. Appropriate PPE must be worn correctly to provide the intended level of protection.

1.10 References

http://www.cdc.gov/ncidod/dvbid/westnile/index.htm
http://www.cdc.gov/ncidod/dvbid/westnile/qa/insect_repellent.htm
http://www.epa.gov/pesticides/health/mosquitoes/insectrp.htm
http://www.cdc.gov/niosh/topics/lyme/
Protecting Yourself from Ticks and Mosquitoes, NIOSH Fast Facts, Publication No. 2010-119
http://npic.orst.edu/pest/mosquito/ptc.html
http://www.cdc.gov/features/valley-fever-10-things/
https://www.cdph.ca.gov/HealthInfo/discond/Documents/VFGeneral.pdf
https://blog.epa.gov/blog/tag/mosquitoes/

1.11 Attachments

None

1.12 Contact

Health&SafetyTeam@geiconsultants.com

1.13 Review History

- June 2016
- June 2014
- November 2013
- October 2010

STANDARD OPERATING PROCEDURES

SOP No. HS-002 Infectious Materials and Bloodborne Pathogens Exposure Control Plan

1.1 Objective

GEI personnel may come in contact with potentially infectious agents (materials) when performing first aid or cardiopulmonary resuscitation (CPR). Employees may also come into contact with these agents when working at certain contaminated sites (i.e., urban sites, discarded contaminated needles, or sewer outfall exposures). This standard operating procedure (SOP) has been developed to minimize the potential for exposure to employees who may contact, directly or indirectly, infectious agents.

1.2 General

This SOP is intended for use by employees engaged in work with the potential for contact with infectious materials and bloodborne pathogens. The site-specific health and safety plan (HASP) should include a hazard assessment for the project that identifies the potential for encounters with infectious materials or bloodborne pathogens and the control methods to be implemented by GEI employees. Exposure determinations are made by listing job functions impacted by potential exposure. The HASP will list job classifications or tasks in which occupational exposure could occur such as employees collecting samples or expected to provide First Aid. These hazards should be reviewed in the project safety briefing and documented on the Project Safety Briefing form, found on the Safety page of the GEI intranet.

Engineering and work practice controls shall be used to eliminate or minimize employee exposure. Exposure determinations are made without regard to the use of personal protective equipment. When differentiation between body fluid types is difficult or impossible, all body fluids shall be considered potentially infectious materials. *Universal Precautions (i.e., treat all potentially infectious materials as if it were infected) will be used by GEI employees.*

1.3 Exposure Control Plan

1.3.1 Standard Procedures

A written Exposure Control Plan applicable to potential occupational exposure to blood or other potentially infectious materials will be developed as necessary based on project hazards. This plan will be accessible to each affected employee.

Sampling of materials containing potentially infectious materials will be performed in a manner that minimizes the potential for creating splashes, droplets, or aerosols. Mechanical pipetting devices will be used for manipulating sanitary sewer effluent. Mouth pipetting is prohibited.

The use of glassware or equipment with sharp or pointed edges will be kept at a minimum to reduce the potential of injury that would create a direct route of entry into the body for infectious materials.

Minor cuts, scratches, or other breaks in the skin barrier will be covered prior to the handling of infectious materials. Employees experiencing exudative lesions or weeping dermatitis will refrain from direct contact with infectious materials.

Eating, drinking, smoking, or application of cosmetics is not permitted in areas where potentially infectious materials are handled or sampled.

Employees will wash and disinfect their hands, face, or other potentially contaminated skin surfaces upon completing the handling of infectious or potentially infectious agents or after rendering first aid. Hand washing facilities are readily available at all work locations. When provision of hand washing facilities is not feasible, either an appropriate antiseptic hand cleanser used in conjunction with clean cloth/paper towels or antiseptic towelettes will be provided.

All equipment and environmental and working surfaces will be cleaned and decontaminated after contact with blood or other potentially infectious materials. Specimens of blood or other potentially infectious materials (i.e. bandages) will be placed in a container which prevents leakage during collection, handling, processing, storage, transport, or shipping.

1.3.2 Personal Protective Equipment

When there is a potential for occupational exposure GEI will provide, at no cost to the employee, appropriate personal protective equipment (PPE). PPE will be worn to reduce the potential of exposures to splashes or aerosols. At a minimum, PPE will include safety glasses and appropriate gloves, but may also require the use of face, respiratory, foot, and full-body protection. Refer to the site-specific HASP for specific PPE requirements.

Disposable PPE used in the handling or sampling of infectious materials will be appropriately disposed of and not reused.

1.3.3 Medical Monitoring

Medical monitoring is required for an employee when a potential workplace exposure has occurred. GEI will make available the hepatitis B vaccine and vaccination series to all employees who have occupational exposure, and post-exposure evaluation and follow-up to all employees who have had an exposure incident. These are made available at no cost to the employee. The employee must follow the GEI Incident Reporting procedures regarding the potential exposure as soon as possible. For infectious agents in which a medically accepted vaccination has been developed (e.g., hepatitis B virus) (HBV) potentially exposed employees will be given the option to receive an inoculation at no cost. Employees who have been exposed will be given the option to receive a confidential medical evaluation also at no cost. Required records for exposed

employees will be kept confidential. GEI will keep these medical records for at least the duration of employment plus 30 years and will be maintained for 3 years from the date on which the training occurred. All records required to be maintained shall be made available and GEI will comply with the requirements involving transfer of records set forth in 29 CFR 1910.1020(h).

1.3.4 Training

Training will be conducted at the time of initial assignment to tasks where exposure may take place and at least annually thereafter. All training for employees shall be provided within one year of their previous training. Employees with a reasonable risk for exposure must complete Bloodborne Pathogen training covering the following topics:

- An explanation of the Occupational Health and Safety Administration (OSHA) bloodborne pathogen standard.
- A general explanation of bloodborne diseases.
- An explanation of the modes of transmission of bloodborne diseases.
- Communications of hazards to employees.
- An explanation of the GEI's Bloodborne Pathogen SOP and exposure control plan.
- Appropriate methods for recognizing tasks that involve potential exposure.
- An explanation of the use and limitations of methods to prevent exposure.
- Proper types, use, handling, decontamination, and disposal of PPE.
- The availability of HBV vaccines and the procedures for obtaining a vaccination.
- Appropriate actions to take during an emergency involving bloodborne pathogens.
- Post-exposure procedures.
- An explanation of required signs and labels.

1.4 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Health and Safety Officer (RHSO).

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or Supervisor/Project Manager and submitted to the People and Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, Human Resources, and/or the receipt of the Incident Report Form, the RHSO will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health and Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other

appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.5 Limitations

Follow safety procedures as defined in the site-specific HASP. Appropriate PPE must be worn correctly to provide the intended level of protection.

1.6 Attachment

None

1.7 Reference

OSHA 29 CFR 1910.1030 – Bloodborne Pathogens

1.8 Contact

SafetyTeam@geiconsultants.com

1.9 Review History

- October 2018
- June 2016
- June 2014
- November 2013
- January 2011
- November 2010

STANDARD OPERATING PROCEDURE
HS-004 Driver Safety

1.1 Objective

GEI has implemented a Driver Safety Procedure to encourage safe driving habits and promote the ongoing safety of our staff and the communities where we work. For more information, refer to the Operation of Vehicles section of GEI's Employee Handbook.

This Standard Operating Procedure (SOP) provides requirements and recommendations to minimize the potential risks while operating or riding in a motor vehicle.

1.2 General

GEI employees will adhere to the following requirements when operating a vehicle while conducting business on behalf of GEI. These requirements apply to GEI fleet, rental, and personal vehicles used to conduct GEI business:

- Employees must maintain a valid driver's license.
- Employees using a personal vehicle for work-related travel must have proper insurance coverage that meets the requirements in the state/province in which they reside.
- Employees are expected to follow state, local, and federal laws including road signs, vehicle operating requirements, and other applicable regulations for the juristiction they are driving.
- Employees must wear their safety belt while in a moving vehicle.
- Vehicle incidents will be reported in accordance with GEI's Incident Reporting procedures (refer to GEI's Safety App or the Safety Resources page on GEI Connections).
- Vehicles will be properly maintained and safely operated (refer to Driving for Company Business in GEI's Handbook and the vehicle Owner's manual).
- Employees will follow safe driving behaviors, which include limiting distractions such as manipulating radios, use of cell phones (see Section 1.5) or other equipment/items that may cause a distraction.
- Smoking in GEI vehicles and rentals is not permitted. When driving a personal vehicle for business, avoid smoking while driving.

1.3 Mobilization and Demobilization of a Vehicle

GEI employees should follow the following guidelines to confirm their safety prior to and while operating a vehicle:

- Before entering the vehicle, complete a 360° inspection of the vehicle and surrounding area to identify vehicle safety issues and hazards that could be within the travel path. Do not drive a vehicle that has any safety issues.
- If driving an unfamiliar vehicle (rental or GEI fleet vehicle), it is the driver's responsibility to orient themselves to the vehicle by becoming familiar with the size, dashboard, center console, steering controls, locating the turn signals, windshield wipers, lights, emergency flashers, and the heating, air conditioning, and defrost controls, as well as noting if the vehicle has anti-lock braking system (ABS[1]).
- Adjust mirrors (rear and side).
- Adjust seats to be situated as far back as safely practical, away from the air bag, located in the steering wheel.
- Confirm that cell phones, tablets, or other potentially distracting items are safely secured and put away prior to operating the vehicle.
- At the end of the workday, complete a 360° inspection of the vehicle and report any vehicle safety issues.

1.3.1 Parking a Vehicle

- Park in designated areas or a safe area away from heavy equipment, hazardous features, or heavily trafficked areas.
- Avoid crowding neighboring vehicles (park in the middle of your spot)
- When parking a vehicle, the employee should position the vehicle in a manner which reduces or eliminates the need to operate the vehicle in reverse.
- When parking on the side of the road, the employee should minimize the encroachment of the vehicle onto the travelled lanes of the road and use pylons to mark a safe area around their vehicle.
- At night, park in a well-lit area and as close to your destination as possible.

[1] ABS is a mechanism that allows the wheels on a vehicle to maintain contact with the surface of the road, based on inputs from the driver (braking), to prevent the wheels from locking up (ceasing rotation) and to avoid an uncontrolled skid.

- When leaving the vehicle, confirm that any supplies or equipment that is meant to stay in the vehicle are secured and cannot be seen. If these items cannot be hidden, take them with you.

1.3.2 Backing Up a Vehicle

- If backing up a vehicle is necessary, complete a 360° inspection with emphasis on identifying nearby fences, poles, drop-offs, buildings, or other objects that may be in your path.
- Know your vehicles clearance and check for overhead obstructions such as tree limbs, wires, or other potential clearance-related obstacles.
- Use a spotter whenever possible. The spotter can be another GEI employee, or on-site personnel. Do not allow the spotter to be positioned directly behind the vehicle or walk backwards while giving you instructions. They should be off the driver's side where you can see them in your side mirror.
- Use available backup cameras as supplementary support while in reverse to assist with blind spots and identify if conditions have changed.
- When backing up at night, use additional lights to increase visibility.

1.4 Driving Defensively

Driving defensively means not only taking responsibility for oneself and actions but also keeping an eye on "the other guy." Good defensive drivers may be able to anticipate what the other driver will do next. GEI recommends the following guidelines to help reduce risks while driving:

- Do not start the vehicle until each passenger and any belongings are secured in the vehicle.
- Remember that driving above or below the speed limit can increase the likelihood of a collision.
- Be aware of impaired drivers; if a car is straddling the center line, weaving, making wide turns, stopping abruptly, or responding slowly to traffic signals, the driver may be impaired or using a cellular telephone. Avoid an impaired driver by turning right at the nearest corner or exiting at the nearest exit.
    - If it appears that an oncoming car is crossing into your lane, pull over to the roadside, sound the horn, and flash the headlights.
    - If an unsafe or suspicious driver is observed, notify the police.
- Follow the rules of the road. Do not contest the "right of way" or try to race another car during a merge. Be respectful of other motorists.
- Allow large vehicles, including tractor trailers, extra breaking distance, turning radius, and avoid traveling in the other driver's blind spots.

- Do not follow too closely. GEI employees should use a minimum of "3-second following distance." Or two vehicle lengths, whichever is greater.
- While driving, be cautious, aware, and responsible.
- Use extra caution, observe road signs, and reduce speed in construction areas and school zones.
- Maintain situational awareness by scanning your surroundings and regularly checking your blind spots. Look for and avoid pedestrians, bicyclists, and motorcyclists.

1.5 Cellular Phone Use and Other Distractions

Refer to the *Cell Phone Use/Texting While Driving* section of the GEI Employee Handbook for GEI's policy on the use of cellular telephones while operating a vehicle.

1.6 Drugs and Alcohol

The use of illegal drugs or alcohol is prohibited when driving a vehicle on GEI business. Be aware of the side effects of prescription and over-the-counter medications which can impair an employee's ability to drive.

1.7 Adverse Driving Conditions

When operating a vehicle, its possible adverse driving conditions may be encountered. Below is a list of possible conditions and how they can be mitigated.

1.7.1 Driving at Night

Vision maybe limited at night due to impairment of the driver's depth perception, color recognition, and peripheral vision. Another factor adding danger to night or early morning driving is fatigue. Drowsiness makes driving more difficult by dulling concentration and slowing reaction time. Effective measures to minimize these hazards by preparing the car and following guidelines:

- Check the headlights to confirm they are properly aimed. If you notice the headlights are not properly aimed, report it to the Branch Manager, or if applicable the rental car agent. Misaimed headlights blind other drivers and reduce the driver's ability to see the road.
- Smoking before driving and while driving is discouraged. Nicotine and carbon monoxide can hamper night vision.

- Increased vigilance 1 hour before sunset and 1 hour after sunset is encouraged. Twilight is one of the most difficult times to drive, because the eyes' pupils are constantly changing to adapt to the growing darkness:
    - Always use headlights at dusk and at dawn; lights will not help the driver see better in early twilight, but they will make it easier for other drivers to see your car.
    - Drive at a speed that allows you to see the road that is within the headlights span. Driving in a manner that prevents you from seeing hazards as they are illuminated is known as overdriving the headlights; it may be necessary for the driver to reduce speed to be prepared to brake within the illuminated area of the headlights.
    - If an oncoming vehicle does not lower beams from high to low, avoid glare by watching the right edge of the road and using it as a steering guide.
- The driver should make frequent stops for light snacks and exercise. If the driver is too tired to drive, stop in a safe area and get some rest.

1.7.2 Snow/Freezing Conditions

When snow and ice are present, be prepared by following these winter driving safety tips.

Prepare the Vehicle Before a Snowstorm

- Check under the hood and take a look at the vehicles cooling system. Make sure the vehicle contains adequate antifreeze and the hoses are in good condition.
- Test heaters and defrosters ahead of time to make sure they are in good working condition.
- Test the windshield wipers and check the condition of the wiper blades. If wipers leave streaks on the windshields, replace the blades at the next possible opportunity. Keep the receipt to expense the cost with GEI or with the car rental company.
- It is recommended that a windshield washer/antifreeze solution is used during winter conditions.
- Check the lights on the vehicle and periodically clear them of snow and dirt.
- Vehicle batteries need extra power in cold conditions. Make sure the battery's terminals are clean and cables are secure.
- Determine if the vehicle has an ABS system.
- Keep the gas tank at least half-full in the winter to help avoid gas line freeze up.

Driving During and After a Snowstorm

- Wear sunglasses to aid in limiting reflection from snow and eye fatigue, as needed.
- Be aware of blind spots created by snowbanks. Be extra cautious of pedestrians and other vehicles in intersections.
- Allow extra time for braking and increase the distance between your car and the car immediately in front of the car.
- Reduce speed and do not exceed the posted limit.
- If the tires starts to lose traction, remove the foot off the gas and gradually reduce speed. Accelerate slowly once traction is regained.
- Use appropriate braking technique:
    - No ABS: steer into the skid. This will bring the back end of the car in line with the front. Avoid using the brakes.
    - ABS: firmly brake as you steer into the skid.

1.7.3 Driving In the Rain

To prevent losing control of the car on wet pavement, take these preventive measures.

- Prevent skids by driving slowly and carefully, especially on curves.
- Steer and brake with a light touch.
- When necessary to stop or slow, do not brake hard or lock the wheels.
- Maintain foot on the brake pedal.

Skidding

If the car begins to skid, ease the foot off the gas, and carefully steer the car in the direction you want the front of the car to go. For cars without ABS, avoid using the brakes. This procedure, known as “steering into the skid,” will bring the back end of the car in line with the front. If the car has ABS, brake firmly as you steer into the skid.

Hydroplaning

Hydroplaning happens when the water in front of the tires builds up faster than the car’s weight can push it out of the way. The water pressure causes the car to lose contact with the road surface and slide on a thin layer of water between the tires and the road. At this point, the car can be completely out of contact with the road, making it possible for the driver to skid or drift out of the lane, or even off the road.

To avoid hydroplaning, keep the tires properly inflated and maintain good tread on the tires. If tires need to be replaced on a company vehicle, notify the branch manager or their designee. Slow down when roads are wet, and stay away from puddles. Try to drive in the tire tracks left by the cars in front of the vehicle. If the car begins to hydroplane, do not brake or turn suddenly. This could throw the car into a skid. Ease the foot off the gas until the car slows; accelerate slowly once traction is regained. If braking is needed, do so gently with light pumping actions. If the car has ABS, brake normally; the car's computer will mimic a pumping action, as necessary.

If weather conditions worsen to the point where the driver is not comfortable driving, pull the vehicle over to a safe location until conditions improve. Do not drive during severe weather conditions. Do not attempt to drive on roads with standing water or that have been flooded. Find an alternate route if these conditions exist.

1.7.4 Off Road

If operation of a vehicle is required off public or private roads or in situations where four-wheel-drive vehicles are required, the appropriate vehicle for the situation will be used.

Be sure any gear or equipment is secured inside the vehicle so it doesn't bounce around while the vehicle is off-road.

- Clearance: know the underside of the vehicle. Look under the vehicle and learn where the lowest-hanging parts are located so they are not damaged.
- Scout tricky terrain on foot. Don't hesitate to get out of the vehicle to examine, up close, the terrain and soil conditions. And be sure to scout out what's on the other side of a hill ahead of time so there are no surprises.
- Drive cautiously. Drive, "as slow as possible, as fast as necessary." Remember to use the gears to efficiently manage engine power, braking, and torque.
- Create a mental picture. Look ahead and visualize the paths to the vehicle will travel. Follow those paths.
- Drive straight up and down hills. Avoid diagonal lines that put the vehicle in a situation where it might roll.

1.8 Driver Training

GEI employees take driver safety training as part of their annual safety training. This training will be assigned through GEI's e-learning provider.

1.9 Injury Reporting

GEI employees will report incidents involving GEI personnel or subcontractor personnel, such as: lost time injuries, injuries requiring medical attention, near miss incidents, fires, fatalities, accidents involving the public, chemical spills, vehicle accidents, and property damage. The following steps must be followed when an incident occurs:

1. For incidents involving life-threatening situations or serious injury that require emergency response personnel (Police, Fire, EMS), call 9-1-1 from a safe area.
2. **Stop work** activity to address any injury, illness, property damage, spill, or other emergency.
3. Call Medcor Triage at 1-800-775-5866 to speak with a medical professional following any injury or illness.
4. Notify your Supervisor/Project Manager of the incident or injury.
5. Complete an incident report using the GEI Incident Report Form located on the GEI Safety Smartphone App, GEI Connections intranet page, or in the project HASP.
6. Resume work activity if all steps above have been completed and it is safe to do so.

For vehicle accidents involving another vehicle or damage to property, the employee will take pictures of each vehicle or property involved in the incident and obtain a police report. In some municipalities police will not be dispatched to a non-injury accident, but every effort needs to be made to try and obtain the report.

1.9.1 Injury Triage Service

If a GEI employee experiences a work-related injury that is not life-threatening, the employee will initiate a call to Medcor Triage at 1-800-775-5866. The injured employee will detail any medical symptoms or complaints which will be evaluated by a Registered Nurse (RN) specially trained to perform telephonic triage. The RN will recommend first aid self-treatment or refer the injured employee for an off-site medical evaluation by a health professional at a clinic within GEI's workers compensation provider network. GEI employees are still required to follow our Accident Reporting procedures as listed above.

1.10 Limitations

Follow safety procedures as defined in the site-specific HASP.

1.11 References and Resources

GEI Consultants, Inc. Employee Handbook
Ontario Occupational Health and Safety Act (OHSA)
US Occupational Health and Safety – Motor Vehicle Safety
National Safety Council

1.12 Attachments

None

1.13 Contact

Safety_Team@geiconsultants.com

1.14 Review History

- July 2023
- December 2017
- November 2016
- May 2014
- November 2013
- January 2011

STANDARD OPERATING PROCEDURES

HS-005a Electrical Safety

1.1 Objective

Electrical hazards are typically the most serious physical hazards GEI employees encounter when working on or near an electric substation, conducting intrusive activities such as excavation or drilling, using powered hand tools, or working near overhead utilities. This Standard Operating Procedure (SOP) has been developed to minimize the potential for exposure to energized electrical equipment while performing work activities.

1.2 General

Employees who face a risk of electric shock will be trained and become familiar with electrically-related safety practices in the GEI annual Health and Safety training. This training will include safety-related work practices that pertain to their respective job assignments and minimum approach distances.

An activity hazard analysis conducted for the project scope of work will evaluate the potential for electrical shock and be incorporated in the site-specific health and safety plan (HASP). In addition, site-specific training will be conducted by the Project Manager or their designee to discuss the project electrical hazards and include a review of the HASP requirements.

Annual health and safety training will include review of this electrical safety SOP and GEI's lockout/tagout requirements. Retraining will be required when there is a change in job assignments; a change in the energy control procedures; or a new hazard is introduced. Retraining may also be conducted through the Project Safety Briefing and documented on the Project Safety Briefing form.

Safe work practices will be employed to prevent electric shock or other injuries resulting from either direct or indirect electrical contacts when work is performed near or on equipment or circuits that are or may be energized. Employees may not enter spaces containing exposed energized parts unless proper illumination, protective shields, protective barriers, or insulating materials (if necessary) are provided to enable employees to work safely. If portable ladders are needed near electrical equipment, they will have non-conductive side rails.

1.3 Personal Protection

Measures to mitigate exposure to overhead and subsurface electrical transmission and distribution lines should be adhered to at all times when working adjacent to electrical hazards. These measures include:

- Electrical Hazard (EH)-rated footwear and hardhat are required when working onsite. Additional personal protective equipment (PPE) may be needed as referenced in the site specific HASP.
- Avoid carrying tools/equipment above waist height if overhead electric hazards exist.
- Maintain the minimum approach distance (MAD) from bus bars, transformer/capacitor electrodes, and overhead transmission/distribution lines.
- Stop work immediately and vacate the work area in the event lightning is observed.

Measures of protection that should be adhered to within an active substation perimeter or if work adjacent to the substation perimeter consists of intrusive activities:

- Contact utility clearance agency at least 48 hours prior to any invasive activities for mark out of underground public utilities.
- Obtain the most recent as-built drawings of the transmission/distribution line layout from the client.
- Mark out of underground transmission/distribution lines by the client survey/mark out personnel if applicable.
- Conduct work under the supervision of the client's Health and Safety representative per client requirements.
- Conductive items such as jewelry or clothing containing metals will not be worn unless they are rendered non-conductive by insulating means such as covering or wrapping with materials that specifically rated as non-conductive, or other insulating means.
- Use hand digging tools specifically designed for use on substation property (i.e., insulated digging bar, long-handled spoon shovel, etc.). In addition, rubber gloves and fire-resistant clothing are required if hand digging in a substation.
- Use insulated lineman's gloves when handling equipment that may come into contact with electrical utilities.
- EH-rated footwear is required when working onsite.
- Flame resistant clothing that meets National Fire Protection Association (NFPA) 70E standards is to be worn.

- Avoid carrying tools/equipment above waist height if overhead electric hazards exist.
- Ground vehicles or equipment **within the substation perimeter** using 4-aught gauge grounding cable. Adhere to specific client or site requirement for grounding.
- Maintain a minimum clearances on substations in accordance with 29 CFR 1910.333 and NFPA 70E standards when working near energized equipment.
- Maintain a minimum offset of 3 feet from marked underground transmission/distribution lines.
- Avoid working within or outside of a substations in conditions of high humidity, rain, or thunderstorms.
- Stop work immediately and vacate the work area in the event lightning is observed.

1.3.1 Minimum Approach Distance

According to OSHA, the MAD is "…the closest distance an employee is permitted to approach an energized or grounded object." MADs ensure that employees do not approach or take any conductive object closer to energized equipment than the allowed distances. All GEI employees conducting work onsite must follow the MADs shown below:

Nominal Voltage in Kilovolts	Minimum Approach Distance: Phase to Ground Exposure
0.05 to 1.0	Avoid Contact
1.1 to 15.0	2'1"
15.1 to 36.0	2'4"
36.1 to 46.0	2'7"
46.1 to 72.5	3'
72.6 to 121	3'2"
138 to 145	3'7"
161 to 169	4'
230 to 242	5'3"
345 to 362	8'6"
500 to 550	11'3"
764 to 800	14'11"

Reference: Table R-6 in OSHA regulation CFR 1910.269 (l) (10)

Unqualified employees must maintain a 10-foot distance from all electrical sources. Vehicular and mechanical equipment will also maintain minimum clearance distances of 10 feet.

Site personnel will assume that all electrical equipment at surface, subsurface, and overhead locations are energized until the equipment has been designated as de-energized by a client representative. Client representatives will be responsible for de-energizing and lockout/tagout of all electrical equipment. If the equipment cannot be de-energized, work will stop and the GEI Field Representative and/or the Site Safety Officer (SSO) will consult with the Project Manager and CHSO. GEI will notify the client prior to working adjacent to this equipment, and will verify that the equipment is energized or de-energized in the vicinity of the project area.

All power lines that have been indicated by the client to be de-energized must be locked out by the client or their representative, such that the lines cannot be energized when personnel are working near them. The lines will not be unlocked and re-energized until GEI notifies the client that they have completed work in the area and that all personnel are clear of the area. The client representatives will thoroughly familiarize GEI personnel with site-specific lockout/tagout procedures during the site orientation. Conductors and parts of electrical equipment that have been de-energized, but not been locked or tagged out, shall be treated as live/energized. Only qualified persons may work on electric circuit parts or equipment that has not been de-energized. Such persons will be made familiar with the use of special precautionary techniques, PPE, insulating and shielding materials, and insulated tools.

If power lines cannot be de-energized, the SSO will consult with the client to discuss how to proceed. Work tasks will only commence after determining that a safe working distance (MAD) can be maintained and all personnel working in the area have been informed of the limitation. All work performed within the substation boundaries requires the use of task-specific PPE as described in the HASP.

GEI will verify that the contractor or subcontractor has located and marked all electric, gas, water, steam, sewer, and other utility service lines before any intrusive work is started. In each case, any utility company that is involved should be notified in advance by the contractor, and its approval or services will be obtained, if necessary.

1.3.2 Ground-Fault Circuit Interrupters (GFCI)

GEI employees will use approved ground-fault circuit interrupters for personnel protection when using electrical tools and equipment. Equipment will be plugged into receptacles protected by a GFCI, extension cords with built-in GFCIs, or a GFCI adapter is to be used.

1.3.3 Equipment Inspection

Each extension cord, plug and receptacle, and any equipment connected by the cord and plug, will be visually inspected before each day's use for external defects, such as deformed or missing pins or insulation damage, and for indications of possible internal damage. Each receptacle and plug will be tested for correct attachment of the equipment grounding conductor. The equipment grounding conductor will be connected to its proper terminal.

Inspection of equipment will be performed before first use; before equipment is returned to service following any repairs; before equipment is used after any incident which can be reasonably suspected to have caused damage (e.g., when an extension cord is run over), and at intervals not to exceed 3 months. Cords and receptacles which are fixed and not exposed to damage will be tested at intervals not exceeding 6 months. Equipment found damaged or defective will not be used and will be properly tagged as "Out of Service"; notify the Branch Manager so that the equipment can be replaced or repaired. If the equipment cannot be repaired it will be disposed of properly.

1.4 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Safety Officer.

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or supervisor/project manager and submitted to the People & Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, Human Resources, and/or the receipt of the Incident Report Form, the Regional Health & Safety Officer (RHSO) will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health & Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.5 Limitations

- Follow safety procedures as defined in the site-specific HASP at all times.
- If lockout/tagout procedures are going to be performed by GEI employees or GEI subcontractors, the specific procedure will be reviewed with the CHSO and the RHSO.

1.6 References

OSHA 29 CFR 1910.147 Appendix A – The control of hazardous energy (lockout/tagout)

1.7 Attachments

None

1.8 Contact

Health&Safetyteam@geiconsultants.com

1.9 Review History

- February 2017
- May 2014
- October 2011
- August 2010 (Titled Lock Out/Tag Out)

STANDARD OPERATING PROCEDURES

HS-005b Lockout/Tagout

1.1 Objective

Lockout/tagout, otherwise referred to as the control of hazardous energy, is the procedure used to eliminate the hazards from equipment that contains hazardous energy prior to working on or around this equipment. Energy sources that are controlled using lockout/tagout procedures include electrical, mechanical, hydraulic, pneumatic, chemical, or thermal. Other energy sources can include stored energy such as equipment held under tension, pressurized lines, and capacitors. Lockout/tagout also applies to electric generation, and pumping plant inspections and appurtenances.

This Standard Operating Procedure (SOP) has been developed to protect employees from exposure to these types of hazardous equipment while performing work activities on and around equipment. GEI employees will follow the client's lockout/tagout procedures, when available. If the client does not have specific lockout/tagout procedures in place, use these guidelines at a minimum.

This lockout/tagout program will be reviewed by the Safety Team annually. Health and Safety reviews are conducted by the Safety Team to document compliance with these requirements.

1.2 General

Employees servicing, maintaining, or working near machines or equipment may be seriously injured or killed if hazardous energy is not properly controlled. Injuries resulting from the failure to control hazardous energy during maintenance activities can be serious or fatal. Injuries may include electrocution, burns, crushing, cutting, lacerating, amputating, or fracturing body parts.

Employees who have the potential to work with or around equipment where lockout/tagout is needed, will be trained and become familiar with GEI's lockout/tagout procedures. This training will include safety-related work practices that pertain to their respective job assignments.

An activity hazard analysis conducted for the project scope of work will evaluate the potential for exposure to hazardous energy and be incorporated in the site-specific Health and Safety Plan (HASP). In addition, site-specific training will be conducted by the Project Manager or their designee to discuss the project lockout/tagout requirements and include a review of the HASP requirements.

Annual health and safety training will include review of GEI's lockout/tagout requirements. Retraining will be required when there is a change in job assignments, a change in the energy control procedures, or a new hazard is introduced. Retraining may also be conducted through the Project Safety Briefing and documented on the Project Safety Briefing form.

1.3 Roles and Responsibilities

There are two categories of employees in a lockout/tagout program, Authorized and Affected. Regardless of category, employees are required to comply with the restrictions and limitations imposed upon them during the use of lockout/tagout procedures.

1.3.1 Authorized Employees

Authorized employees are responsible for following established lockout/tagout procedures. An authorized employee is defined as a person who locks out or tags out machines or equipment in order to perform service, maintenance, or work near a piece of equipment that contains or has the potential to contain hazardous energy.

Authorized employees will receive training on the following topics:

- Recognition of hazardous energy sources, types, and magnitudes of energy.
- Methods and means required for energy isolation and control.
- Purpose and use of the lockout/tagout procedures.
- Limitations of the use of tagout systems.

1.3.2 Affected Employees

An affected employee is defined as a person whose job requires him/her to operate or use a machine or equipment on which work activities are being performed under lockout or tagout, or whose job requires him/her to work in an area in which such activities are taking place. Affected employees do not attempt to restart or re-energize machines or equipment that are locked out or tagged out.

Authorized employees will receive training on the following topics:

- Purpose and use of the lockout/tagout procedures.
- Prohibition relating to attempting to restart or reenergize equipment that has been locked or tagged out.

1.4 Lockout/Tagout Procedure

Lockout/tagout procedures will be followed when working on or around machines, equipment, or any system where the unexpected energization or start-up of the machine or equipment or release of stored energy can cause injury to the employee.

GEI uses a red Master Lock® 1 31/32-inch high body high-visibility aluminum padlock, keyed differently, with a 1 1/16-inch shackle, or its equivalent for lockout activities. An employee's lock and tag must not be removed by anyone other than the employee who installed the lock and tag unless removal is accomplished under the direction of the CHSO.

When GEI employees are working on project sites where a group lockout is required, the locked-out device will first be secured using a folding scissors clamp that has many padlock holes capable of holding it closed. Each employee/subcontractor will apply their own padlock to the clamp. The locked-out device cannot be activated until all workers have signed off on their portion of the project and removed their padlock from the clamp. The lead contractor responsible for the group lockout will assign an authorized employee who has primary responsibility for the number of employees working under the protection of a group lockout or tagout device. GEI employees will not act as the Authorized Employee.

Only GEI employees who have completed lockout/tagout training may conduct a lockout/ tagout procedure. Normally GEI employees will follow the client's lockout/tagout procedures. If the client does not have specific lockout/tagout procedures to follow, use these guidelines at a minimum:

- Prior to conducting any lockout/tagout procedures, notify all affected employees.
- Identify the type, magnitude, and hazards of the energy that the machine/ equipment uses.
- Shut down the machine/equipment by the normal stopping procedure (stop button, open switch, close valve, etc.).
- Disconnect the machine/equipment from the energy source.
- Stored or residual energy (such as capacitors, springs, elevated machine members, rotating flywheels, hydraulic systems, and air, gas, or water pressure, etc.) must be dissipated or restrained by methods such as grounding, repositioning, blocking, bleeding down, etc.
- Using the correct type of lockout/tagout device, lockout the machine/equipment with an assigned individual lock by placing a lock on each energy isolating device. Only the employee performing the work should have a key to this lock.
- Locks can be obtained from the RHSO. This lock will have a GEI-approved tag that allows the employee to write their name and date on the tag.

- Lockout devices, where used, will be affixed in a manner that will hold the energy isolating devices in a safe or off position. Locking devices will always be used on energy isolating devices designed with the capability of being locked.
- Tagout devices, where used, will be affixed in such a manner as will clearly indicate that the operation or movement of energy isolating devices from the safe or off position.
- Where a tag cannot be affixed directly to the energy isolating device, the tag shall be located as close, and as safely as possible, to the device in a position that will be immediately obvious to anyone attempting to operate the device.
- Confirm that the machine/equipment has been disconnected by attempting to operate or turn the machine/equipment on. If the machine does not operate, it is now locked out.
- If, during repair or maintenance activities, the machine or equipment is required to be energized, identify the location of lockout/tagout device(s) that can be removed to accomplish this. Only the lockout/tagout devices absolutely necessary to allow the equipment or machine to be energized or partially energized should be removed. After the equipment or machine has been cycled or jogged, the lockout/tagout devices will be reapplied and the steps above will be repeated prior to commencing work.
- When the machine/equipment is ready to be returned to service, make sure all tools are removed and the system is operationally intact. Verify that all employees are in a safe position and have been removed from the area.
- Remove the lockout/tagout devices and reenergize the machine/equipment.
- Notify affected employees that the service or maintenance work is complete and the machine/equipment is ready for use.

1.5 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Safety Officer.

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or supervisor/project manager and submitted to the People & Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, Human Resources, and/or the receipt of the Incident Report Form, the Regional Health & Safety Officer (RHSO) will

conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health & Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.6 Limitations

Follow safety procedures as defined in the site-specific HASP at all times.

If lockout/tagout procedures are going to be performed by GEI employees or GEI subcontractors, the specific procedure will be reviewed the Safety Team, prior to any work being performed.

1.7 References

OSHA 29 CFR 1910.147 Appendix A – The control of hazardous energy (lockout/tagout)

1.8 Attachments

None.

1.9 Contact

Health&SafetyTeam@geiconsultants.com

1.10 Review History

- November 2016 (Separated from a previous SOP)

STANDARD OPERATING PROCEDURES

SOP No. HS-008a Non-Powered Hand Tools

1.1 Objective

This Standard Operating Procedure (SOP) is intended for use by employees working with non-powered hand tools. The site-specific health and safety plan (HASP) should include a hazard assessment for the project that identifies the hazards associated with the non-powered hand tools that will be used. These hazards should be reviewed during the project safety briefing and documented on the Project Safety Briefing form, found on the Safety page of the GEI intranet.

1.2 General

Misuse of hand tools accounts for the majority of accidents and injuries involving hand tools. Only use a tool for the task which it was designed for. If the right tool isn't available contact the Project Manager and discuss what is needed. Improper maintenance is another leading cause of injuries. Employees using hand tools may be exposed to a number of other potentially serious hazards: falling objects (i.e., objects can fall as a result of contact with tools or objects which are abrasive or splash), harmful dust, fumes mists, vapors, and gases, as well as contact with electrical power sources.

1.2.1 Condition of Tools

All hand tools, whether furnished by GEI or the employee, will be maintained in safe working condition. All hand tools must be inspected before use. Never use a tool if its handle has splinters, burrs, cracks, splits or if the head of the tool is loose. Never use impact tools such as hammers, chisels, punches or steel stakes having mushroomed (flattened) heads. Tag worn, damaged or defective tools "Out of Service" and do not use them; notify your Branch Manager or Project Manager so that the tool can be replaced or repaired. If the tools cannot be repaired they will be disposed of properly. GEI does not issue or permit the use of unsafe hand tools.

1.2.2 Personal Protective Equipment

Employees using hand tools will be provided with the personal protective equipment (PPE) necessary to protect them from the hazard of the tool as well as the associated hazards with using the tool. (i.e., projectile debris, dust, etc.). <u>All employees will wear work gloves, steel toe or composite toe boots, and safety glasses at a minimum</u>. In addition, face shields and hearing protection may be required. Most hand injures can be avoided with the proper use of PPE. PPE must be maintained in good condition, kept clean and properly stored when not in use. More information regarding PPE is located in Section 6 of GEI's Corporate Health and Safety Program.

1.2.3 General Safe Practices

Never wear sandals, open-toed or canvas shoes when working with tools. Always tie back long hair. Avoid loose-fitting clothes which might become entangled in a tool. Always remove rings and other jewelry. Make sure your grip and footing are secure when using large tools. Never carry tools up ladders; use a tool belt, hoist, or a rope. Use extra caution when using tools at heights – a falling tool could kill a co-worker. Always pass a tool to another person by the handle – never toss it to them. Never use a tool with hands are wet, oily, or greasy. Select ergonomically-designed tools for work tasks when movements are repetitive and forceful. Always make sure observers are at a safe distance. Always secure work with a vice, clamp, or other support.

1.3 Non-Power Hand Tools

Non-powered hand tools include anything from axes to wrenches. Even though the tool is powered by human inertia, injuries from improper use of non-powered hand tools often involve severe disabilities.

1.3.1 Knives

Only use a knife with a sharpened blade. Pull the knife through the object and away from your body; pulling motions are easier to manage. Never use a knife if its handle has splinters, burrs, cracks, splits or if the blade is loose. Knives should never be used as screwdrivers, pry bars, or can openers. Never pick up knives by their blades. Always carry knives with their tips/points toward the floor. Never carry knives, scissors, or other sharp tools in pockets. Never attempt to catch a falling knife. When not in use, knives should be stored in sheaths. Box cutters will be self-retracting.

1.3.2 Wrenches

Never use wrenches that are bent, cracked, badly chipped, or having loose or broken handles. Discard any wrench with spread or battered jaws; if the handle is bent; or if a wrench has broken or battered points and notify your Branch Manager so that a replacement can be made. Never slip a pipe over a single head wrench handle to increase leverage. Never use a shim to make a wrench fit. Pull on a wrench using a slow, steady motion. Do not use push force on a wrench; you could lose your balance if the wrench slips.

1.3.3 Screwdrivers

Always match the size and type of screwdriver blade to fit the head of the screw. Do not hold the work piece against your body while using a screwdriver. Never put your fingers near the tip of a screwdriver when tightening a screw. Never use a screwdriver to make a starting hole for screws. Never use a screwdriver as a chisel, pry bar, or nail puller. When performing electrical work, always use an insulated screwdriver. Never use a screwdriver to test the charge of a battery.

1.3.4 Hammers

Never use a hammer if your hands are oily, greasy or wet. Always check behind you before swinging a hammer. Use a claw hammer for pulling nails. Never strike nails or other objects with the "cheek" of the hammer. Do not strike a hardened steel surface, such as a cold chisel, with a claw hammer. Never strike one hammer against another hammer. Never use a hammer as a wedge or a pry bar.

1.3.5 Pliers

Never use pliers which are cracked, broken, or sprung. Never use pliers as a wrench or a hammer. Do not attempt to force pliers by using a hammer on them. Never slip a pipe over the handles of pliers to increase leverage. When performing electrical work, always use insulated pliers. When using diagonal cutting pliers, shield loose pieces of cut material from flying into the air by using a cloth or your gloved hand.

1.3.6 Snips

Never use snips as a hammer, screwdriver, or pry bar. Always wear safety glasses or safety goggles when using snips to cut materials. Always wear work gloves when cutting materials with snips. Keep the blade aligned by tightening the nut and bolt of the snips. Never use straight cut snips to cut curves. Always use the locking clip on the snips when you have finished using them. Never leave or store snips in the open position.

1.3.7 Hand Saws

Always keep handsaws sharp and free of rust to prevent them from binding or jumping. Never carry a saw by the blade. Always hold the work piece firmly against a work table. Keep control of saws by releasing downward pressure at the end of the stroke. Never use an adjustable blade saw such as a hacksaw, coping saw, keyhole saw, or bow saw, if the blade is not taut. Oil saw blades after each use. Never force the saw through the cut as this may cause the saw to buckle or fly out of the groove and cause injury.

1.3.8 Chisels

Only use sharpened chisels. Never use chisels having mushroomed (flattened) striking heads. Whenever possible, hold a chisel by using a tool holder. Clamp small work pieces in a vise and chip towards the stationary jaw of the vise. Chip or cut away from yourself and keep both hands in back of the cutting edge. Always wear safety glasses or a face shield.

1.3.9 Vise and Clamps

Never use a vise having worn or broken jaw inserts, or having cracks or fractures in the body of the vise. Position the work piece in the vise so the entire face of the jaw supports the work piece. When clamping a long work piece in a vise, support the far end of the work piece by using an adjustable pipe stand or saw horse. Never slip a pipe over the handle of a vise to increase leverage. Never use a C-clamp for hoisting materials. Never use a C-clamp as a permanent fastening device.

1.3.10 Jacks

A manufacture's rated capacity must be clearly marked on all jacks and all jacks must have a stop indicator. When using a jack, never exceed the capacity of the stop indicator. Jacks should be lubricated and inspected regularly. When setting up a jack, ensure the base is centered on a firm, level surface. The jack head should also be placed against a level surface. Lift force should be applied evenly. Put a block under the base of the jack when the foundation is not firm. If it seems likely the cap could slip, place a block between the jack cap and load. Immediately block the load after it is lifted.

1.4 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Safety Officer.

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or supervisor/project manager and submitted to the People & Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, Human Resources, and/or the receipt of the Incident Report Form, the Regional Health & Safety Officer (RHSO) will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health & Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.5 Limitations

Follow safety procedures as defined in the site-specific HASP or in the manufacturer's specifications. Appropriate PPE must be worn correctly to provide the intended level of protection. If a hand tool is being used that is not identified in this SOP consult the manufacturer's literature and contact the Safety Team so we can include the information in a future version of this SOP.

1.6 References

OSHA Standards for the Construction Industry, Subpart I
Risk Analytics, LLC Hand Tools Training, 2006

1.7 Attachments

None

1.8 Contact

Health&SafetyTeam@geiconsultants.com

1.9 Review History

- July 2016
- May 2014
- August 2011
- October 2010
- One revision date unable to be found

STANDARD OPERATING PROCEDURES

SOP No. HS-008b Powered Hand Tools

1.1 Objective

This Standard Operating Procedure (SOP) is intended for use by employees working with powered hand tools. The site-specific health and safety plan (HASP) should include a hazard assessment for the project that identifies the hazards associated with the powered hand tools that will be used. These hazards should be reviewed during the project safety briefing and documented on the Project Safety Briefing form, found on the Safety page of the GEI intranet.

1.2 General

Misuse of hand tools accounts for the majority of accidents and injuries involving hand tools. Only operate power tools according the manufacturer's instructions. Employees using power tools may be exposed to a number of potentially serious hazards including being hit by flying material from the work piece; hit by a flying part of a broken tool; explosion or fire resulting from sparks from a tool igniting combustible materials; electric shock from a broken tool, frayed or defective power cord, or improper grounding; exposure to harmful dust, fumes mists, vapors, and gases. Hazards are usually caused by misuse, improper maintenance, improper or inefficient training, and complacency.

1.2.1 Condition of Tools

All hand tools, whether furnished by GEI or the employee, will be maintained in safe working condition with regular maintenance. Always inspect each tool, as well as power cords and attachments, for damage before use. Make sure the power is off and locked out before inspecting. Insure the tool guards are in place and functioning. Ensure that blades, bits, and other attachments are securely fastened. Tag worn, damaged, or defective tools "Out of Service" and do not use them; notify your Branch Manager or Project Manager so that the tool can be replaced or repaired. If the tools cannot be repaired they will be disposed of properly. GEI does not issue or permit the use of unsafe hand tools.

1.2.2 Personal Protective Equipment

Employees using hand tools will be provided with the personal protective equipment (PPE) necessary to help protect them from the hazards of the tool as well as the associated hazards with using the tool. (i.e., projectile debris, dust, etc.). All employees will wear work gloves, steel toe or composite toe boots, and safety glasses at a minimum. In addition, face shields and hearing protection may be required. Most hand injures can be avoided with the proper use of PPE. PPE must be maintained in good condition, kept clean and properly stored when not in use. More information regarding PPE is located in Section 6 of GEI's Corporate Health and Safety Program.

1.2.3 General Safe Practices

Never wear sandals, open-toed or canvas shoes when working with tools. Always tie back long hair. Avoid loose-fitting clothes which might become entangled in a tool. Always remove rings and other jewelry. Never use a tool without its guard in place. Make sure your grip and footing are secure when using large tools. Never carry tools up ladders; use a tool belt, hoist, or a rope. Use extra caution when using tools at heights – a falling tool could kill a co-worker. Always pass a tool to another person by the handle – never toss it to them. Select ergonomically-designed tools for work tasks when movements are repetitive and forceful. Always make sure observers are at a safe distance. Always secure work with a vice, clamp, or other support. Moving work surfaces can cause the tool to "kick back." Use extra caution when using power tools around flammable materials. Use fire curtains when appropriate and keep a properly charged fire extinguisher within a reasonable distance. Never surprise someone using a power tool. Check above, underneath, and behind solid surfaces if possible, to make sure it's safe to proceed and there isn't another person working on the other side.

1.2.4 Guarding

When power tools are designed to accommodate guards, they will be equipped with such guards prior to, and at all times during use. All guards will be in good condition and be adequate to provide protection to the employee. Regulations stipulate that the following parts of a power tool must be guarded: gears, sprockets, chain drives, belts, pulleys, drums, revolving or reciprocating parts, exposed shafts and projecting shaft ends, collars, clutches, and couplings.

1.2.5 Safety Switches

Safety switches allow the tool to be turned "off" quickly. Hand-held power tools must be equipped with a positive on-off, a momentary on-off, or a constant pressure switch. A positive on-off is a standard on-off switch. Platen sanders, disc sanders, scroll saws, and grinders with less than 2-inch-diameter discs may have a standard on-off switch. A momentary on-off can be turned "off" by a single motion of the same finger or fingers that turn it on. Drills, reciprocating and saber saws, grinders, and belt sanders may have a momentary on-off switch. A constant pressure switch shuts off power upon release. Circular saws and chain saws may have a constant pressure switch. Always test switch to insure it is functioning properly.

1.2.6 Blind Operations

A "blind" operation is any circumstance using any type of saw, drill, or other cutting or penetrating tool where you can't see behind what is being cut. When making a blind cut or drilling operation, be sure that hidden electrical wiring, water pipes, or any mechanical hazards are not in the blade path. If wires are present, they must be disconnected at the power source by a qualified person or avoided. Contact with live wires could cause lethal shock or fire. Water pipes should be drained and capped. Always hold the tool by the insulated grasping surfaces.

1.2.7 Kickback

Kickback is a sudden, uncontrolled reaction to a pinched blade, causing the tool to lift up and out of the work piece toward the operator. Misuse, buildup of sap or dirt on the blade, insufficient *set*, dullness, and unguided cuts, can all cause kickback. Avoid kickback by keeping saw blades sharp, having proper amount of *set* in the teeth, keeping saw blades clean, and support large panels so they will not pinch the blade. Set blade depth to no more than 1/4 inch greater than the thickness of the material being cut. Release the switch immediately if the blade binds or the saw stalls.

1.2.8 Power Tool Accessories

The choice of a wrong accessory or incorrect use can result in serious injury. Read and understand the recommendations in the owner/operators manual for the tool and the accessory literature. Don't use an accessory or attachment unless: the power tool manufacturer recommends its use on their product; the accessory's limitations and specifications match the limitations and specification of the power tool; the use of the accessory does not require the removal of any guards; and you understand the instructions that describe the safe use of the accessory or attachment. Always unplug tools before installing, adjusting, and changing any accessory or attachment of any kind.

1.3 Types of Power-Operated Hand Tools

Power tools include electric, battery-powered, liquid fuel, hydraulic, pneumatic (air), and powder-actuated. Power tools operate at high speeds; when things go wrong, it happens fast.

1.3.1 Electric Power-Operated – Corded

Electric power-operated tools that use a cord will either be double-insulated type or have a three-wire cord plugged into a grounded receptacle, grounded according to Occupational Safety and Health Administration (OSHA) regulations. A ground fault circuit interrupter (GFCI) will be used between the power operated tool and the power source. Test the GFCI before each use and use a portable GFCI if necessary. Power tools should always be stored in a dry place when not in use. Never use a tool in wet/damp conditions unless designed to be used in such an environment. Never carry power tools by the cord or yank the cord to disconnect it. Always keep tools and cords away from heat, oil, and sharp edges. Always disconnect power tools when not in use and when changing accessories such as blades and bits.

1.3.2 Electric Power-Operated – Battery (Cordless)

Electric power-operated tools that run on batteries should be charged in a dry location and away from all combustible materials. Do no operate cordless tools in or near flammable liquids or explosive atmospheres. Motors in these tools may spark and ignite fumes. Always recharge a cordless tool and its battery with its own specified charging unit. Never attempt to recharge a cordless tool in a recharging unit not specifically recommended for that tool. Remove batteries or lock the switch in its “OFF” position before changing accessories, adjusting or cleaning tools. This removes the power supply while hands are in vulnerable

locations such as near switches, bits, or blades. Do not store the battery pack in a container with metal objects such as wire, nails, or coins as it could short the battery. Do not expose the battery pack to moisture, frost, or temperature extremes of over 110 degrees Fahrenheit or under -20 degrees Fahrenheit.

1.3.3 Liquid Fuel Power Tools

Liquid fuel power tools will be stopped, turned "off," and cooled while being refueled, serviced, or maintained. Fuel will be transported, handled, and stored in accordance with federal regulations. Safety Data Sheets (SDS) for fuel or chemicals will be accessible during use of the tools. The tool must be used in a well-ventilated area as the carbon monoxide generated can displace or deplete oxygen. Before refilling a fueled powered tool fuel tank, shut down the engine and allow it to cool as fuel fumes, combined with the heat from the tool, can cause an explosion. Use only Type 1 or Type 2 approved flammable liquid containers. Properly clean any spills from the refueling process.

1.3.4 Hydraulic Power Tools

The fluid used in hydraulic power tools will be fire-resistant and approved for use with the hydraulic powered tool as specified by the manufacturer. The purpose of the specialized fluid is to allow the tool to be safely used in extreme temperatures."

1.3.5 Pneumatic Power Tools

Pneumatic (air) power tools will be properly maintained and operated according to the manufacturer's safe operating procedures. Make sure air hose connections are secure. Use a short wire or positive locking coupler to attach the air hose to the tool. Check hoses regularly for cuts, bulges, and abrasions (tag and replace if defective). Ensure the safety clip for attachments is installed and secure. Ensure the muzzle is in contact with the surface. Never point the tool at anyone. Avoid using on easily penetrated materials unless they are backed by material that will prevent fastener from passing through. Don't drive fasteners into very hard or brittle material that could chip, splatter, or make the fasteners ricochet. Avoid using compressed air for cleaning.

1.3.6 Powder-Actuated Tools

Only employees who have been trained in the operation of the particular tool in use will be allowed to operate a power-actuated tool. Never use in an explosive or flammable atmosphere. Never load the tool unless it will be used immediately. Never leave a loaded tool unattended. Never point the tool at anyone. Always keep hands and feet clear of the barrel end. Always select a powder level that will do the work without excessive force. Avoid using on easily penetrated materials unless they are backed by material that will prevent fastener from passing through. Don't drive fasteners into very hard or brittle material that could chip, splatter, or make the fasteners ricochet.

1.4 Powered Hand Tools

1.4.1 Drills

Be sure the trigger switch actuates properly. If equipped with a lock-on, be sure it releases freely. Be sure the chuck is tightly secured to the spindle. Tighten the drill bit securely as prescribed by the manual. Check auxiliary handles to be sure they are securely installed. Never force a drill; apply only enough pressure to keep the drill bit cutting smoothly. If the drill binds in the work, release the trigger immediately. Unplug the drill from the power source and then remove the bit from the work piece. Never attempt to free a jammed bit by starting and stopping the drill. Review the manufacture's manual for how to unjam the equipment. Unplug the tool before changing bits, accessories, or attachments.

1.4.2 Saws

Circular Saws

Always use sharp blades. Dull blades can cause binding, stalling, and possible kickback. Check blades carefully before each use for proper alignment and possible defects. Be sure all cords are out of the blade path and are sufficiently long to freely complete the cut. Clamp materials whenever possible. Never hold a work piece in your hand when sawing.

Set blade depth to no more than 1/4 inch greater than the thickness of the material being cut. Always allow the blade to reach full speed before the work piece is contacted. Never overreach and never reach under the saw or work piece. Never use a circular saw for cutting logs or roots, trimming trees, or shrubs.

Reciprocating Saws

Always use sharp blades. Dull blades cause binding, stalling, and possible kickback. Only use the blade specifically recommended for the job being done. Be sure all cords are out of the blade path and are sufficiently long to freely complete the cut. Position yourself to maintain full control of the tool and avoid cutting above shoulder height. The work piece must be clamped securely and the shoe of the saw held firmly against the work. When making anything other than a through cut, allow the tool to come to a complete stop before removing the blade from the work piece. Remember that the blade and blade clamp may be hot immediately after cutting. Avoid contact until they have cooled.

Jig/Saber Saws

Check that the blades are secured in position before plugging in. Make sure the cord is not in the line of cut. Firmly position the tool's base plate/shoe on the work piece before turning on the tool. Keep your hands and fingers well clear of moving parts. After making partial cuts, turn "off" and remove the blade from the work piece only after the blade has fully stopped. Maintain firm contact between the base and the material being cut, throughout cutting procedures. Remember that the blade and blade clamp may be hot immediately after cutting. Keep your hands away until cooled down and never overreach.

1.4.3 Abrasive Wheels and Tools

Sanders

Sanding dust can be highly explosive if the concentration becomes too great. Ensure the work area has adequate ventilation. Always use of exhaust type systems or bag collection. Check the power supply to be sure the switch and switch lock are in the "off" position. Always use the appropriate size disk or belt. Use jigs or fixtures to hold your work piece whenever possible. When sanding, always be aware of the cord location.

Never force a sander – the weight of the tool applies adequate pressure. Do not expose the tool to liquids, or to use in wet locations. When adjusting the tracking of the belt, be certain to avoid accidental contact with yourself or other objects.

Grinders

Test grinding wheels before mounting by tapping the wheel lightly with a nonmetallic implement. If it produces a ringing sound, it is in good condition. If it sounds dull, replace the wheel, Never use a cracked wheel. Use only those wheels and discs marked with a rated speed at or above the speed rating on the nameplate of the tool. Never operate a grinder without the proper guards in place. Always allow the wheel to come up to full speed before you contact the work piece. Do not apply excessive pressure to the wheel or disc. Use grinding wheels when working with hard materials, and use rotary files for soft materials such as aluminum, brass, copper, and wood. Using grinding wheels on soft materials will excessively load the wheel and could cause the wheel to shatter or disintegrate.

Power

Grinding machines will be supplied with sufficient power to maintain the spindle speed at safe levels under all conditions of normal operations. Follow manufacturer recommendations for sufficient power supply.

Guarding

Grinding machines will be equipped with safety guards in conformance with the requirements of the American National Standards Institute (ANSI) B7.1-1970.

Routers

Always disconnect the plug from the electrical outlet before changing bits or making any adjustments. Install router bits securely. Make certain that the cutter shaft is engaged in the collet at least ½-inch. Always face the cutter blade opening away from your body. The switch should be in the “off” position before plugging into the power outlet. Always allow the motor to reach full speed before feeding the router into the work. Never attempt to remove debris while the router is operating. Secure clamping devices on the work piece before operating router. When removing a router from your work piece, always be very careful not to turn the base and bit toward you.

1.4.4 Woodworking Tools

Disconnect Switches

Fixed power driven woodworking tools will be provided with a disconnect switch that can either be locked or tagged in the "off" position.

Speeds

The operating speed will be etched or otherwise permanently marked on all circular saws over 20 inches in diameter or operating at over 10,000 peripheral feet per minute. Blades used on these types of saws must be rated to operate at or below the operating speed of the saw.

Self-feed

Automatic feeding devices will be installed on machines whenever the nature of the work will permit. Feeder attachments will have the feed rolls or other moving parts covered or guarded so as to protect the operator from hazardous points.

Guarding

Portable, power-driven circular saws will be equipped with guards above and below the base plate or shoe.

Personal Protective Equipment

Project-specific PPE will be identified in the HASP based on the hazards present during work tasks. Required PPE must be worn when operating power tools. More information regarding PPE is located in Section 6 of GEI's Health and Safety Program.

Other Requirements

Woodworking tools and machinery will meet other applicable requirements of ANSI 01.1-1961, Safety Code for Woodworking Machinery.

1.4.5 Jacks – Lever and Ratchet, Screw, and Hydraulic

General Requirements

The manufacturer's rated capacity will be legibly marked on all jacks and will not be exceeded. All jacks will have a positive stop to prevent over-travel.

Blocking

When the working area does not have a solid working surface and it is necessary to provide a firm foundation, the base of the jack will be blocked or cribbed.

Operation and Maintenance

Hydraulic jacks exposed to freezing temperatures will be supplied with adequate antifreeze liquid. Jacks will be properly lubricated at regular intervals. Jacks will be thoroughly inspected, before each use. Repair or replacement parts will be examined for possible defects. Tag worn, damaged or defective jacks "Out of Service" and do not use them; notify your

Branch Manager so that the jack can be replaced or repaired. Parts subjected to wear will be inspected on a regular basis and repaired or replaced as needed.

1.5 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Safety Officer.

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or Supervisor/Project Manager and submitted to the People & Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, Human Resources, and/or the receipt of the Incident Report Form, the Regional Health & Safety Officer (RHSO) will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health and Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.6 Limitations

Follow safety procedures as defined in the site-specific HASP. Appropriate PPE must be worn correctly to provide the intended level of protection. Read and understand the recommendations in the owner/operators manual for the tool, and the accessory literature.

1.7 References

OSHA Standards for the Construction Industry, Subpart I

Risk Analytics Power Tool Safety Training, 2006

1.8 Attachments

None

1.9 Contact

Health&SafetyTeam@geiconsultants.com

1.10 Review History

- July 2016
- May 2015 – Separated from SOP HS-008

STANDARD OPERATING PROCEDURES

SOP NO. HS-009 Hazardous Substances Exposure Management

1.1 Objective

This Standard Operating Procedure (SOP) is intended to outline the steps GEI employees will take to identify potential hazards associated with exposure to hazardous substances, the risks associated with these hazards, and the proper controls to use to minimize exposure. The site-specific health and safety plan (HASP) should include a hazard assessment for the project that identifies the potential of encountering a hazardous substance and the control methods to be implemented by GEI employees. These hazards should be reviewed in the project safety briefing and documented on the Project Safety Briefing form, found on the Safety page of the GEI intranet.

1.2 General

A hazardous substance is any substance that has one or more of the following intrinsic properties:

- Explosiveness
- Flammability
- Ability to oxidize
- Human toxicity (acute or chronic)
- Corrosiveness (to human tissue or metal)
- Ecotoxicity (with or without bioaccumulation)
- Capacity, on contact with air or water, to develop one or more of the above properties

1.3 Hazard Identification

An initial identification of hazards should be done based on a review of available documents including lists of chemicals used on site, analytical data from soil, surface water, groundwater, air, spill history, site history, equipment on site, maps, photos, and a preliminary survey.

Once hazardous substances are identified the regulated exposure limits need to be identified. Each substance may have a state/federal exposure value for each of the following (if applicable):

Action Level – An airborne level, typically one-half of the permissible exposure limit (PEL) designated in Occupational Safety and Health Administration's (OSHA's) substance-specific standards, 29 CFR 1910, Subpart Z, calculated as an

8-hour time weighted average, which initiates certain required activities such as exposure monitoring and medical surveillance.

Ceiling Limit – The exposure limit a worker's exposure may never exceed.

Sampling and Analytical Error – A statistical estimate of the uncertainty associated with a given exposure measurement.

Short-Term Exposure Limit (STEL) – The average exposure to a contaminant to which a worker may be exposed during a short time period (typically 15-30 minutes).

Time Weighted Average (TWA) – The average exposure to a contaminant over a given period of time, typically 8 hours.

1.4 Risk Identification

Once the presence and concentrations of specific hazardous substances and health hazards have been established, the risks associated with these substances will be identified. GEI employees and GEI subcontractors who will be working on the site will be informed of risks that have been identified.

Risks to consider include, but are not limited to:

- Potential exposures exceeding the permissible exposure limits and published exposure levels
- Potential Immediately Dangerous to Life and Health (IDLH) concentrations
- Potential skin absorption and irritation sources
- Potential eye irritation sources
- Potential hazardous atmospheres, including oxygen deficiency and fire and explosion hazards

1.5 Engineering Controls, Work Practices, and Personal Protective Equipment for Employee Protection

Engineering controls, work practices, and personnel protective equipment (PPE) for substances regulated in OSHA Subpart G (Occupational Health and Environmental Control) and Subpart Z (Toxic and Hazardous Substances) will be implemented in to protect employees from exposure to hazardous substances and safety and health hazards.

1.5.1 Elimination/Substitution

The first control method should be to try and eliminate or substitute the hazards with a safer alternative. This is the most effective solution as shown is Figure 1 below. If you can remove the hazard than you no longer need to find a way to protect the employee

from it. Or you can substitute a different piece of equipment or chemical to use that doesn't pose the same hazard and doesn't create a new one.

1.5.2 Engineering Controls

Engineering controls implement physical change to the workplace, which eliminates/reduces the hazard on the job/task. Examples include:

- Change the process to minimize contact with hazardous chemicals
- Isolate or enclose the process
- Use of wet methods to reduce generation of dusts or other particulates
- General dilution ventilation
- Use of fume hoods

1.5.3 Administrative Controls (Work Practices)

Administrative controls establish efficient processes or procedures to help protect the employee. Examples of these are:

- Rotate job assignments
- Adjust work schedules so that workers are not overexposed to a hazardous chemical

1.5.4 Personal Protective Equipment

The use of PPE to reduce exposure to risk factors is the last line of defense. All other options should be exhausted before use of PPE. Examples of PPE are:

- Chemical protective clothing
- Respiratory protection
- Gloves
- Eye or hearing protection
- Steel toe boots

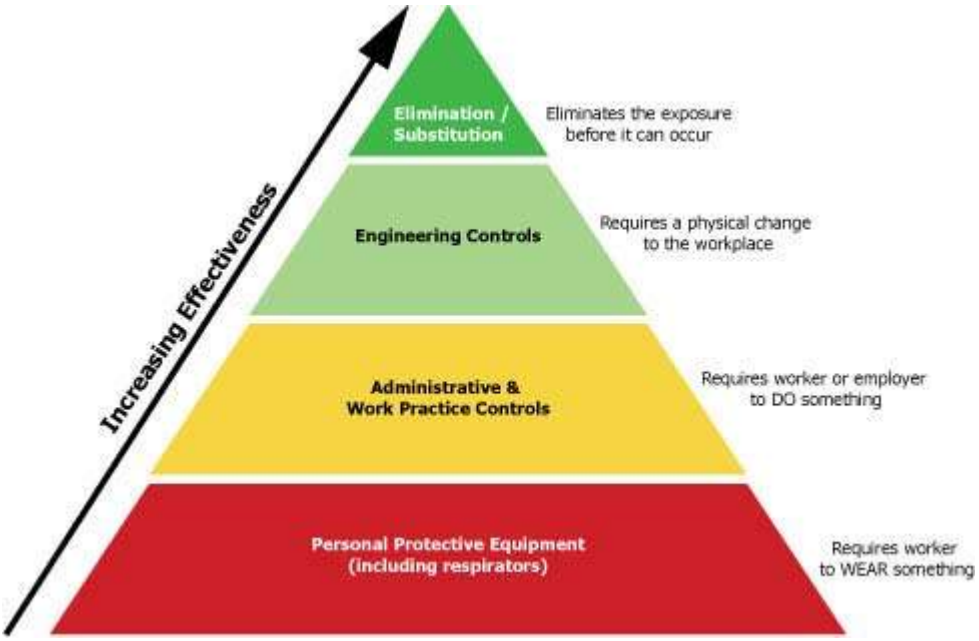


Figure 1: Hazard Mitigation Effectiveness Pyramid

1.5.5 Engineering Controls, Work Practices, and PPE for Substances Regulated in Subparts G and Subpart Z

Engineering controls and work practices will be instituted to reduce and maintain employee exposure at or below the PELs for substances regulated by 29 CFR Part 1910.

Engineering controls that may be feasible include the use of pressurized cabs or control booths on equipment, and/or the use of remotely operated material handling equipment. Work practices may include removing non-essential employees from potential exposure during opening of drums, wetting down dusty operations, and positioning employees upwind of potential hazards.

If engineering controls and work practices are not feasible, or not required, a reasonable combination of engineering controls, work practices, and PPE will be used to reduce and maintain at or below the PELs or dose limits for substances regulated by 29 CFR Part 1910, Subpart Z.

GEI will not implement a schedule of employee rotation as a means of compliance with PELs or dose limits except when there is no other feasible way of complying with the airborne or dermal dose limits for ionizing radiation.

The provisions of 29 CFR, subpart G, will be followed.

1.5.6 Engineering Controls, Work Practices, and Personal Protective Equipment for Substances Not Regulated in Subparts G and Subparts Z

An appropriate combination of engineering controls, work practices, and PPE will be used to reduce and maintain employee exposure to or below published exposure levels for hazardous substances and health hazards not regulated by 29 CFR Part 1910, Subparts G and Subparts Z. GEI will use published literature and Safety Data Sheets (SDS) as a guide in making the determination of what level of protection is appropriate for hazardous substances and health hazards for which there is no permissible exposure limit or published exposure limit.

1.5.7 Decontamination Procedures

Decontamination procedures will be developed, communicated to employees, and implemented before employees or equipment enter areas on site where potential for exposure to hazardous substances exists. Procedures will be developed to minimize employee contact with hazardous substances or with equipment that has contacted hazardous substances.

GEI employees leaving a contaminated area will be properly decontaminated; contaminated clothing and equipment leaving a contaminated area will be properly disposed of or decontaminated.

Decontamination procedures will be monitored by the site safety officer (SSO) to determine their effectiveness. When such procedures are found to be ineffective, the site safety officer will contact the Corporate Health and Safety Officer and appropriate steps will be taken to correct deficiencies.

Location
Decontamination will be performed in areas that will minimize the exposure to employees, equipment, and the environment.

Equipment and Solvents
Equipment and solvents used for decontamination will be decontaminated or disposed of properly.

Personal Protective Clothing and Equipment
Protective clothing and equipment will be decontaminated, cleaned, laundered, maintained, or replaced as needed to maintain their effectiveness.

Employees whose clothing comes in contact with hazardous substances will immediately remove that clothing and follow the directions on packaging or SDS sheet for how to properly clean the exposed area. The clothing will be disposed of or decontaminated before it is removed from the work zone.

Commercial Laundries or Cleaning Establishments
Commercial laundries or cleaning establishments that decontaminate protective clothing or equipment will be informed of the potentially harmful effects of exposures to hazardous substances.

Showers and Changing Rooms
Where the decontamination procedure indicates a need for regular showers and change rooms outside of a contaminated area, these will be provided and meet the requirements of 29 CFR 1910.141 (Sanitation). If temperature conditions prevent the effective use of water, then other effective means for cleansing will be provided and used.

1.6 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Health and Safety Officer.

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or Supervisor/Project Manager and submitted to the People & Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, Human Resources, and/or the receipt of the Incident Report Form, the Regional Health & Safety Officer (RHSO) will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health and Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.7 Limitations

None

1.8 References

OSHA 1910.120 Hazardous Waste Operations and Emergency Response
OSHA 1910 Subpart G Occupational Health and Environment Control
OSHA 1910 Subpart Z Toxic and Hazardous Substances
OSHA 1910.141 General Environmental Controls – Sanitation
http://www.business.govt.nz/worksafe/information-guidance/legal-framework/hsno-act-1996/defining-hazardous-substances/ (Viewed 7/8/2016)
https://www.osha.gov/SLTC/hazardoustoxicsubstances/ (Viewed 7/8/2016)
https://www.osha.gov/SLTC/hazardoustoxicsubstances/control.html (Viewed 7/11/2016)

1.9 Attachments

None

1.10 Contact

Health&SafetyTeam@geiconsultants.com

1.11 Review History

- July 2016
- May 2014
- November 2013
- August 2011 known as Hazard Identification and Management
- February 2011 known as HS-008 Contaminant Properties

STANDARD OPERATING PROCEDURES

SOP No. HS-010 Inclement Weather

1.1 Objective

This Standard Operating Procedure (SOP) is intended for use by employees engaged in work with the potential to be affected by inclement weather. The site-specific health and safety plan (HASP) should include a hazard assessment for the project that identifies the potential for working in inclement weather and the control methods to be implemented by GEI employees. These hazards should be reviewed in the project safety briefing and documented on the Project Safety Briefing form, found on the Safety page of the GEI intranet.

1.2 General

Employees should be aware of local weather conditions and monitor advisories issued by the National Weather Service and other local reporting services. Depending on location and season, storms are capable of producing heavy rain, floods, extreme temperatures, high wind conditions, lighting, tornados, and/or snowfall.

1.2.1 Heavy Rain

If working or driving in a rain storm, use extreme caution. When driving, turn your low beam lights on when the rainfall becomes heavy. Employees should be aware of the following:

- Heavy rain decreases visibility, especially when driving.
- Surfaces and tools become slippery.
- If you are working in the rain and your clothes become wet there is a risk of hypothermia when exposed to winds, even in warm temperatures.
- If the storms are going to produce thunder and/or lightning, leave the work area immediately and move to a safe area.
- Use your best judgment to determine if the rainfall becomes too heavy to continue working safely.

1.2.2 Lightning

Lightning can strike as far as 10 miles from the area where it is raining. That's approximately the distance you can hear thunder. **If you can hear thunder, you are within striking distance. Seek safe shelter immediately.** This can be within a building or vehicle. Wait 30 minutes after the last clap of thunder or flash of lightning before going outside again.

1.2.3 Flooding

Flooding may occur as a result of heavy rain in a short period of time. Flooding can be particularly acute in canyon areas where dry creek beds can turn into raging rivers from rainfall in distant or higher elevation areas. Be aware of this and your surroundings and move to a safe place if you begin to see signs that flooding may occur. Signs of potential flooding include sudden appearance of water in dry creek beds, increased water flow in rivers or streams, or quick rise in water levels.

Do not attempt to drive through areas or streets that are flooded. Seek alternate routes. Be particularly cautious at night when flooded areas are difficult to see. Urban flooding can stop traffic; increase the potential for traffic accidents; and can trap people in vehicles.

1.2.4 Extreme Temperatures

Work activities may take place in extreme heat or cold. Be prepared if these conditions are anticipated. Have the appropriate personal protective equipment (PPE) available; exercise proper fluid intake; and take breaks to prevent heat and cold stress. For more information about these conditions see the heat stress and cold stress programs found in GEI's Health and Safety Program.

1.2.5 High Winds, Tropical Storms, and Tornados

High Winds can be extremely dangerous. Appropriate measures will be taken to secure equipment and loose items when working in windy conditions. The project manager should be contacted about the weather conditions and, if necessary, work should be postponed.

Tropical storms are described as storms with sustained winds ranging from 39 to 73 miles per hour (mph) and hurricanes produce sustained winds that exceed 74 mph. When winds approach 40 mph (gale force winds) twigs begin to break off of trees and vehicles will veer off of the road. When winds approach 40 mph or the GEI employee feels unsafe based on the activities being performed, stop work and seek shelter as soon as possible. Blowing or falling debris and overhanging limbs/signs can be a significant hazard. If possible, avoid driving in these conditions; 70 percent of injuries during hurricanes are a result of vehicle accidents. Note that tall or elevated equipment will have manufacturer's safe operating wind speeds defined that could be less than 40 mph. The operator's manual should be consulted prior to operation of the equipment.

A tornado is a violent, dangerous, rotating column of air that is in contact with both the surface of the earth and a cumulonimbus cloud or, in rare cases, the base of a cumulus cloud. The Fujita Scale is used to rate the intensity of a tornado by examining the damage caused by the tornado after it has passed over a man-made structure. Based on the Fujita Scale, or F-Scale, numbers begin at F0: 40-72 mph and go to F6: 319-379 mph (F6 is

generally theoretical). Nearly three-fourths of tornados are on the weak F0-F1 scale with just over two-thirds of deaths resulting from the violent F4-F5 tornados.

If a tornado is seen, stop work and seek shelter immediately. If a tornado siren is sounded move immediately to safety indoors and then move to a windowless interior space, basement, stairwell, or designated fall-out shelter. Windows should not be opened before an oncoming tornado. If there is no shelter available, seat belt yourself into your stationary vehicle or seek a depression or low spot on the land surface.

1.2.6 Snowfall and Ice Conditions

Working in the winter months may result in activities taking place during periods of snowfall or icy conditions. If you are working during or after snow has fallen, dress appropriately for the conditions. Snow and ice can cause working surfaces to become slippery. Clear snow and ice from work areas to prevent slip hazards. Use caution when performing snow or ice removal activities to prevent injuries. Driving in snowy and icy conditions is also hazardous. Reduce speed and use caution if you must drive in these conditions.

If the weather conditions deteriorate and you do not feel safe working in these conditions, stop work, move to a safe indoor location, and contact your project manager to let them know the weather, work conditions, and your location.

1.3 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Safety Officer.

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or Supervisor/Project Manager and submitted to the People & Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, Human Resources, and/or the receipt of the Incident Report Form, the Regional Health & Safety Officer (RHSO) will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health and Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.4 Limitations

Follow safety procedures as defined in the site-specific HASP. Appropriate PPE must be worn correctly to provide the intended level of protection. Protection in extreme weather conditions can best be accomplished if the conditions are anticipated and actions are taken. Monitor local weather conditions prior to starting work.

1.5 References

Center for Disease Control and Prevention – Natural Disasters and Severe Weather
http://www.bt.cdc.gov/disasters/

National Lightning Safety Institute

NOAA, National Weather Service

Office of Climate, Water, and Weather Services

1.6 Attachment

None

1.7 Contact

Safety Team
Health&SafetyTeam@geiconsultants.com

1.8 Review History

- Previous revision dates were not documented
- May 2014
- July 2016

STANDARD OPERATING PROCEDURES

SOP No. HS-012 Noise Exposures

1.1 Objective

This Standard Operating Procedure (SOP) is intended for use by employees engaged in work with elevation noise levels. The site-specific health and safety plan (HASP) should include a hazard assessment for the project that identifies the potential for work in loud environments and the control methods to be implemented by GEI employees. These hazards should be reviewed in the project safety briefing and documented on the Project Safety Briefing form, found on the Safety page of the GEI intranet.

1.2 General

Working in loud environments can cause hearing damage and loss if the proper protection is not in place. The following procedures describe methods to mitigate unhealthy noise levels and protect hearing.

1.3 Hazard Identification

If projects involve noise levels above OSHA regulations, employees should take steps to remove the noise exposure. Common sources of elevated noise levels are heavy equipment, power tools, pumps, and generators. GEI has an established Hearing Conservation Program located in the GEI Health and Safety Program.

1.4 Risk Identification

Hearing protection is required if noise levels in a work area are known to be above 85 decibels (dB), which can be measured with a noise meter. When decibel levels are not known, hearing protection is required if you need to raise your voice to talk to someone standing within a normal speaking distance from you.

1.5 Mitigation

There are three options that can be used to help mitigate a noise hazard:

1.) Remove the hazard by taking away the source of the noise.

2.) Remove the employee from the source of the noise.

3.) Provide the employee with appropriate personal protective equipment (PPE).

The first option for employee protection is to remove the hazard by taking away the source of the noise or using engineering controls to reduce the level.

If this cannot be accomplished, the next control measure is to remove the employee from the source. This can be done by moving the work area to a quieter location or distancing the employee from the noise source. For example, GEI employees do not need to be standing next to an operating drill rig or other heavy equipment. By distancing themselves from heavy equipment or other noise sources the need for hearing protection can be eliminated/reduced.

The final option, if the above two options aren't feasible, disposable ear plugs that are made available to GEI employees are to be used. Additional means of hearing protection will be provided, such as ear muffs, if the disposable ear plugs are not adequate.

When using hearing protection, employees will need to make a greater effort to be aware of the surroundings which may include moving equipment, traffic, and other site hazards.

1.6 Proper Use of Hearing Protection

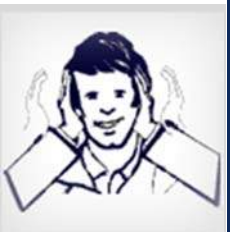
DISPOSABLE EAR PLUG FITTING INSTRUCTIONS

Before fitting any ear plugs, make sure your hands are clean.
Foam ear plugs are disposable and not intended for reuse.

Hold the ear plug between your thumb and forefinger. Roll and compress the entire ear plug to a small, crease-free cylinder. While still rolling, use your other hand to reach over your head and pull up and back on your outer ear. This straightens the ear canal, making way for a snug fit.

Insert the ear plug and hold for 20 to 30 seconds. This allows the ear plug to expand and fill your ear canal.

Test the fit. In a noisy environment, and with earplugs inserted, cup both hands over your ears and release. You should not notice a significant difference in the noise level. If the noise seems to lessen when your hands are cupped over your ears, your ear plugs are not fitted properly. Carefully remove the earplugs (see instructions below) and refit following instructions, above.



Always remove ear plugs slowly, twisting them to break the seal. If you remove them too quickly, you could damage your ear drum.	

REUSABLE EAR PLUG FITTING INSTRUCTIONS

Before fitting any ear plugs, make sure your hands are clean.

Reusable ear plugs should be inspected and cleaned often in soapy water. If they become hard, torn, or deformed they should be discarded and replaced.

Reach around your head and pull up and back on your outer ear. This straightens out the ear canal, making way for a snug fit. Hold the stem end of the ear plug and insert it well inside your ear canal until you feel it sealing and the fit is comfortable.	
Test the fit. In a noisy environment, and with ear plugs inserted, cup both hands over your ears and release. You should not notice a significant difference in the noise level. If the noise seems to lessen when your hands are cupped over your ears, your ear plugs are not fitted properly. Carefully remove the ear plugs (see instructions below) and refit following instructions, above.	
Always remove ear plugs slowly, twisting them to break the seal. If you remove them too quickly, you could damage your ear drum.	

1.7 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Safety Officer.

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or Supervisor/Project Manager and submitted to the People & Safety

Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, People Team, and/or the receipt of the Incident Report Form, the Regional Health & Safety Officer (RHSO) will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health and Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.8 Limitations

Follow safety procedures as defined in the site-specific HASP. Appropriate PPE must be worn correctly to provide the intended level of protection.

1.9 References

OHSA 29 CFR 1910.95 – Occupational Noise Exposure

OHSA 29 CFR 1926.101 – Hearing Protection

Texas American Safety Company (TASCO)

1.10 Attachments

None

1.11 Contact

Health&SafetyTeam@geiconsultants.com

1.12 Review History

- June 2016
- May 2014
- November 2013
- February 2011
- November 2010

STANDARD OPERATING PROCEDURE

SOP HS-014 Utility Mark-out

1.1 Objective

This Standard Operating Procedure (SOP) provides guidance for utility mark-out procedures related to drilling, excavation, or other sub-surface or intrusive activities to avoid injury to GEI employees or property damage. This SOP is applicable when GEI is responsible for its operation or our subcontractor's operation for utility mark-out. A utility mark out is when paint, flags or other markers are put in place to identify the location of an underground utility.

Clients or local agencies may have additional requirements or procedures to mark out of utilities. If local utility mark-out procedures differ from those described within this SOP, applicable state or municipal regulations should be followed.

1.2 General

This SOP is intended for use by employees engaged in work with sub-surface or intrusive activities. The site-specific health and safety plan (HASP) should include a hazard assessment for the project that identifies the potential for subsurface hazards and the control methods to be implemented by GEI employees. These hazards should be reviewed in the project safety briefing and documented on the Project Safety Briefing form, found on the Safety page of the GEI intranet.

1.2.1 Contractor/GEI Responsibilities

- The contractor or GEI employee will pinpoint each exploration area with white paint, flags, or stakes. personal protection equipment (PPE), including eye protection when using spray paint will be worn.
- Exploration locations should be marked-out with sample identification number(s) and type of sample (e.g., boring, test-pit, or monitoring well).
- The contractor compiles information about the work areas on a request form specified by the state utility mark-out program and submits it. Work area location maps can be sent to the utility mark-out program to clarify locations.
- The mark-out program customer service representative will provide a mark-out ticket number and a list of utilities notified upon receipt of the request information. This information will be recorded on the GEI documentation form in Appendix B and/or in other project documents.
- If known, the contractor or GEI employee will also notify non-member utility operators (e.g., apartment complexes, commercial complexes, railroads with communication cables, etc.).

1.2.2 Utility Mark Outs

- Utility companies or their sub-contractors will only mark-out, or clear, utilities under their responsibility. Generally, this means that they will only mark-out utilities within the public right-of-way up to private property boundaries. Information needed to determine the location of utilities on private properties will be requested from the property owner. This may include available property drawings or as-built figures. If this information is not available, additional non-intrusive surveys of the property may be required by a private utility locator to find underground utilities by using techniques such as ground penetrating radar (GPR).
- American Public Works Association (APWA) Uniform Color Code For Marking Underground Utility Lines are:
    1. **White** – Proposed Excavation
    2. **Pink** – Temporary Survey Markings
    3. **Red** – Electric Power Lines, Cables, Conduit and Lighting Cables
    4. **Yellow** – Gas, Oil, Steam, Petroleum, and Gaseous Material
    5. **Orange** – Communications, Alarm, Signal Lines, Cables or Conduit
    6. **Blue** – Water
    7. **Purple** – Radioactive Materials
    8. **Green** – Sanitary and Storm Sewers and Drain Lines

1.2.3 Utility Mark Out Review

- Before the intrusive work activities begin, the contractor or GEI employee will verify that each utility company has completed a utility location for the work area or the location has been cleared by a private locator and record this on the mark-out request information sheet.
- A visual survey of the project area will be done prior to the start of intrusive activities. This visual inspection will be done to identify signs, manholes, utility boxes, or other evidence of an underground utility is present and has been considered.
- The contractor or GEI employee can begin work on the scheduled work date and time if the utility operators have responded, taking care to find and preserve markings that have been made.
- Completed clearance documentation will be located on the excavation site during excavation activities and kept in project files.

1.2.4 Excavations

- When excavating near a buried utility, observe the approximate location around that utility.
- If exposing a utility, proper support and protection must be provided so that the utility will not be damaged.
- If the excavation work requires significant spans of the utility to be exposed, it is the contractor's responsibility to support the infrastructure (to prevent sagging or collapse) as needed. Contact the utility operator for support, guidance, or assistance.
- When the excavation is complete, provide proper backfill for utilities that have been exposed.
- Take care not to damage the conduit or protective coating of a utility. If the damage occurs, leave the damaged utility exposed and immediately call the utility owner.
- If a gas line is encountered, everyone will be evacuated according to the site evacuation procedures and the contractor must notify police, fire, and emergency personnel. No attempt should be made to tamper with or correct the damaged utility. All site personnel are to evacuate to the site's predetermined meeting point or a location a minimum of 300 feet away from the incident location.
- If the contractor needs to dig within the approximate location of a combustible, hazardous fluid, or gas line (natural gas, propane or gasoline), soft digging is required (hand digging, vacuum extraction) to a maximum depth of 5 feet. The approximate location is defined as 24 inches on either side of the designated center line of the utility if the diameter is not provided or 24 inches from each outside edge if the diameter is provided.

1.3 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Health & Safety Officer (RHSO).

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or Supervisor/Project Manager and submitted to the People & Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification and/or the receipt of the Incident Report Form, RHSO will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health and Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.4 Limitations

- Follow safety procedures as defined in the site-specific HASP. Appropriate PPE must be worn correctly to provide the intended level of protection.
- Mark-out notification time usually does not include holidays. Make sure holidays are considered and mark-out time is scheduled accordingly. Under no circumstances are intrusive activities allowed to be performed prior to the required mark-out.
- Do not use white paint if precipitation is eminent. Consider using stakes if snow is predicted.

1.5 References

Reference the website for the "Call Before You Dig – 811" for the utility mark-out agency for the state you working in prior to site work. If you have issues locating the appropriate agency, contact the Safety Team for assistance.

1.6 Attachments

Attachment A – Standard Utility Color Codes

Attachment B – GEI Utility Clearance Documentation Form

1.7 Contact

Health&SafetyTeam@geiconsultants.com

1.8 Review History

- June 2016
- May 2014
- November 2013
- February 2011
- November 2010

ATTACHMENT A

COLOR CODE FOR UTILITY MARKING

(BASED ON 'THE AMERICAN PUBLIC WORKS ASSOCIATION' RECOMMENDATIONS AND THE ANSI STANDARD Z-53.1 FOR SAFETY COLORS)

UTILITY	COLOR
PROPOSED EXCAVATION	WHITE
ELECTRIC POWER LINES, CABLES, CONDUIT AND LIGHTING CABLES	RED
POTABLE WATER	BLUE
STEAM, CONDENSATE, GAS OR OIL COMPRESSED AIR	YELLOW
TELECOMMUNICATIONS, ALARM OR SIGNAL LINES, CABLES OR CONDUIT	ORANGE
TEMPORARY SURVEY MARKINGS	PINK
SEWER AND STORM DRAINS	GREEN
CHILLED WATER, RECLAIMED WATER, IRRIGATION AND SLURRY LINES	PURPLE
OTHER	LIGHT BLUE

ATTACHMENT B

Utility Clearance Documentation

Please print clearly. *For more room, use back of page.*

Client: _____

GEI Project Name & Number: _____

Site: _____

Excavation/Drilling Location ID: _____

Excavator/Driller: _____

GEI PM: _____ GEI Field Team Leader: _____

Utility Drawings Reviewed: _____

Provided By: _____ Reviewed By: _____

Utility Clearance Call Date: _____ Name of Utility: _____

Utility Clearance Call Date: _____ Name of Utility: _____

Utility Clearance Received from (utility & rep name): _____ Date: _____

Utility Clearance Received from (utility & rep name): _____ Date: _____

Company that completed clearance: _____ Date: _____

GEI Staff Responsible for Oversight: _____

Metal Detector Survey (yes/no): _____ Drilling Location Cleared by: _____

Contractor Name: _____ Company Name: _____

Contractor Signature: _____ Date: _____

GEI Staff Responsible for Oversight: _____

Private Location Clearance Required (yes/no): _____ Date: _____

Contractor Name: _____ Company Name: _____

Contractor Signature: _____ Date: _____

Methods used for utility location (i.e. GPR, electronic pipe location) _____

GEI Staff Responsible for Oversight: _____

Hand clearing Performed (yes/no): _____ Methods: _____ Date: _____

Contractor Name: _____ Company Name: _____

Contractor Signature: _____ Date: _____

GEI Staff Responsible for Oversight: _____

GEI Consultants, Inc. Representative (name & title): _____

GEI Consultants, Inc. Representative Signature: _____ Date: _____

Based upon the best available information, appropriate utility clearance procedures were performed for the invasive work specified. If client ordered site specific deviations from existing GEI utility clearance procedures, they are approved by the client signature below:

Client Representative (name & title): _____

Client Representative Signature: _____ Date: _____

Notes:

STANDARD OPERATING PROCEDURES

SOP No. HS-016 Traffic Hazard Management

1.1 Objective

The objective of this Standard Operating Procedure (SOP) is to prevent or limit the potential for GEI personnel to encounter traffic hazards during field activities.

1.2 General

This SOP is intended for use by employees engaged in work with the potential for traffic hazards. The site-specific health and safety plan (HASP) will include a hazard assessment for the project that identifies the potential for exposure to traffic hazards and the control methods to be implemented by GEI employees, including review or attainment of necessary permits, traffic control plans, and flagger/police detail requirements for the local jurisdiction. Routine checks of the work zone will be made to ensure there are adequate levels of protection. These hazards will be reviewed in the project safety briefing and documented on the Project Safety Briefing form, found on the Safety page of the GEI intranet.

1.3 Traffic Hazard Management

Traffic Hazard Management is the process of identifying and managing the potential risks associated with the movement of traffic through, around, or past a work area. This Traffic Hazard Management SOP is designed to assist employees in identifying and managing these hazards. Work areas should be as safe as possible. It is the responsibility of GEI employees to follow the Traffic Hazard Management SOP and adhere to these safety standards. Safety is not negotiable.

Under no circumstances are GEI employees permitted to commence work in a situation that the employee believes or knows their health and safety, or the health and safety of others, is at risk.

Major risk factors for work site Traffic Hazard Management include:

- The speed of traffic moving through a work site.
- The distance and clearance between moving traffic, workers, vehicles and equipment, and over-head power lines.
- Traffic volume and vehicle composition.
- Nature and conditions at the work site and approaches to the work site.

- Other factors such as the time of day, sight distance, weather, presence of pedestrians, or cyclists, and the type of work being carried out.
- Other hazards in proximity to the work site (e.g., power lines, open excavations) that may have conflicting safety management measures that need to be considered when developing the HASP.

1.4 Site Preparation

The following management measures will be considered whenever working in traffic areas. In addition, remain aware of the amount of traffic around the working area. The work space should be large enough for the job to be completed safely. Check permit, traffic control plans, and flagger/police detail requirements for the local jurisdiction. Perform routine checks of the work zone to make sure there are adequate levels of protection.

1.4.1 Traffic Barriers and Warning Signs

GEI employees will comply with the U.S. Department of Transportation's (DOT) Manual on Uniformed Traffic Control Devices (MUTCD) and/or state regulations for temporary traffic barriers (cones, barriers) and sign placement when required for working in traffic areas. Clearly define the work site by placing traffic barriers around the work space to indicate the space that is needed to safely perform the work. The traffic barrier will help make the work site more visible to other workers, pedestrians, cyclists, and moving vehicles. Place traffic barriers in such a way as to give yourself and equipment adequate space to work within the barriers. OSHA suggests placing the first warning sign at a distance calculated to be 4 to 8 times (in feet) the speed limit (in MPH).

1.4.2 Adequate Light

Requirements for night conditions and work areas with poor visibility are similar to day requirements. However there are a number of additional things to consider, such as visibility of the work site to advancing traffic and sufficient lighting. OSHA requires lighting for workers on foot and equipment operators to be at least 5-foot-candles or greater.

Visibility of the work area can be increased by employing the following measures:

- Using parked vehicles hazard and flashing lights.
- Wearing reflective personal protective equipment (PPE), such as a safety vest, in good condition.
- Providing adequate lighting to illuminate the work area with lights positioned so that there is no glare to approaching drivers.
- Placing reflective advance warning signs and traffic barriers so that they are visible to road users.

1.4.3 Distance from the Nearest Traffic Lane

Work areas located along roadsides will have a minimum clearance as defined by DOT's MUTCD and/or state or local DOT regulations for traffic barrier and sign placement.

1.4.4 PPE

The proper PPE, as outlined in the project HASP, will be worn when appropriate. The color/type of safety vest will comply with site regulations.

1.5 Equipment Operation

Vehicles and heavy equipment operators should use a spotter when possible if it is necessary to drive in reverse to reduce risk of collision with oncoming traffic. If it is necessary to drive against the flow of traffic make sure this area is within the work zone and properly blocked off from oncoming traffic.

1.6 Pedestrian Safety

When working near pedestrian traffic, a safe alternate pedestrian route will be established. Refer to local regulations when establishing pedestrian walkways.

1.7 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Health & Safety Officer (RHSO).

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or Supervisor/Project Manager and submitted to the People & Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, Human Resources, and/or the receipt of the Incident Report Form, the RHSO will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health and Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.8 Limitations

Follow safety procedures as defined in the site-specific HASP, federal DOT, and local jurisdictions. Appropriate PPE must be worn correctly to provide the intended level of protection.

1.9 References

DOT's Manual on Uniformed Traffic Control Devices (2009 Edition)

Hazard Exposure and Risk Assessment Matrix for Hurricane Response and Recovery Work: https://www.osha.gov/SLTC/etools/hurricane/work-zone.html

1.10 Attachments

None

1.11 Contact

Health&SafetyTeam@geiconsultants.com

1.12 Review History

- November 2016
- May 2014
- November 2013
- August 2011
- October 2010 Initially HS-027 Traffic Hazards

STANDARD OPERATING PROCEDURES
HS-017 Water Safety

1.1 Objective

The objective of this Standard Operating Procedure (SOP) provides requirements and best practices for GEI personnel during field activities taking place on board a boat/barge or when working in or near water. If conducting aquatic surveys or electrofishing activities, please *refer to* **HS-022 Aquatic Ecological Surveys and Electrofishing Safety** for additional safety information for working in or near water.

1.2 General

This SOP is intended for use by employees where there is the potential to perform work while on board a boat/barge or when working in or near water. The site-specific health and safety plan (HASP) will include a hazard assessment for the project that identifies the potential hazards and the control methods to be implemented by GEI employees. The site-specific HASP, project hazards, and control methods will be reviewed during the project safety briefing and documented on the Project Safety Briefing form found on the Safety page of the GEI intranet.

1.3 Working near Water

OSHA Construction Industry Standards (29 CFR 1926.106) state, "…employees working over or near water, **where the danger of drowning exists**, will be provided a U.S. Coast Guard-approved life jacket or buoyant work vests." (*Emphasis added, not in original text*) OSHA General Industry Standards (1910) do not address working over or near water; therefore, GEI uses the OSHA Construction Standard for any work near water.

The following safety devices, when appropriate, will be used by employees working near water:

- **Life Jacket or Personal Flotation Devices (PFD).** Employees will be required to wear U.S. Coast Guard-approved personal flotation devices that are marked or labeled Type III PFD or a Type V PFD that is marked or labeled for use as a work vest for commercial use or for use on vessels. GEI employees will inspect personal floatation devices for defects that could alter their strength or buoyancy prior to, and after, each use. Tag worn, damaged, or defective PFDs "Out of Service" and do not use them; notify the Branch Manager so that the PFD can be disposed of and replaced. GEI does not issue or permit the use of unsafe PFDs.
- **Ring Buoys.** U.S. Coast Guard-approved ring buoys with at least 90 feet of line will be readily available for emergency rescue operations. Distance between ring buoys will not exceed 200 feet.

- **Life-saving Skiff.** When GEI employees are working over or adjacent to water, at least one lifesaving skiff will be immediately available for potential rescue purposes.

These requirements can be superseded by the use of 100 percent fall protection. If an employee cannot fall into the water as a result of the use of active or passive fall protection, there is no danger of drowning and the bullet points above are not required. The type of fall protection or the need for the above mentioned safety measures will be described in the site specific HASP. If 100 percent fall protection is in place, the boat will, at a minimum, have U.S. Coast Guard-approved PFDs on board for each person and at least one throw-able flotation device, such as a ring buoy.

GEI employees will not work alone when working near water.

1.4 Boat/Barge Safety Practices

Boat safety practices will be conducted in general accordance with guidance provided in the U.S. Army Corps of Engineers (USACE) Safety and Health Requirements Manual (EM) 385-1-1 and the U.S. Coast Guard. The following safety practices will be adhered to:

- If there is an accident the U.S. Coast Guard must be notified. The attached U.S. Coast Guard accident report form can be used to document the accident. Federal law requires the operator, or owner, if the operator is deceased or unable to make the report, to file a boating accident report with the state reporting authority when, as a result of an occurrence that involves a boat or its equipment:
    - Incident involving a fatality.
    - A person disappears from the vessel under circumstances that indicate death or injury.
    - A person is injured and requires medical treatment beyond first aid.
    - Damage to vessels and other property totals $2,000 (lower amounts in some states and territories) or more.
    - The boat is destroyed.
- Boats operated by GEI will comply with U.S. Coast Guard regulations.
- Any GEI employee that is operating a boat is required to take a Boating Safety Course. The course will be in accordance with the requirements of the state in which the boat will be operated. Documentation of course completion will be sent to the People & Safety Team.
- The boat operator will be familiar with United States Geological Survey navigation rules.

- Any boat operator, whether GEI or a subcontractor, is not to exceed 12 hours of duty time in a 24-hour period.
- GEI employees will wear a PFD as described in Section 1.3 when on board a boat /barge.
- For a boating activity where the vessel launches and is no longer in view of on-shore personnel, a Float Plan will be completed and left with a reliable person who can be depended upon to notify the U.S. Coast Guard, or other rescue organizations, should you not return or check-in as planned. If there is a change of plans after leaving, be sure to notify the person holding the Float Plan. Please *see* the attached U.S. Coast Guard Float Plan form.
- For a task requiring more than 1 day, daily communications with the reliable person will be maintained.
- The consumption of alcoholic beverages and the use of illegal drugs will not be permitted aboard boats/barges on which GEI employees are conducting company business, this includes boats/barges that may belong to someone else (subcontractor, client, etc.).
- Employees working on a boat will monitor the weather, incorporating, as appropriate, National Oceanic and Atmospheric Administration (NOAA) marine weather broadcasts and other local commercial weather forecasting services as may be available.
- For retrieving a person overboard, the boat operator, or other competent person will throw a life ring, that is attached to the boat with at least 90 feet of rope, and use a ladder attached to the boat or the boat step transom to allow the person to climb out of the water.
- Emergency procedures for fire, person overboard, and capsizing will be reviewed on the first day of operations and when there is a change of personnel occurs.
- Check with your local and state agency to verify their boat law/regulations.
- Beware of carbon monoxide poisoning when the boat engine is running in closed or poorly ventilated areas.

1.4.1 Boating Safety Equipment

The U.S. Coast Guard requires particular safety equipment to be aboard a boat. This list includes those items as well as other equipment that GEI requires as a minimum. Other safety equipment may be required based on the type and size of the boat being operated. The boat will be equipped with:

- At least one mounted ABC-1 type U.S. Coast Guard-approved portable fire extinguisher.
- A Type IV life ring attached to at least 90 feet line. Distance between ring buoys will not exceed 200 feet.
- A Coast Guard approved PFD for each person on board.
- A visual distress signal, such as flare.
- VHF-FM Marine Radio with Digital Selective Calling System.
- An efficient sound-producing device, such as a whistle or horn.
- One U.S. Coast Guard approved backfire flame arrestor device on each carburetor of all gasoline engines installed after April 25, 1940, except outboard motors.
    - Devices will be marked to show compliance with SAE J-1928 or UL 1111 Standards.
- Ladder attached to the boat or the boat step transom.
- An adequately-sized first aid kit.
- Charts and navigation aids.

1.5 Training

1.5.1 General

Training will be conducted for employees unfamiliar with the use of safety equipment and personal protective equipment (PPE) required by this SOP. Employees working over or near water will be trained in their responsibilities and the safe work practices associated with working on or near water.

1.5.2 Operating a Boat

A Boating Safety Course is required for employees that will be operating a boat. The course will in accordance with the requirements of state in which the boat will be operated. There is no regulated refresher date; therefore, GEI will require the course be retaken every 5 years to demonstrate continued proficiency. Documentation of course completion will be sent to the Safety Team.

1.6 Wading In Water

GEI employees may be required to wade in water during the performance of project work including but not limited to aquatic surveys and other ecological activities. Employees will be familiar with safe wading procedures and emergency preparedness as described below. Working in and around water can be an inherently hazardous activity (hypothermia, drowning risk) for which safety will be the primary concern.

1.6.1 General Safety

Field sampling operations in and around water will not be carried out alone. A minimum team size is two people. Employees will wear appropriate clothing and PPE in accordance with Section 1.6.2, Personal Protective Equipment Requirements for Wading, as defined below and as discussed in HS-022 Aquatic Ecological Survey and Electrofishing Safety.

Instream crews will have a First Aid kit onsite during wading operations and will work in a manner that is safe for themselves and their co-workers. The crew leader is the recognized authority and operational decision-maker. Crew members have the duty to comply with all of the crew leader's instructions concerning their health and safety and to report to the crew leader anything that is likely to be hazardous to their health or safety or to that of other employees or persons granted access to the work area.

Employees undertaking instream sampling or wading operations will be familiar with the hazards associated with those activities and agree to use common sense and safe wading practices during sampling. Field staff will have up-to-date Cardio-Pulmonary Resuscitation (CPR) and First Aid training.

1.6.2 Personal Protective Equipment Requirements for Wading

The following PPE is required when wading in water and will be provided by GEI:

- Leak-free chest waders or hip waders (neoprene, heavy weight polyurethane, PVC, or high-quality breathables), dependent upon depth. Hip waders can be worn when water depth is known not to be over "knee-deep" on any employee wearing hip waders.
- Non-slip footwear (felt wading shoe or boot, as well as stream wading cleat).
- Wading belt (nylon or rubber acceptable, worn on outside of chest waders).
- Polarized sunglasses (side shields or 'wrap around' style is/are recommended) and a hat with a visor is recommended (e.g., baseball cap style).
- PFDs will be worn where the crew leader considers the water is of sufficient depth or velocity for a PFD to be effective as protection from risk of drowning. PFDs will be U. S. Coast Guard approved personal flotation devices that are marked or labeled Type III or V PFD. Wading should not be carried out where water depth is greater than waist deep.
- In some situations, a wading staff might be useful as a "third leg" to help stabilize personnel during stream crossing or other wading operations.

1.6.3 Safety Procedures for Wading Operations

Office Preparation

- The crew leader will verify all employees have received instruction in the fundamentals of wading safety and working around water.
- It is the crew leader's responsibility to verify that employees are properly outfitted for the intended work.
- Where available, employees should attempt to identify any information on known instream hazards at the intended field site, including current discharge stage.
- Each instream sampling team will establish a means of emergency communication such as using walking talkies or specific hand signals.

Field Preparation

- A crew leader will be designated for activities in water.
- Crew members will be equipped with the personal equipment detailed in Section 1.6.2, Personal Protective Equipment Requirements for Wading.
- Each instream work site will be visually inspected for hazards such as deep holes, submerged logs, animals, etc. before commencing wading operations.
- In order to aid in identifying underwater hazards, all crew members will be equipped with polarized sunglasses. Glasses also protect against eye injury caused by sticks and branches. Wide brimmed hats or 'baseball' style caps are also beneficial in increasing the effectiveness of polarized glasses.
- Using visible landmarks, personnel will assess flow stage for the waterbody and continue to monitor for changes in water level during sampling operations. Changes in water level could indicate the onset of a flash flood.
- Clear command signals will be established between crew members prior to beginning operation. Crew members will inform the crew leader of any dangerous situations. Do not go into water that is above the level you have experienced before and are physically capable of handling.

1.6.4 Wading Procedures

- Operate slowly and carefully. Footing in most streams is poor and most falls occur when crew members are hurrying. Operations should cease when fatigue sets in.
- Plan a route that minimizes your exposure to the strongest currents/deepest water. Sand and gravel bottoms are usually secure and safe bottoms to wade on. Avoid standing on large, smooth rocks as much as possible.

- Plan on generally working in a downstream to upstream direction, ensuring solid footing before shifting positions
- Be aware of floating debris and changing water levels.
- In shallow water, less than knee deep, it may be possible to walk "normally" with a modified, wide stance. As water gets deeper and footing becomes obscured by water depth or turbidity, sidestepping will help maintain a wide, stable base.
- In moderate currents, position your feet so that you are facing upstream with your body side-on to the current to brace yourself and reduce your profile in the current.
- When crossing streams, it is easier and safer to move at a slight downstream angle with the current than to move directly across or upstream against the current.
- In difficult situations, take small shuffling steps, angling your body slightly upstream. If using a wading staff, position it upstream, maintaining two points of contact with the bottom at all times.
- In particularly difficult situations, use a buddy system in which you and a partner cross the stream together arm in arm, one upstream of the other so that you are supporting each other
- If you do happen to lose your footing and begin to float downstream, don't try to swim. Bring your knees up and roll over on your back with your feet downstream, working your way to the sides or to a suitable place to get out.

1.7 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Safety Officer.

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or Supervisor/Project Manager and submitted to the People & Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, Human Resources, and/or the receipt of the Incident Report Form, the Regional Health & Safety Officer (RHSO) will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health and Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.8 Limitations

Follow safety procedures as defined in the site-specific HASP. Appropriate PPE will be worn correctly to provide the intended level of protection.

1.9 References

Occupational Safety and Health Administration (OSHA) 29 CFR 1926.106 – Subpart E: Personal Protective and Life Saving Equipment: Working Over or Near Water.

U.S. Code of Federal Regulations (CFR) Title 33 – Navigation and Navigable Waters; Chapter I – United States Coast Guard.

United States Army Corps of Engineers, Safety and Health Requirements Manual (EM), 385-1-1. November 3, 2003 – Section 19 Floating Plant and Marine Activities

United States Army Corps of Engineers, Safety and Health Requirements Manual (EM), 385-1-1 September 15, 2008 – Section 01.C Physical Qualifications of Employees

1.10 Attachments

U.S. Coast Guard Float Plan Form

U.S. Coast Guard Navigation Rules

U.S. Coast Guard Accident Report Form

1.11 Contact

Health&SafetyTeam@geiconsultants.com

1.12 Review History

- April 2025
- November 2015
- December 2014
- November 2013
- August 2011
- June 2008 –previously HS-024

STANDARD OPERATING PROCEDURES

SOP No. HS-018 Working Around Heavy Equipment

1.1 Objective

The objective of this Standard Operating Procedure (SOP) is to prevent or limit the physical hazards when working around heavy equipment.

1.2 General

This SOP is intended for use by employees engaged in work with the potential for working near heavy equipment. The project site-specific health and safety plan (HASP) should include a hazard assessment for working near heavy equipment to be implemented by GEI employees. These hazards should be reviewed in the project safety briefing and documented on the Project Safety Briefing form, found on the Safety page of the GEI intranet.

1.3 Heavy Equipment Precautions

Heavy equipment (e.g., excavators, backhoes, drill rigs, etc.), can present many physical hazards that can result in serious injury or death if the proper safety precautions are not followed. The following is a list of precautions to be aware of when working around heavy equipment:

- Wear appropriate personal protective equipment (PPE), including at a minimum reflective, high-visibility safety vest, hard hat, safety glasses, and steel/composite toe boots.
- Always keep your distance from moving equipment.
- Do not assume the operator knows where you are or where you are going.
- Make sure to make eye contact and receive acknowledgement of your presence with the operator.
- Avoid working near heavy equipment, but if unavoidable, communicate your location with the operators. If using hand signals, discuss the signals with the equipment operator prior to starting work.
- Watch for moving equipment. Construction sites can have a lot of activity and equipment may be moving in an unpredictable manner.
- Do not rely on back-up or other alarms. They may not be working or you may not hear them with the noise of other activities taking place in the area.
- Stay out of the swing radius of cranes, excavators, or other equipment that swings or rotates.
- Do not walk beside a moving vehicle, the vehicle may turn, slip, or the load may shift causing the vehicle to go off course.
- Do not ride on the outside of a moving equipment.

- Never walk under or stand too close to a load suspended by cranes or hoists.
- Do not walk behind a piece of equipment that is backing up without acknowledgment from the operator it is safe to proceed. If working next to heavy equipment is unavoidable, be aware of the hazards including pinch points and moving parts. Use a spotter to watch the work area for moving equipment.
- If necessary, ask the operator to stop equipment operation to perform your work tasks.
- Verify the location and operation of emergency shut-off devices on the equipment.
- Be aware of the fuels and chemicals associated with the equipment. Have a spill prevention and response plan in place that includes the appropriate containment materials (i.e., spill kit).
- Do not wear loose fitting clothing when working around moving equipment (i.e., drill rig augers).
- Do not operate heavy equipment.
- Do not use cellular telephones near operating equipment.

1.4 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Safety Officer.

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or Supervisor/Project Manager and submitted to the People & Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, Human Resources, and/or the receipt of the Incident Report Form, the Regional Health & Safety Officer (RHSO) will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health and Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.5 Limitations

Follow safety procedures as defined in the site-specific HASP. Appropriate PPE must be worn correctly to provide the intended level of protection.

1.6 References

OSHA 29 CFR 1926.600 – Subpart O; Motor Vehicles, Mechanized Equipment, and Marine Operations.

www.toolboxtopics.com/Construction/ (Viewed 10/16)

Caterpillar Safety – http://safety.cat.com/ (Viewed 10/16)

1.7 Attachments

None

1.8 Contact

Health&SafetyTeam@geiconsultants.com

1.9 Review History

- October 2016
- May 2014
- November 2013
- August 2011
- October 2010

STANDARD OPERATING PROCEDURES
SOP NO. HS-026 Hazard Identification and Management

1.1 Objective
This Standard Operating Procedure (SOP) is intended to outline the steps GEI employees will take to identify potential hazards on site, the risks associated with these hazards, and the proper engineering controls, work practices, and personal protective equipment (PPE) to use to minimize the associated risks.

1.2 Hazard Identification
Establishing proper work procedures by conducting a job hazard analysis will should be performed for all projects involving field work. An initial identification of hazards will be completed based on past and current property usage of the site, what tasks are required to perform the job, what equipment is needed to complete the assigned tasks, what hazards are in the working area etc.

The site-specific health and safety plan (HASP) will include a hazard assessment for the project that identifies the potential hazards and how to alleviate the hazard. These hazards will be reviewed in the project safety briefing and documented on the Project Safety Briefing form, found on the Safety page of the GEI intranet.

1.3 Risk Assessment
A risk assessment will be performed for all aspects of field work. This analysis is to determine the quantitative or qualitative value of risk related to a tangible situation and a recognized hazard. Identification, studies, and monitoring of any hazard to determine its potential, origin, characteristics, and behavior are examples of what could be included and performed during a risk assessment. The assessment will increase awareness of workplace hazards and provide an opportunity to identify and control workplace hazards.

1.3.1 Assessment Guidelines
It is necessary to consider certain general guidelines for assessing the foot, head, eye and face, and hand hazard situations that exist in an occupational operation or process, and to match the protective devices to the particular hazard.

Assessments should be conducted:

- Prior to starting any work at the site
- As conditions change
- Workplace layout changes
- Environmental changes
- Process changes

- Yearly or other pre-determined interval

1.3.2 Hazard Sources

Some examples of hazard sources include but are not limited to:

- Items, materials, or machinery in motion
- Extreme temperatures
- Chemical exposures
- Harmful dust
- Light radiation
- Falling objects or potential from dropping objects
- Sharp objects
- Rolling or pinching objects
- Layout of workplace and location of co-workers
- Electrical hazards
- Noise exposures
- Confined spaces
- Working near or on water
- Fall hazards
- Traffic or other activities taking place on the site
- Air quality issues

1.4 Prevention – Control Methods

Control methods should be considered in the following hierarchy:

- Elimination
- Substitution
- Engineering Controls
- Administrative Controls
- Personal Protective Equipment

1.4.1 Elimination and Substitution

Elimination and substitution, while most effective at reducing hazards, also tend to be the most difficult to implement in an existing process. If the process is still at the design or development stage, elimination and substitution of hazards may be inexpensive and

simple to implement. For an existing process, major changes in equipment and procedures may be required to eliminate or substitute for a hazard. Employees should work with the Safety Team to find solutions.

1.4.2 Engineering Controls

Engineering controls are used to remove a hazard or place a barrier between the work and the hazard. It's implemented to control the hazard at the source. Examples may include machine guards, sound deading/dampening panels, traffic barriers, guardrails, and shields.

1.4.3 Administrative Controls

Administrative controls change the work procedures such as programs, schedules, and supervision to reduce employee exposure to hazards. The controls are frequently used with existing processes where hazards are not particularly well controlled. Examples of administrative controls are requiring frequent breaks or implementing a specific method to perform a task.

1.4.4 Personal Protective Equipment Selection

To select the proper PPE, the potential hazards must be known. The protective equipment selected must ensure a level of protection *greater than* the minimum required in order to help protect employees. The user must be supplied with a properly fitting protective device and given instructions on care and use. Users must be aware of all warning labels for and limitation of the PPE. Employees must be aware that the PPE does not eliminate the hazard.

1.4.5 Hazard Re-Assessment

As necessary, the workplace should be re-assessed for hazards by identifying and evaluating new equipment and processes, reviewing accident records, and re-evaluating the suitability of previously selected PPE. Re-assessment should occur at a defined regular schedule interval.

1.5 Job Safety Analysis

A job safety analysis (JSA) sometimes referred to as a job hazard analysis (JHA) or an activity hazard analysis (AHA) is the breaking down of any method or procedure into its component parts to determine the hazards connected with each key step and the requirements for performing it safely.

When a JSA is being created, make sure it isn't too general where the resulting information is not enough to assess the hazard and select proper controls, and be careful not to add unnecessary steps.

1.6 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Health & Safety Officer (RHSO).

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or Supervisor/Project Manager and submitted to the People & Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, Human Resources, and/or the receipt of the Incident Report Form, the RHS) will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health and Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.7 Limitations

Limitations may arise on a project specific basis and will be addressed as they arise.

1.8 Attachments

None.

1.9 References

Risk Analytics, LLC Hazard Assessment Training Program, January 2011

1.10 Contact

Health&SafetyTeam@geiconsultants.com

1.11 Review History

- November 2016
- June 2015

STANDARD OPERATING PROCEDURES

SOP No. HS-028 Trailer Use

1.1 Objective

This Standard Operating Procedure (SOP) provides requirements and recommendations to minimize the potential risks of towing a trailer behind a vehicle.

1.2 General Trailer Operation

Licensing and requirements for towing a trailer vary from state to state. Know and adhere to the specific requirements for your state and for the states in which you may travel with a trailer in tow.

1.2.1 Personal Protective Equipment

Employees will be provided with the personal protective equipment (PPE) necessary to help protect them from the hazards associated with towing a trailer. All employees will wear work gloves and steel toe or composite toe boots, at a minimum. Most hand injures can be avoided with the proper use of PPE. PPE must be maintained in good condition, kept clean and properly stored when not in use. More information regarding PPE is located in Section 6 of GEI's Corporate Health and Safety Program.

1.2.2 Trailer Hook-up and Detachment

Proper attachment of the trailer to the towing vehicle is essential to safety transport equipment and materials and to avoid vehicle and trailer damage, citations/fines, or traffic accidents. Potential risks may include overloading, separation of the trailer from the vehicle, and improperly working trailer brakes or lights.

Consult the tow vehicle owner's manual to confirm that the vehicle is capable of towing a trailer, and note any additional requirements or necessary equipment. Check the tow vehicle's tire pressure, which may need to be increased for hauling heavy loads. If you are towing a trailer that is wider than your tow vehicle, extended side-view mirrors will be needed to see rear and side approaching traffic. Know and understand the weight capacities of the tow vehicle and trailer as well as the weight of your equipment and cargo. If weight of the unloaded trailer is in question, go to a truck scale to confirm the weight.

> Gross Vehicle Weight Rating (GVWR) = amount the tow vehicle may weigh when fully loaded
>
> Gross Combined Weight Rating (GCWR) = amount the tow vehicle actually weighs when fully loaded

Follow these steps when attaching a trailer to the tow vehicle:

- Check tow vehicle's GVWR and trailer's GVWR to find maximum load limit for the vehicle/trailer combination.
- Confirm that the ball size on the tow vehicle matches the trailer tongue receiver.
- Back up to trailer with the assistance of a spotter (if available).
- Always wear appropriate PPE, at a minimum this includes work gloves, safety glasses, and steel or composite toe boots.
- Lower trailer hitch receiver onto ball until jack stand is off the ground, then properly stow jack stand. Make sure fingers and hands are not between the hitch and ball when lowering.
- Lock the latch and insert pin to secure (if possible, lift trailer tongue to check).
- Attach the trailer's safety chains to the tow vehicle, crisscrossing in an "X" shape.
- Plug trailer wiring harness into truck.

After completing attachment, walk around the trailer to verify all connections are secured, hitch is latched, jack stand is stowed, and tire blocks (chocks) are removed.

Prior to departure, these items must be tested and confirmed to be in working order on both the trailer and the tow vehicle:

- Running lights.
- Turn signals.
- Brake lights.
- Tire pressure, per vehicle owner's manual, specific to towing.
- Adjust side view mirrors to allow sight of trailer sides and tires.
- Check that the lug nuts/bolts on the tow vehicle and trailer are tightened to the correct torque, per vehicle owner's manual.
- If the trailer is equipped with brakes, check them by rolling forward and slightly applying the brake pedal. The trailer brakes should engage and you may feel a slight tug.
- Most trucks are equipped with a "Tow Haul" option. Engage the "Tow Haul" option and adjust the braking tension as need. See the vehicle owner's manual for how to do this. Make sure this option is used when hauling the trailer.

1.2.3 Loading Equipment onto Trailer

Confirm that the trailer tongue is securely attached to the ball of the vehicle. Park on level ground and engage the tow vehicle's emergency brake. Have another employee help with the loading/unloading of equipment onto the trailer. *Refer to* SOP No. HS-025 Manual Lifting to ensure safe lifting practices are followed. Lower the loading ramps or place detached loading ramps in designated rail and attach safety straps, if equipped. Adjust

ramps as necessary to accommodate the width of the tires on the equipment being loading if applicable. Align the equipment's tires with the load ramps. If the equipment has an implement attached, lift it to the highest position to prevent dragging on the ground. If the equipment is equipped with a front-end loader, lift the bucket to sufficient height to avoid hitting the trailer or ramps, but do not lift bucket all the way up. Using the lowest gear available, slowly drive equipment onto the trailer, paying close attention to where the tires are tracking on the ramps. Once the equipment is in position, if applicable place it in "park", apply the parking brake, and turn the engine off. Replace the ramps in the transport position and secure with pins.

Cargo Weight Distribution

Position a tractor or other heavy machinery so that it is centered over the trailer's axle(s) or slightly ahead of the axle(s) to provide adequate tongue weight. Too much weight toward the rear of the trailer can result in fishtailing and loss of control while in tow. Distribute cargo weight evenly along the length of the trailer and balance weight from side to side. Refer to the trailers owner's manual for proper load distribution.

Securing Cargo

- Inspect all chains, binders, and ratchet straps for knots, wear, and/or cuts or other damage. Never use compromised materials to secure equipment on the trailer. Damaged equipment must be tagged as "damaged" and reported to the Branch Manager for replacement.
- All cargo shall be secured such that it cannot shift, tip, or roll during transit. Chock the front and rear of the tires of the equipment to prevent rolling.
- Secure tractors or other heavy machinery to trailer with a minimum of two chains, one pulling tractor forward and one pulling backward. The collective working load limit (WLL) for all chains must equal at least half the weight of the tractor/machine.
- Secure implement using at least one chain or strap with the appropriate working load limit.
- All straps, chains, and other tie-downs must be attached and secured in a manner that prevents them from becoming loose, unfastening, opening, or releasing while the vehicle is in transit. Do not place tie-downs where they may be cut or torn by sharp edges on the cargo or trailer.

1.2.4 Driving with Trailer

Approximately 5 to 10 miles from your starting point, pull over in a safe place such as a parking lot to check that the load has not shifted and all cargo is secure. When driving with a trailer take these items into consideration:

- Recheck lights, hitch, and securement at each stop during travel.
- Acceleration is slower with a trailer in tow, so allow extra time/distance when

merging into traffic, changing lanes, and crossing traffic.

- Make wider turns to avoid collision at curves and corners.
- Know your trailer height to ensure you may safely clear bridges and overpasses along your route.
- Allow considerably more distance for stopping. Increase and maintain a greater distance between the tow vehicle and the vehicles in front of it. The amount of distance needed will depend on many factors for example the size of the vehicle and trailer, total weight of both, weather conditions, speed, etc.
- Do not apply brakes if trailer starts to sway; instead, release the accelerator pedal to reduce speed. Once the vehicle is under control, find a safe location to park and re-check the weight distribution.

1.2.5 Backing Up

Whenever possible, use a spotter while backing up. If a spotter is not available, walk around the vehicle and trailer before backing up to ensure no people or objects are in the path. Do not allow the trailer to jackknife. Pull forward to straighten out when necessary.

1.2.6 Parking

When parking the trailer, select level ground and confirm that the parking space is of sufficient size (length and width) to accommodate the trailer's dimensions, including wheel wells and tongue. Place chocks at the front and rear of the trailer wheels to prevent rolling.

1.3 Training

Drivers must complete training and pass a skills test before being approved as an operator qualified to tow vehicles.

1.4 Injury Reporting

If a GEI employee suffers an injury on the job that is not life threating, call Medcor Triage at 1-800-775-5866 to speak with a medical professional. Then, immediately report the injury to the Supervisor/Project Manager and Regional Safety Officer.

After verbal notification has been made, an Incident Report Form is to be completed by the employee and/or Supervisor/Project Manager and submitted to the People & Safety Team immediately following care of the incident. This form is available on the Safety App (smart phones) and on the Safety page on the GEI intranet.

Upon notification from a Branch or Office Manager, Human Resources, and/or the receipt of the Incident Report Form, the Regional Health & Safety Officer (RHSO) will conduct an investigation and evaluation on what happened and how and why it happened. The Corporate Health and Safety Officer (CHSO) will then recommend (as necessary) engineering controls, personal protection equipment, training or other appropriate

measures to minimize the potential for future injuries. The CHSO/RHSO may develop educational information based on lessons learned for distribution to GEI employees.

1.5 Limitations

Each driver must operate within the specific limitations set forth by his or her current driver's or operator's license.

1.6 References

http://www.nhtsa.gov/cars/problems/Equipment/towing/
http://www.dmv.org/how-to-guides/pulling-trailer.php
http://www.fmcsa.dot.gov/regulations/cargo-securement
29 CFR 1926.600 (Equipment)
29 CFR 1926.601 (Motor Vehicles)

1.7 Attachments

None

1.8 Contact

HealthandSafetyTeam@geiconsultants.com

1.9 Review History

- June 2016
- April 2016 - Created

RI/FS Schedule
GLLA Program - Lower Scajaquada Creek OU
Buffalo, NY

Task Name	2025 Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	2026 Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	2027 Jan	Feb	Mar	Apr	May	Jun	Jul	Aug	Sep	Oct	Nov
Complete Draft Work Plans		■		■																															
Submit Final Work Plans						◆																													
Subcontractor Agreements		■	■																																
Access Agreements/Permits			■	■																															
RI Landside Field Activities					■	■	■	■	■	■	■	■																							
Quarterly RI Landside Activities														■			■			■			■												
RI Sediment Field Activities							■	■																											
Laboratory Analysis/Validation/Data Compilation						■	■	■	■	■	■	■	■	■																					
RI Figures/Mapping Preparation/Findings Summary								■	■	■	■	■	■	■	■	■																			
RI Data Submission Report																	◆																		
Qualitative Human Health Risk Assessment																	■	■	■																
Fish and Wildlife Resource Impact Analysis							■	■	■	■	■	■	■	■	■	■	■	■	■																
Develop RAOs Consistent with AOC BUIs																				■	■														
RA Options/Technology Screening																						■	■												
Remedial Alternatives Costing/Evaluations																								■	■	■	■	■							
Select Preferred Alternative																													◆						
Draft RI/FS Report Preparation								■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■	■		
Public Meeting																																	●		
Submit Final RI/FS Report																																		◆	
PCT Meetings		●	●	●		●	●	●		●	●	●		●	●	●		●	●	●		●	●	●		●	●	●		●	●	●	●	●	