# Remedial Investigation Work Plan (RIWP)

**Steps at Saratoga Family Building**
2030 Fulton Street
Brooklyn, New York
BCP Site ID C224450

September 30, 2026

Prepared for:

**Steps Family LLC**
**c/o Riseboro Community Partnership**
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# Acronym List

µg/kg ..... Micrograms per Kilogram
µg/L ..... Micrograms per Liter
µg/m3 ..... Micrograms per Cubic Meter
mg/kg ..... Milligrams per Kilogram
4,4'-DDE ..... 4,4'-Dichlorodiphenyldichloroethylene
4,4'-DDD..... 4,4-Dichlorodiphenyldichloroethane
4,4'-DDT ..... 4,4-Dichlorodiphenyltrichloroethane
AMI ..... Area Median Income
amsl ..... Above Mean Sea Level
AOCs ..... Areas of Concern
ASP ..... Analytical Services Protocol
AWQSGVs ..... Ambient Water Quality Standards and Guidance Values
BCP ..... Brownfield Cleanup Program
BERs ..... Business Environmental Risks
bls ..... Below Land Surface
BNAs ..... Base Neutral Acids
CAMP ..... Community Air Monitoring Plan
CEQR ..... City Environmental Quality Review
CPP ..... Citizen Participation Plan
CRECs ..... Controlled Recognized Environmental Conditions
CSM ..... Conceptual Site Model
CVOCs ..... Chlorinated Volatile Organic Compounds
DCP ..... Department of City Planning
DEC ..... Department of Environmental Conservation
DER ..... Department of Environmental Remediation
DER-10 ..... NYSDEC DER-10 Technical Guidance for Site Investigation and Remediation
DER-31 ..... NYSDEC DER-31 Technical Guidance for Green and Sustainable Remediation
DUSR ..... Data Usability Summary Report
DVS ..... Data Validation Services, Inc.
ECs ..... Emerging Contaminants
EDD ..... Electronic Data Deliverable
EDR ..... Environmental Data Resources
ELAP ..... Environmental Laboratory Approval Program
ESA ..... Environmental Site Assessment
eV ..... Electron Volt
FEMA ..... Federal Emergency Management Agency
ft ..... Feet/Foot
GPR ..... Ground Penetrating Radar

# Acronym List (continued)

GSR ..... Green and Sustainable Remediation
HASP ..... Health and Safety Plan
HDFC ..... Housing Development Fund Corporation
HRECs ..... Historical Recognized Environmental Conditions
MEK ..... Methyl Ethyl Ketone; 2-butanone
NTUs ..... Nephelometric Turbidity Units
NYCRR ..... New York Codes, Rules, and Regulations
NYCOER ..... New York City Office of Environmental Remediation
NYSDEC ..... New York State Department of Environmental Conservation
NYSDOH ..... New York State Department of Health
ORP ..... Oxidation-Reduction Potential
PAHs ..... Polycyclic Aromatic Hydrocarbons
PBVs ..... Project Based Vouchers
PCBs ..... Polychlorinated Biphenyls
PCE ..... Tetrachloroethene (Perchloroethene)
PCSM ..... Preliminary Conceptual Site Model
PFAS ..... Per- and Polyfluoroalkyl Substances
PID ..... Photoionization Detector
PVC ..... Polyvinyl Chloride
PGWSCOs ..... Protection of Groundwater Soil Cleanup Objectives
QAPP/FSP ..... Quality Assurance Project Plan/Field Sampling Plan
QA/QC ..... Quality Assurance/ Quality Control
QHHEA ..... Qualitative Human Health Exposure Assessment
RAWP ..... Remedial Action Work Plan
RECs ..... Recognized Environmental Conditions
RI ..... Remedial Investigation
RIR ..... Remedial Investigation Report
RIWP ..... Remedial Investigation Work Plan
RRSCOs ..... Restricted Use Soil Cleanup Objectives
SCGs ..... Standards, Criteria, and Guidance
SVI ..... Soil Vapor Intrusion
SVOCs ..... Semivolatile Organic Compounds
TAL ..... Target Analyte List
TCL ..... Target Compound List
TICs ..... Tentatively Identified Compounds
TOGS ..... Technical & Operational Guidance Series
USEPA ..... United States Environmental Protection Agency
USCS ..... Unified Soils Classification System
USGS ..... United States Geological Survey
UUSCOs ..... Unrestricted Use Soil Cleanup Objectives
VOCs ..... Volatile Organic Compounds

# Certification

I, Jessica L. Taylor, certify that I am currently a qualified environmental professional and a NYS registered Professional Geologist and that this Remedial Investigation Work Plan was prepared in accordance with all applicable statutes and regulations and in substantial conformance with the DER Technical Guidance for Site Investigation and Remediation (DER-10) and DER Green Remediation (DER-31).

| Jessica L. Taylor, P.G. | September 30, 2026 |  |
|---|---|---|
| Professional Geologist NY #000320 | Date | Signature |

# 1. Introduction

Roux Environmental Engineering and Geology, D.P.C. (Roux), on behalf of Steps Family LLC c/o RiseBoro Community Partnership (Steps Family or the Applicant), has prepared this Remedial Investigation Work Plan (RIWP) for the property located at 2030 Fulton Street, Block 1549 in the Borough of Brooklyn, New York (Site). The Site previously included several tax lots (Lots 23, 24, 25, 26, 28, 29, 30, 31, 32, 33, 34, and 35) but has been merged into a single tax lot, Lot 28, which is referenced throughout this document and shown on Figure 1 and Figure 2.

The proposed development consists of an affordable residential housing building for families with 12 floors, a rooftop and a cellar. The building will be comprised of 168 units for affordable housing. The cellar and ground floor of the building will feature community facility space. The second floor through the 12th floor will consist of residences. The building will be accompanied by a backyard with a greenhouse, gardening plots, seating areas, and a fitness center. The Family Building will serve a range of affordability levels. 26 units (16% of total units) will be a homeless set aside, 8 of these 26 units will receive Project Based Vouchers (PBVs) and 18 of them will be underwritten at shelter rents. 28 units (17% of total units) will be affordable to extremely or very low-income households earning 40% of AMI. 38 units (22% of total units) will be available to households earning 50% of AMI. 31 units (18% of total units) will be available to households earning 60% of AMI. 45 units (27% of total units) will be available to households earning 80% of AMI.

## 1.1 Brownfield Cleanup Program Application and Environmental Work Plans

This RIWP is being submitted to the New York State Department of Environmental Conservation (NYSDEC) concurrently with an application to enroll the Site into the NYSDEC Brownfield Cleanup Program (BCP) with Steps Family LLC c/o RiseBoro Community Partnership as a volunteer.

The purpose of the Remedial Investigation (RI) will be to determine the nature and extent of contamination at the Site, characterize environmental media, qualitatively assess the potential exposure of receptors to Site contaminants, and develop any other additional data necessary to support the development of a Remedial Action Work Plan (RAWP).

## 1.2 RIWP Document Organization

This RIWP contains a background information describing the Site, its history, and results of previous environmental investigations (Section 2); the objectives and scope of the RI (Section 3); Sections 4 and 5 describing the project operations plans (e.g., Quality Assurance/Quality Control, Health and Safety); and the reporting requirements and the proposed project schedule are discussed in Section 6. Figures are provided to illustrate Site conditions and the locations of proposed investigation activities.

## 1.3 Project Team and Contact Information

A list of names, contact information, qualifications and roles of the principal Roux personnel who will participate in the investigation are included below and in the Quality Assurance Project Plan/Field Sampling Plan (QAPP/FSP) in Appendix B.

| Roux Personnel | Title | Contact Information |
|---|---|---|
| Jessica Taylor, P.G.<br>Principal Hydrogeologist | Project Principal | (631) 232-2600<br>jtaylor@rouxinc.com |
| Brooke Hildebrand<br>Project Scientist | Project Manager | (631) 232-2600<br>bhildebrand@rouxinc.com |
| To Be Determined | Field Manager | (631) 232-2600 |
| To Be Determined | Quality Assurance Officer | (631) 232-2600 |

The name and contact information for the Applicant is:

Mr. Stephan Petryczka
Steps Family LLC
c/o Riseboro Community Partnership
217 Wyckoff Avenue
Brooklyn, New York 11237

Contact information for the major subcontractors involved in the investigation (driller, data validator and laboratory) are also included below and in the QAPP/FSP.

| Subcontractor | Company Name | Contact Information |
|---|---|---|
| Laboratory Analysis | Eurofins Environment Testing Northeast, LLC | (203) 308-0880 |
| Data Usability Summary Report Preparation | Judy Harry of Data Validation Services, North Creek, NY | (518) 251-4429 |
| Drilling | To Be Determined | To Be Determined |
| NYS Licensed Surveying | To Be Determined | To Be Determined |

Eurofins Environment Testing Northeast, LLC, a New York State Department of Health (NYSDOH) Environmental Laboratory Accreditation Program (ELAP)-certified laboratory, will be used to analyze all samples collected during the RI. Drilling and other subcontracted services have not been selected as part of the preparation of this RIWP. This information will be provided to the NYSDEC prior to the start of field activities.

# 2. Background

This section provides pertinent background information, including a description of the Site and its setting, the history of the Site, and the results of previous environmental work conducted at the Site.

## 2.1 Site Description and Setting

A Site location map is included as Figure 1 and a Site plan is included as Figure 2.

| Property Location | |
|---|---|
| Property Name: | Steps at Saratoga Family Building |
| Property Address: | 2030 Fulton Street |
| Property Town, County, State: | Brooklyn, Kings County, New York |
| Property Tax Identification: | Block 1549, Lot 28 |
| Property Topographic Quadrangle: | USGS-Brooklyn, New York |
| Nearest Intersection: | Fulton Street and Hull Street to the north, and Fulton Street and Saratoga Avenue to the east. |
| Area Description: | The Site encompasses an area of approximately 0.587-acres and is currently vacant and undeveloped, and surrounded by a locked chain-link fence. |

| Property Information | |
|---|---|
| Property Acreage: | 0.587-acres |
| Property Shape: | Rectangle |
| Improvements: | None |

The previous environmental reports, including the Phase I Environmental Site Assessment (ESA) prepared by Roux, dated October 2023 (Roux Phase I ESA), and the Limited Phase II ESA prepared by Roux, dated November 7, 2025 (Roux Phase II ESA), are included in Appendix A.

### 2.1.1 Utilities

No utilities were observed to be connected to the Site during the Roux Phase I Environmental Site Assessment (ESA).

### 2.1.2 Topography

The grade of the Site is generally flat. The average elevation of the Site is approximately 80 feet above mean sea level (ft amsl) as shown in the USGS 7.5 Minute Series (Appendix F of the Roux Phase I ESA [Appendix A]).

### 2.1.3 Wetland Areas and Surface Water Bodies

According to the Environmental Data Resources (EDR) Report (Appendix B of the Roux Phase I ESA [Appendix A]) and National Wetlands Inventory, the Site is not located in or adjacent to any wetlands. According to a review of Federal Emergency Management Agency (FEMA) Flood Maps, the Site is not located within the 100-year or 500-year flood zones.

### 2.1.4 Soil

Based on the Roux Phase II ESA, the Site is underlain with a mixture of historical fill consisting of predominantly sand, with gravel, brick, and other demolition debris throughout the 0-5 feet below land surface (ft bls) range surveyed. Bedrock was not encountered during drilling to a terminal depth of 5 ft bls.

### 2.1.5 Groundwater

No formal groundwater flow study was conducted in the previously mentioned environmental investigations. Based on the surrounding topography and regional direction of groundwater flow in the area, the presumed groundwater flow in the vicinity of the Site is toward East River, located approximately 3.15 miles northwest of the Site.

### 2.1.6 Neighboring Properties and Zoning

The Site is located in an area that has historically been residential mixed with commercial and industrial, with numerous adjoining facilities identified in environmental databases, including two listings in the NY SPILLS database. Several upgradient facilities were identified in environmental databases, including listings in the EDR HIST AUTO and DRYCLEANERS databases. The Site is bounded to the north by Fulton Street and Hull Street, beyond which are residential properties and churches. The Site is bounded to the south by residential properties. The Site is bounded to the east by Saratoga Avenue, beyond which is I.S. 271 John M. Coleman School. The Site is bounded to the west by vacant properties.

The Site is currently zoned R7D/C2-4, allowing for greater residential development while incorporating ground floor retail/service spaces to maintain the vitality of the street. The area surrounding the Site is similar with mixed commercial and residential zoning. I.S. 271 John M. Coleman School to the east, Saratoga Urban Agro-Ecological Center to the northeast and Louis Place Pocket Park to the south are sensitive human receptors in close proximity of the Site. There are no other land uses within close proximity of the Site that are sensitive human or ecological receptors.

## 2.2 Site History

Based on review of Roux's Phase I ESA, dated October 25, 2023, the Site was first developed prior to 1908 with mixed use buildings on all lots except for 224 Saratoga Avenue, which was developed with a dwelling. Listings for the property consisted of commercial and manufacturing operations including a machine shop, noodle manufacturing and packing facility, upholstery facility, blind manufacturer, tin smith, sheet metal and roofing company. All buildings remained present on the 1965 Sanborn Map. Demolition of the buildings began in 1978 and occurred over time until at least 1994. By 2001, the Site was depicted as completely vacant and remained vacant until the most recent 2007 Sanborn Map. During the 2023 reconnaissance, the Site was observed to be vacant and undeveloped.

## 2.3 Environmental Conditions/Results of Previous Environmental Investigations

This section provides an overview of previous environmental-related activities completed at the Site, based on a review of readily available information and the following environmental reports (provided in Appendix A). The following environmental reports were available for review:

- Phase I ESA, prepared by Roux on behalf of Steps at Saratoga Housing Development Fund Corporation, dated October 25, 2023; and
- Limited Phase II ESA, prepared by Roux on behalf of Steps at Saratoga Housing Development Fund Corporation (HDFC), dated November 7, 2025.

A summary of the findings from assessments of the Site is provided below.

### 2.3.1 Phase I ESA, prepared by Roux on behalf of Steps at Saratoga HDFC, dated October 25, 2023.

The Phase I ESA was prepared for the Site and additional offsite lots adjoining the Site to the west (Block 1549, the remainder of former Lot 18). Based on the information gathered during the Phase I ESA process, Roux identified the following Recognized Environmental Conditions (RECs) specific to the Site:

- Historical Use of the Site: According to the EDR City Directory Abstract, the Site was occupied by multiple commercial operations including a tinsmith, sheet metal and roofing facility from 1993 to 2007. No records of any releases or waste generation were identified in the EDR environmental database report. However, the potential for a historical petroleum or chemical release from these historical uses that negatively impacts the environmental quality of the Site cannot be ruled out, and is considered a REC.

Roux did not identify any Controlled RECs (CRECs) or Historical RECs (HRECs) in connection with the Site.

Roux identified the following Business Environmental Risks (BERs) in connection with the Site:

- Suspected Presence of Fill Material: The Site has had buildings associated with former buildings that have been demolished and there is potential to encounter former foundations, historical fill, and concrete in the Site subsurface.
- E-Designations: The Site is associated with a hazardous materials and noise E-Designation (E-185) associated with City Environmental Quality Review (CEQR# 07DCP070K) placed by the Department of City Planning. Any remediation of the Site will need to be coordinated with the New York City Office of Environmental Remediation (NYCOER) concurrently with development.

### 2.3.2 Limited Phase II ESA Report, prepared by Roux on behalf of Steps at Saratoga HDFC, dated September 12, 2025.

Previous sample data from the Roux Limited Phase II ESA is shown on Figure 3 and Figure 4. Roux performed twelve soil borings to 5 ft bls and collected two soil vapor samples. The following conclusions were determined based on the results of the Roux Phase II ESA:

- Historical fill was observed extending to the terminal depth of 5 ft bls at each soil boring location.
- Soil samples including one duplicate were collected from twelve borings at the Site; eleven soil samples were collected from 0-2 ft bls and one soil sample was collected from 1-3 ft bls. No evidence of contamination was observed during sample collection.

- Analytical data indicates soils at the Site are impacted with semivolatile organic compounds (SVOCs) above Unrestricted Use Soil Cleanup Objectives (UUSCOs), Restricted Residential Soil Cleanup Objectives (RRSCOs) and Protection of Groundwater Soil Cleanup Objectives (PGWSCOs), and metals and pesticides above UUSCOs.
- Volatile Organic Compounds (VOCs) and polychlorinated biphenyls (PCBs) were all below UUSCOs, RRSCOs and PGWSCOs.
- Concentrations of VOCs in soil vapor were detected above laboratory reporting limits at all sampling locations and included petroleum-related and chlorinated VOCs (CVOCs). Butane specifically was detected at an elevated concentration.

### 2.3.3 Identification of Areas of Concern (AOCs) Based on Previous Investigations / Remediation

Based on the findings of the prior investigations, the following AOCs that will be further investigated as part of this RIWP include:

- Presence of contaminated historical fill; and
- Presence of contamination from former Site and surrounding property operations.

# 3. Preliminary Conceptual Site Model, RI Work Plan Objectives, Scope, and Rationale

Roux, on behalf of the Applicant, has developed a RI scope of work that satisfies NYSDEC BCP requirements. Data collected during the RI will determine the basis for remedial actions for the Site.

Standards, Criteria, and Guidance (SCGs) for soil at BCP sites are the numerical SCOs presented in Part 375. The SCOs are categorized into unrestricted use criteria and restricted use (residential, restricted-residential, commercial, or industrial) criteria, as well as criteria for protection of groundwater and ecological resources. The applicability of each category of SCOs is determined based upon the current and reasonably anticipated future use of the Site, as well as cleanup tracks being evaluated. The anticipated redevelopment of the Site will include a mixed-use, affordable housing residential building for families, with accompanying ground floor community service spaces.

The SCGs for groundwater are the NYSDEC Ambient Water-Quality Standards and Guidance Values (AWQSGV)s for Class G groundwater – Technical & Operational Guidance Series (TOGS) 1.1.1 (groundwater is not and will not be used for drinking water, as the Site is/will be connected to the public water supply).

SCGs for evaluating Soil Vapor Intrusion (SVI) are the NYSDOH's Final Guidance for Evaluating Soil Vapor Intrusion in the State of New York, October 2006, Revised February 2024 (2006 NYSDOH SVI Guidance and its updates). Potential soil vapor impacts identified in accordance with the 2006 NYSDOH SVI Guidance and its updates will be considered.

## 3.1 Preliminary Conceptual Site Model

This section describes the Preliminary Conceptual Site Model (PCSM) developed for the Site. The PCSM is a tool used to evaluate and identify potential sources of contamination, target analytes, potential entry and mechanisms for transport in the subsurface environment and into the Site structures, potential exposure routes, and the environmental media and locations most likely to have been impacted by the target analytes.

### 3.1.1 Potential Sources, Target Analytes, Transport Pathways and Receptors

Based on the previous investigation conducted by Roux and historical documentation including Sanborn Fire Insurance Maps and the City Directory, the Site was first developed by 1888 with a mixed-use building present on each lot (typically residential with ground floor retail). Listings for the Site consisted of residence names and machine shop, noodle manufacturing and packing facility, upholstery facility, blind manufacturer, tin smith, sheet metal and roofing company. All buildings remained present on the 1965 Sanborn Map. Demolition of the buildings began in 1978 and occurred over time until at least 1994. By 2001, all former buildings were no longer present the Site appeared as vacant. The Site remained in this layout until the most recent 2007 Sanborn Map. During the 2023 reconnaissance, the Site was observed to be vacant and undeveloped. No reports of releases or waste generation were identified in the EDR database report. However, the use of the Site by multiple commercial operations including a tinsmith, sheet metal and roofing facility had the potential for a release of petroleum products or chemical release into the subsurface, which was considered a REC in the Phase I ESA. The primary environmental AOCs and

potential sources of contamination associated with the Site were the historical fill and former operations at the Site. Roux's Limited Phase II ESA indicated soils at the Site are impacted with SVOCs above UUSCOs, RRSCOs and PGWSCOs, and metals and pesticides above UUSCOs. VOCs and PCBs were all below UUSCOs, RRSCOs and PGWSCOs. The concentrations of SVOCs, metals, and pesticides are typical of urban sites where fill material was placed in the area and are not indicative of any other potential source or historical activity. Soil vapor samples were collected from two locations at the Site. Concentrations of VOCs in soil vapor were detected above laboratory reporting limits at all sampling locations and included petroleum-related and chlorinated VOCs.

Based on the target analytes present and the media in which these contaminants were identified, the following potential transport mechanisms were identified for the Site:

- Target analytes may potentially remain sorbed to soil (soil impacts);
- Leaching of target analytes from soil to groundwater (groundwater impacts);
- Transport of target analytes in the dissolved-state through groundwater flow; and
- Volatilization of target analytes from soil and groundwater into soil pore space and potentially into surface structures (vapor intrusion).

The following potential exposure routes for target analytes have been identified for the Site:

- Inhalation of VOCs/CVOCs from soil vapor;
- Dermal contact and incidental ingestion of soil; and
- Dermal contact with groundwater is also considered minimal risk as groundwater is not used for drinking water or irrigation purposes.

## 3.2 Objectives

The objective of the RI is to further assess the nature and extent of contamination associated with the following AOCs:

- Former Site and surrounding properties operations; and
- The presence of historical fill at the Site.

The RI will evaluate soil, groundwater, and soil vapor impacts on-Site and at the property boundaries to provide the basis for refining the PCSM and remedial action selection and to determine the general potential for off-Site impacts. Environmental data collected during the RI will be used to qualitatively assess the potential exposure of receptors to Site contaminants and develop the information necessary to support the development of a Remedial Action Work Plan (RAWP).

## 3.3 RI Scope

The scope of the RI will include the collection of sufficient Site investigation data so that, together with the historical data, the entire Site will be sufficiently characterized to support the development of the Site-wide RAWP.

To accomplish this, the scope of work for the RI will include the following:

- The performance of Site reconnaissance to confirm proposed sampling locations;

- The advancement of soil borings, collection of soil samples, installation of permanent groundwater monitoring wells, collection of groundwater samples from new monitoring wells, installation of soil vapor points, and sampling of new soil vapor points;
- The collection of soil, groundwater, and soil vapor data sufficient to define the nature and extent of impacted media and current Site conditions and to evaluate potential off-Site groundwater and/or soil vapor migration;
- The collection of a synoptic round of groundwater level measurements and the collection of additional land survey data as needed for developing a groundwater elevation contour map; and
- The performance of a qualitative human health exposure assessment (QHHEA) to identify existing and potential exposure pathways and evaluate contaminant fate and transport.

All investigation activity will be conducted in accordance with the applicable requirements of the NYSDEC DER-10.

Soil samples from all soil borings and all groundwater samples collected from the investigation described in detail in embedded Tables 1 and 2 for soil and groundwater, respectively, will be analyzed for the target compound list (TCL)/Part 375 plus 30/target analyte list (TAL) (TCL + 30/TAL) list of parameters including:

- TCL/Part 375 VOC + 10 Tentatively identified compounds (TICs);
- TCL/Part 375 Base neutral acids (BNA)/SVOCs + 20 TICs;
- TCL/Part 375 Pesticides;
- TCL/Part 375 Herbicides;
- TCL/Part 375 Polychlorinated Biphenyls (PCBs);
- TAL/Part 375 Metals (including hexavalent chromium);
- Total Cyanide; and
- Emerging Contaminants (ECs) list includes 1,4-Dioxane and all Per- and Polyfluoroalkyl Substances (PFAS) compounds listed in the NYSDEC April 2023 “Sampling, Analysis, and Assessment of PFAS.” PFAS in soil and groundwater will be analyzed by USEPA Method 1633A and 1,4-Dioxane in groundwater will be analyzed by USEPA Method 8270D SIM and 1,4Dioxane in soil will be analyzed by USEPA Method 8270D.

Soil samples will also be collected for geologic logging in accordance with the Unified Soils Classification System (USCS) and for organoleptic inspection (for evidence of contamination including staining and/or odors).

All soil vapor air samples will be analyzed for VOCs using USEPA Method TO-15. All soil vapor samples will also be analyzed for methane using USEPA Method 18 (Modified) due to elevated concentration of butane detected in soil vapor samples collected during the Roux Phase II ESA.

All data will be produced in accordance with NYSDEC Analytical Services Protocol (ASP) Category B deliverables and will be reviewed and validated by Data Validation Services, Inc. (DVS), an independent data validator. Data Validation Services will prepare a Data Usability Summary Report (DUSR) before data is incorporated into the RI Report (RIR) for the Site. All data will be submitted to NYSDEC in electronic format, in accordance with DER-10, Section 1.15.

The overall scope of each component of the RI is discussed in the following subsections. Detailed field sampling procedures are provided in the Quality Assurance Project Plan/Field Sampling Plan (QAPP/FSP; Appendix B of this RIWP). The proposed sampling locations are shown on Figure 6 of this RIWP for soil, groundwater, and soil vapor. Additional information, including intervals to be sampled and sample rationale, is provided in embedded Tables 1, 2 and 3 for soil, groundwater, and soil vapor, respectively.

### 3.3.1 Site Reconnaissance and Survey

Roux has performed Site reconnaissance and has identified potential AOCs, described in Section 3.1., which will be targeted during the RI. An inspection of the existing Site conditions will be conducted to determine final locations of soil borings and monitoring wells based on actual field conditions. Proposed sampling locations will be marked with spray paint or flagged and surveyed (horizontal coordinates and elevation) prior to the intrusive work. Proposed sampling locations needing to be relocated more than 10 feet due to inadequate Site conditions will first be reviewed and approved by the NYSDEC project manager.

### 3.3.2 Soil Investigation

To further characterize the soil conditions at the Site, eleven borings (RXS-01 through RXS-11) will be advanced at the locations shown in Figure 5. Four soil borings (RXS-01 through RXS-04) will be advanced ten feet below observed groundwater at the locations shown in Figure 5 for the installation of co-located groundwater monitoring wells as described below. It is anticipated groundwater will be encountered approximately 75 to 80 ft bls. Seven additional soil borings (RXS-05 through RXS-11) will be advanced to two feet below the observed fill layer at the locations shown in Figure 5. Co-located soil vapor points will be installed at each of the seven additional soil borings except for RXS-8, as described below. At each borehole location utility clearances will be performed prior to advancement of soil borings using hand tools and/or vacuum excavation to a minimum of five ft bls to confirm that no subsurface utilities are present. Should a utility or other feature be observed during pre-clearance activities, the sampling location will be relocated. Soil samples will be collected by utilizing a Geoprobe direct-push drill rig, or roto-sonic drill rig, if required. Soil samples will be collected continuously from grade to the terminal depth of each soil boring for observation and collection of lithology and screening with a photoionization detector (PID), and will be sampled as 2 ft composites, with the exception of VOC grab samples and the upper 0 to 0.5 ft soil sample. During soil boring advancement, lithology will be recorded according to the Unified Soils Classification System (USCS), and soil will be inspected for evidence (visual or olfactory) of contamination and field screened continuously for VOCs using a PID with a 10.6 electron volt (eV) lamp.

The proposed soil boring locations, shown on Figure 5, will have soil samples collected from the following sample depths within each soil boring, as shown on Table 1 below. A sample will be collected within the upper 0 to 0.5 ft bls to assess human exposures to exposed surface soils as per DER-10 Section 3.5.1(b)(1). If applicable, a sample will be collected from the most impacted interval as determined by field observations (e.g. staining and odors). If no impacts are observed, then a sample will not be collected. A sample from the bottom of the historical fill layer will be collected to delineate impacts caused by historical fill. A sample will be collected from the first interval beneath observed impacts or from beneath the historical fill layer, whichever is deeper, to provide a vertical profile of native soil conditions. Borings that are not being converted to monitoring wells will be advanced to a minimum depth of 20 ft bls, or to the bottom depth of the fill layer if deeper, and a soil sample will be collected from the terminal depth to provide data on soil conditions at depth. Borings advanced to the groundwater table for monitoring well installation will have a soil sample collected from the two-foot interval above the observed groundwater table to provide data on soil conditions at depth.

The summary table below provides details for the soil sampling locations that are proposed as part of this RIWP:

**Table 1. Proposed Soil Sample Locations, Analyses and Rationale**

| Soil Boring Location | Sample Depth Interval | Analytical Suites | Rationale |
|---|---|---|---|
| RXS-01<br>RXS-02<br>RXS-03<br>RXS-04 | ▪ 0-0.5 ft bls<br>▪ Most impacted 2 ft interval, if applicable*<br>▪ Bottom 2 ft of Fill<br>▪ Clean 2 ft Interval Beneath Impacts or Fill, whichever is deeper<br>▪ 2 ft above Groundwater Table | Full Part 375** and ECs*** | Evaluate soil conditions on Site and determine extent of contamination both vertically to the groundwater interface and horizontally across the Site. |
| RXS-05<br>RXS-06<br>RXS-07<br>RXS-08<br>RXS-09<br>RXS-10<br>RXS-11 | ▪ 0-0.5 ft bls<br>▪ Most impacted 2 ft interval, if applicable*<br>▪ Bottom 2 ft of Fill<br>▪ Clean 2ft Interval Beneath Impacts or Fill<br>▪ Soil Boring Termination Depth | Full Part 375 and ECs | Evaluate soil conditions on Site and determine extent of contamination both vertically to the historical fill/native soil interface and horizontally across the Site. |

* Note: If applicable, a sample will be collected from the most impacted interval as determined by field observations (e.g. staining and odors). If no impacts are observed, then a sample will not be collected.

** Note: Part 375/TCL plus 30/ TAL (TCL + 30/TAL) includes:

- Part 375/TCL VOCs + 10 TICs;
- Part 375/TCL Base Neutral Acids (BNA)/ SVOCs + 20 TICs;
- Part 375/TCL Pesticides;
- Part 375/TCL Herbicides;
- Part 375/TCL PCBs;
- Part 375/TAL Metals (including hexavalent chromium); and
- Total Cyanide.

***Note: Emerging Contaminants (ECs) list includes 1,4-Dioxane and the Per- and Polyfluoroalkyl Substances (PFAS) compounds listed in the NYSDEC April 2023 "Sampling, Analysis, and Assessment of Per-And Polyfluoroalkyl Substances (PFAS)". PFAS in soil will be analyzed by USEPA Method 1633A and 1,4-Dioxane in soil will be analyzed by USEPA Method 8270D.

### 3.3.3 Groundwater Investigation

To evaluate groundwater quality conditions across the Site, four permanent monitoring wells (RXS-01 through RXS-04) will be installed in converted soil boring locations. Roux will collect a groundwater sample from each newly installed monitoring well location, as shown on Figure 5. All monitoring wells will be surveyed for horizontal coordinates, land surface elevation and measuring point elevation.

Monitoring wells will be constructed of 2-inch inside diameter, Schedule 40 polyvinyl chloride (PVC) casing and, 0.020 inch slot, machined screen. Well construction will be as follows:

- Well screens will be 10 feet long, and will be installed with three feet above and seven feet below the water table.
- A sand pack will be placed around the well screen, extending two feet above the top of the screened zone.

- A minimum two-foot thick bentonite pellet seal will be placed above the sand pack. Once the pellets have been allowed to hydrate, cement bentonite grout will be pumped into the remaining annular space from the bottom up.
- The wells will be completed using locking well plugs, and flush mounted, bolt down, watertight, manhole covers cemented into place.

Each monitoring well will be developed to ensure proper hydraulic connection with the aquifer and to reduce/eliminate turbidity of the groundwater. The wells will be developed using a submersible pump, which will be surged periodically until well yield is consistent and has turbidity below 50 nephelometric turbidity units (NTUs), if possible. Detailed procedures regarding well development can be found in the QAPP/FSP (Appendix B).

Prior to sampling, the depth to groundwater in each monitoring well will be measured using an electronic water level meter and a groundwater contour map will be developed using the survey data. At least one week following redevelopment, all on-Site wells will then be sampled consistent with USEPA low-flow sampling requirements using a bladder pump and one round of groundwater samples will be collected in accordance with the QAPP/FSP. Field parameters (e.g., pH, dissolved oxygen, oxidation-reduction potential [ORP], etc.) will also be collected using a water quality meter during purging and prior to sampling.

The summary table below provides details for the groundwater sampling locations that are proposed as part of this RIWP:

**Table 2. Proposed Groundwater Sample Locations, Analyses and Rationale**

| Monitoring Well Location | Sample Depth Interval (ft bls) | Analytical Suites | Rationale |
|---|---|---|---|
| RXS-01 | 75-85 (dependent on where groundwater is observed) | Full Part 375* and ECs** | Evaluate groundwater conditions on Site |
| RXS-02 | 75-85 (dependent on where groundwater is observed) | Full Part 375 and ECs | Evaluate groundwater conditions on Site |
| RXS-03 | 75-85 (dependent on where groundwater is observed) | Full Part 375 and ECs | Evaluate groundwater conditions on Site |
| RXS-04 | 75-85 (dependent on where groundwater is observed) | Full Part 375 and ECs | Evaluate groundwater conditions on Site |

* Note: Part 375/TCL plus 30/ TAL (TCL + 30/TAL) includes:
  - Part 375/TCL VOCs + 10 TICs;
  - Part 375/TCL Base Neutral Acids (BNA)/ SVOCs + 20 TICs;
  - Part 375/TCL Pesticides;
  - Part 375/TCL Herbicides;
  - Part 375/TCL PCBs;
  - Part 375/TAL Metals (including hexavalent chromium); and
  - Total Cyanide.

**Note: Emerging Contaminants (ECs) list includes 1,4-Dioxane and the Per- and Polyfluoroalkyl Substances (PFAS) compounds listed in the NYSDEC April 2023 "Sampling, Analysis, and Assessment of Per-And Polyfluoroalkyl Substances (PFAS)". PFAS in groundwater will be analyzed by USEPA Method 1633A and 1,4-Dioxane in groundwater will be analyzed by USEPA Method 8270D SIM.

### 3.3.4 Soil Vapor Investigation

To evaluate soil vapor and the potential for SVI into the proposed building, Roux will install six soil vapor points (RXS-05, RXS-6, RXS-7, RXS-9, RXS-10 and RXS-11) in converted soil boring locations using a Geoprobe® direct-push rig to the target depth of fifteen ft bls to represent the approximate depth of excavation for the building cellar. One soil vapor sample will be collected from each point. New Teflon®-lined tubing will be attached to an expendable soil vapor sampling point with a 6-inch stainless steel screen. The soil vapor points will be backfilled with #2 Morie sand to approximately one foot above the screen. The remainder of the borehole will be backfilled with a concrete/bentonite slurry to grade.

In accordance with the 2006 NYSDOH Guidance for Evaluating SVI and its updates, the integrity of each soil vapor sampling point seal will be checked following installation to verify that the soil vapor sample will not be compromised by inadvertent introduction of ambient air into the sample. The soil vapor samples will be collected using pre-cleaned (batch certified) 6-liter summa canisters with regulators calibrated to collect samples over a 2-hour period and analyzed using USEPA Method TO-15 for VOCs and USEPA Method 18 (Modified) for methane. The proposed soil vapor sampling locations are shown on Figure 6. Additional details regarding soil vapor sampling methods are provided in the QAPP/FSP (Appendix B).

The summary table below provides details for the soil vapor sampling locations that are proposed as part of this RIWP:

**Table 3. Proposed Soil Vapor Sample Locations, Analyses and Rationale**

| Soil Vapor Point Location | Sample Depth Interval (ft bls) | Analytical Suites | Rationale |
|---|---|---|---|
| RXS-05 | 14.5-15 | VOCs by TO-15 and methane | Evaluate soil vapor conditions on Site. |
| RXS-06 | 14.5-15 | VOCs by TO-15 and methane | Evaluate soil vapor conditions on Site. |
| RXS-07 | 14.5-15 | VOCs by TO-15 and methane | Evaluate soil vapor conditions on Site. |
| RXS-09 | 14.5-15 | VOCs by TO-15 and methane | Evaluate soil vapor conditions on Site. |
| RXS-10 | 14.5-15 | VOCs by TO-15 and methane | Evaluate soil vapor conditions on Site. |
| RXS-11 | 14.5-15 | VOCs by TO-15 and methane | Evaluate soil vapor conditions on Site. |

### 3.3.5 Management of Investigation Derived Waste

Management of investigation derived waste will be completed in accordance with NYSDEC DER-10, Section 3.3 (e).

All waste materials (drill cuttings, decontamination water, etc.) generated during the RI will be consolidated, and stored in appropriate labeled bulk containers (drums, etc.) with labels indicating pending analysis as a hazardous waste material. The drums will be temporarily staged on protective sheeting in an investigation derived waste storage area onsite that will be covered for protection from the elements. Roux will then coordinate waste characterization and disposal by appropriate means.

## 3.4 Green and Sustainable Remediation (GSR)

Green remediation principles and techniques will be implemented to the extent feasible in the design, implementation, and site management of the remedy using best practives described in DER-31: Green Remediation. For each phase of the remediation, starting from the RI through Remedial Action Work Plan (RAWP) preparation, the environmental footprint analysis tool, SiteWise™, will be utilized. During the RI phase specifically, green remediation principles and techniques will be implemented to the extent feasible which may include but is not limited to carpooling of field staff and drilling staff, limiting the number of equipment and laboratory drop-offs, limiting water and energy usage, keeping equipment onsite instead of mobilizing and demobilizing daily, and turning off equipment when not in use.

The Site is entering the program at the investigation Stage; therefore, a Climate Change Screening or Vulnerability Assessment is not required at this time. The remedial design will include a climate change vulnerability assessment to evaluate the impact of climate change on the project site and the proposed remedy. Potential vulnerabilities associated with extreme weather events (e.g., hurricanes, lightning, heat stress and drought), flooding, and sea level rise will be identified, and the measures to minimize the impact of climate change on potential identified vulnerabilities will be implemented. The proposed redevelopment and remediation of the Site will not have an adverse impact on any existing natural resources. The proposed redevelopment is not expected to generate more than 50 tons of solid waste per week and therefore will not have a significant adverse impact to the NYC solid waste management systems. Onsite reuse of NYSDEC-approved material will be prioritized during remediation to minimize waste generation and greenhouse gas emissions due to truck traffic.

# 4. Quality Assurance/ Quality Control (QA/QC)

The goal of the QA/QC aspect of the RI is to ensure that suitable and verifiable data results from sampling and analyses are performed. To accomplish this, a QAPP/FSP has been prepared and is provided as Appendix B.

# 5. Health and Safety

A site-specific Health and Safety Plan (HASP) has been prepared for the Site and is provided in Appendix C. A site-specific Community Air Monitoring Plan (CAMP) has been prepared for the Site and is provided as Appendix C within the HASP document.

# 6. Reporting and Schedule

The following will be provided to the NYSDEC during the course of the RI work.

*Daily Progress Reports*

Daily progress report submittals will be provided to the NYSDEC during RI field activities and will include the following.

- Discovery of any previously unknown contaminated media during RI activities will be promptly communicated to NYSDEC's project manager;
- A Site map will be provided to identify locations discussed in progress reports provided to NYSDEC;
- An update of progress made during the reporting day;
- Locations of work keyed to a map of the Site;
- A summary of CAMP results, including any potential exceedances;
- An explanation of notable Site conditions; and
- Photos of the work.

Daily reports are not intended to be the mode of communication for notification to the NYSDEC of emergencies (accident, spill), requests for changes to the RIWP or other sensitive or time critical information. However, such conditions must also be included in the daily reports. Emergency conditions and changes to the SRIWP will be addressed directly to NYSDEC Project Manager via personal communication.

*Monthly Reports*

Monthly reports will be submitted to NYSDEC and NYSDOH Project Managers by the 10$^{th}$ of the month following the end of monthly reporting period. Monthly reports will commence upon approval of the Citizen Participation Plan (CPP) and will include:

- Activities relative to the Site during the previous reporting period and those anticipated for the next reporting period;
- Description of approved activity modifications, including changes of work scope and/or schedule;
- Sampling results received following internal data review and validation, as applicable; and
- An update of the remedial schedule including the percentage of project completion, unresolved delays encountered or anticipated that may affect the future schedule, and efforts made to mitigate such delays.

*Remedial Investigation Report*

A RIR will be prepared for the Site, which will summarize the results and findings of the investigations on the BCP Site. The RIR will be prepared in accordance with the NYSDEC requirements for the BCP and will meet the technical requirements of DER-10.

*Schedule*

The following schedule outlines the major milestones for the Site:

| Timeframe | Description |
|---|---|
| December 2025 | Submit BCP Application and RIWP |
| August 2026 | Finalize and Execute Brownfield Cleanup Agreement |
| September 2026 | Submit Citizen Participation Plan |
| September 2026 | Finalize Remedial Investigation Work Plan |
| October - November 2026 | Remedial Investigation Implementation |
| January 2027 | Submit Remedial Investigation Report |
| January 2027 | Submit Remedial Action Work Plan |
| June 2027 | Remedial Action Work Plan approved, and Decision Document Issued |
| July 2027 | Begin construction and remedial action |
| July 2027 – July 2028 | Remedial Action and construction |
| June 2028 | Submit Environmental Easement (if necessary) |
| August 2028 | Submit Draft Site Management Plan |
| October 2028 | Submit Draft Final Engineering Report and Approvable SMP (if necessary) |
| November 2028 | Submit Approvable Final Engineering Report and Record Easement |
| December 2028 | Receive Certificate of Completion |

Respectfully submitted,

**ROUX ENVIRONMENTAL ENGINEERING AND GEOLOGY, D.P.C.**

Brooke Hildebrand
Project Scientist

Jessica L. Taylor, P.G.
Principal Hydrogeologist

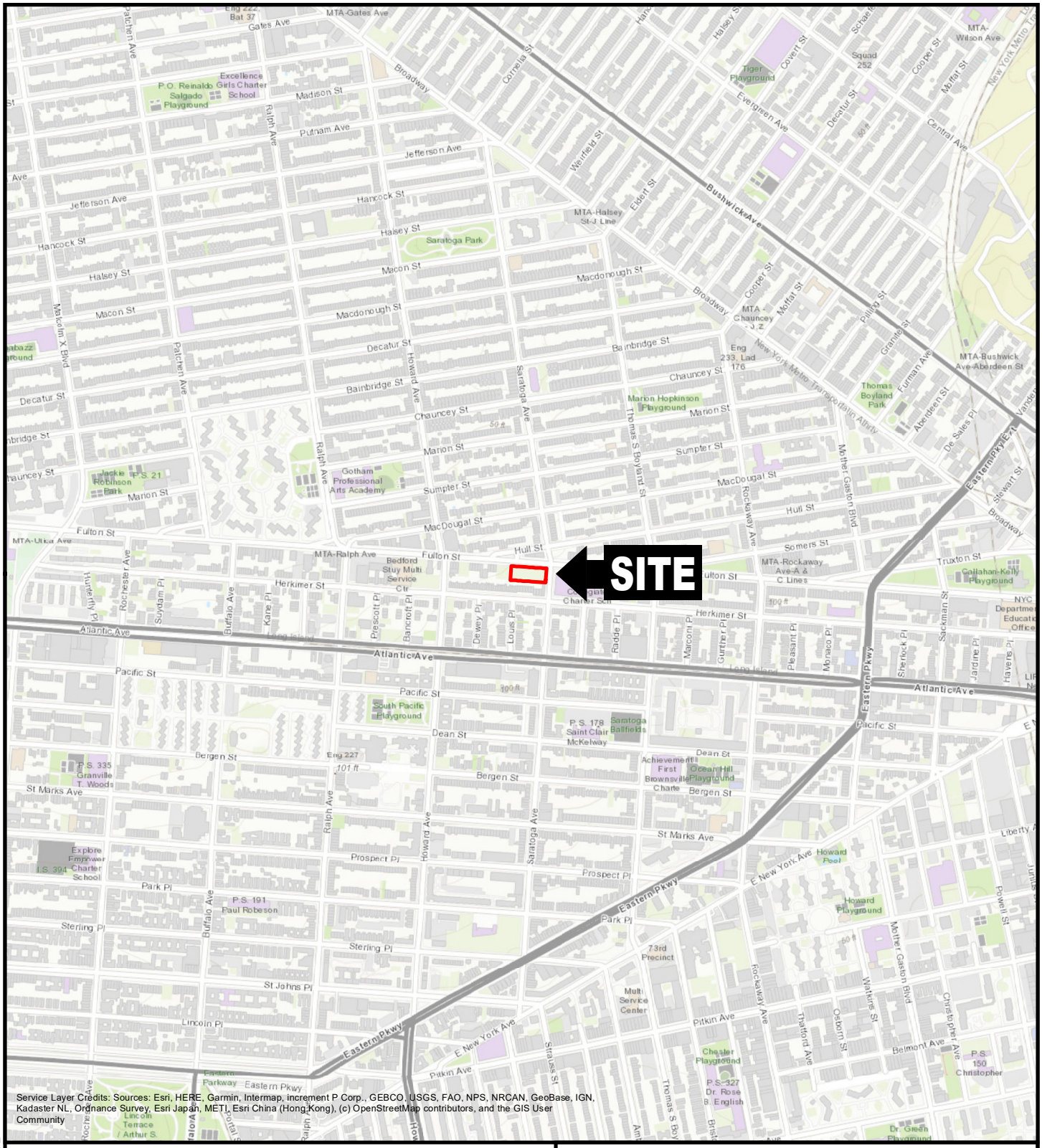
# TABLES

## (Embedded in Text)

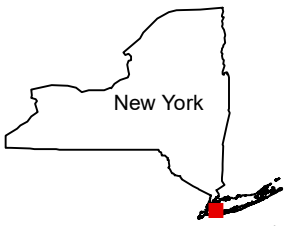
1. Proposed Soil Sample Locations, Analyses and Rationale
2. Proposed Groundwater Sample Locations, Analyses and Rationale
3. Proposed Soil Vapor Sample Locations, Analyses and Rationale

# FIGURES

1. Site Location Map
2. Limited Phase II ESA Sample Locations
3. Soil Sample Exceedances
4. Soil Vapor Detections
5a. Historic Sample Locations
5b. Proposed Sample Locations




QUADRANGLE LOCATION




N

1,000 0 1,000'

Title:

# SITE LOCATION MAP

2030 FULTON STREET
BROOKLYN, NEW YORK

Prepared for:

STEPS FAMILY LLC

ROUX

| | |
|---|---|
| Compiled by: B.H. | Date: 10/13/25 |
| Prepared by: M.S.R. | Scale: AS SHOWN |
| Project Mgr: B.H. | Project: 3805.0002Y003 |
| File: 3805.0002Y113.1.mxd | |

FIGURE 1

V:\GIS\PROJECTS\3805\0002Y\113\3805.0002Y113.1.MXD




N

LEGEND

PARCEL BOUNDARY

NOTES

1. AERIAL SOURCE: NYS OFFICE OF INFORMATION TECHNOLOGY SERVICES GIS PROGRAM OFFICE (GPO)
2. PARCEL SOURCE: DEPARTMENT OF CITY PLANNING (DCP) PLUTO DATA SET
3. THE TAX LOTS WERE ALL MERGED INTO LOT 28 ON 08/21/2025

40 0 40'

Title:

# SITE PLAN

2030 FULTON STREET
BROOKLYN, NEW YORK

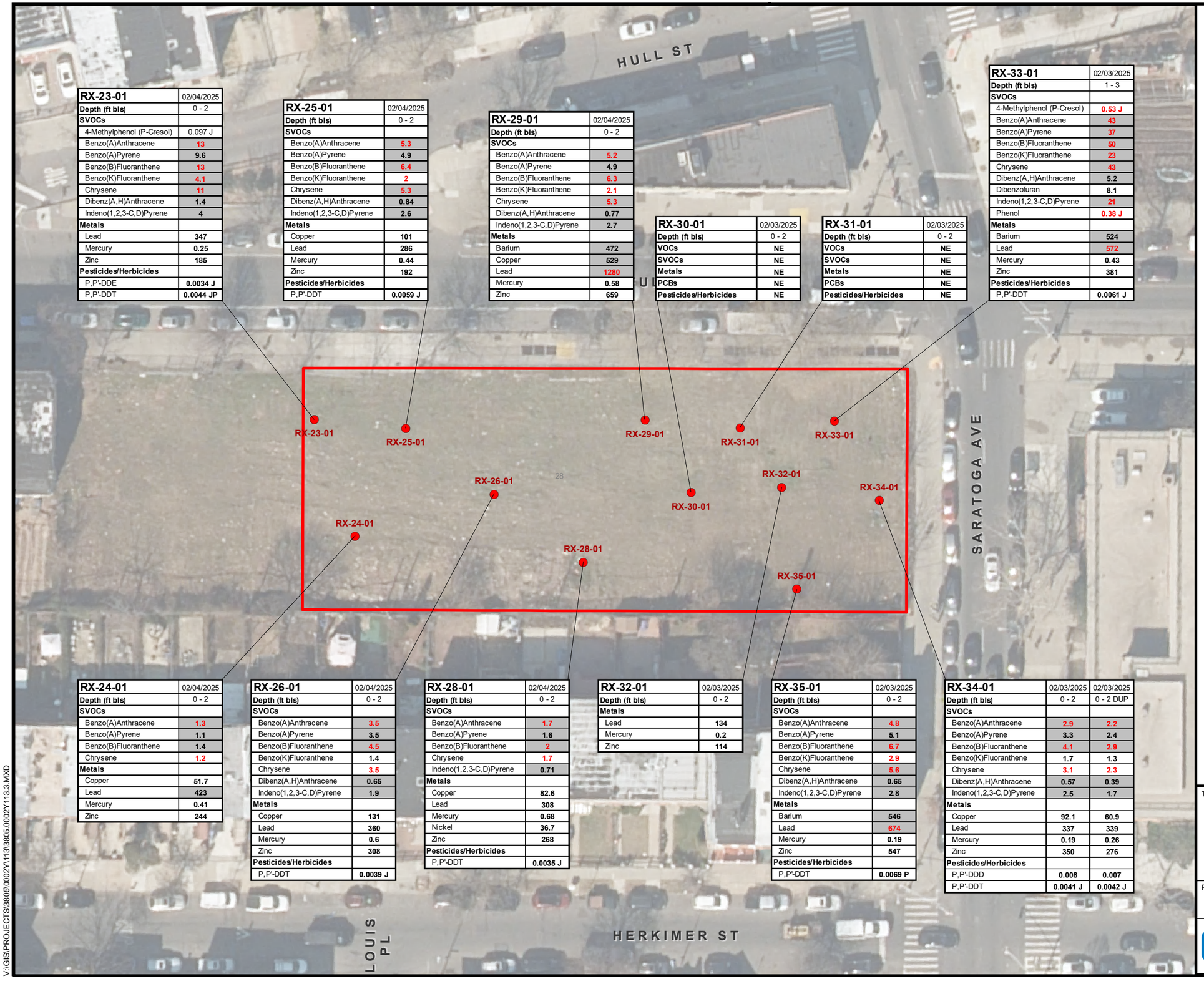
Prepared for:

STEPS FAMILY LLC

ROUX

| | | |
|---|---|---|
| Compiled by: B.H. | Date: 09/11/26 | FIGURE |
| Prepared by: M.S.R. | Scale: AS SHOWN | 2 |
| Project Mgr: B.H. | Project: 3805.0002Y003 | |
| File: 3805.0002Y113.2.mxd | | |

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| RX-23-01 | 02/04/2025 |
|---|---|
| Depth (ft bls) | 0 - 2 |
| SVOCs | |
| 4-Methylphenol (P-Cresol) | 0.097 J |
| Benzo(A)Anthracene | 13 |
| Benzo(A)Pyrene | 9.6 |
| Benzo(B)Fluoranthene | 13 |
| Benzo(K)Fluoranthene | 4.1 |
| Chrysene | 11 |
| Dibenz(A,H)Anthracene | 1.4 |
| Indeno(1,2,3-C,D)Pyrene | 4 |
| Metals | |
| Lead | 347 |
| Mercury | 0.25 |
| Zinc | 185 |
| Pesticides/Herbicides | |
| P,P'-DDE | 0.0034 J |
| P,P'-DDT | 0.0044 JP |

| RX-25-01 | 02/04/2025 |
|---|---|
| Depth (ft bls) | 0 - 2 |
| SVOCs | |
| Benzo(A)Anthracene | 5.3 |
| Benzo(A)Pyrene | 4.9 |
| Benzo(B)Fluoranthene | 6.4 |
| Benzo(K)Fluoranthene | 2 |
| Chrysene | 5.3 |
| Dibenz(A,H)Anthracene | 0.84 |
| Indeno(1,2,3-C,D)Pyrene | 2.6 |
| Metals | |
| Copper | 101 |
| Lead | 286 |
| Mercury | 0.44 |
| Zinc | 192 |
| Pesticides/Herbicides | |
| P,P'-DDT | 0.0059 J |

| RX-29-01 | 02/04/2025 |
|---|---|
| Depth (ft bls) | 0 - 2 |
| SVOCs | |
| Benzo(A)Anthracene | 5.2 |
| Benzo(A)Pyrene | 4.9 |
| Benzo(B)Fluoranthene | 6.3 |
| Benzo(K)Fluoranthene | 2.1 |
| Chrysene | 5.3 |
| Dibenz(A,H)Anthracene | 0.77 |
| Indeno(1,2,3-C,D)Pyrene | 2.7 |
| Metals | |
| Barium | 472 |
| Copper | 529 |
| Lead | 1280 |
| Mercury | 0.58 |
| Zinc | 659 |

| RX-30-01 | 02/03/2025 |
|---|---|
| Depth (ft bls) | 0 - 2 |
| VOCs | NE |
| SVOCs | NE |
| Metals | NE |
| PCBs | NE |
| Pesticides/Herbicides | NE |

| RX-31-01 | 02/03/2025 |
|---|---|
| Depth (ft bls) | 0 - 2 |
| VOCs | NE |
| SVOCs | NE |
| Metals | NE |
| PCBs | NE |
| Pesticides/Herbicides | NE |

| RX-33-01 | 02/03/2025 |
|---|---|
| Depth (ft bls) | 1 - 3 |
| SVOCs | |
| 4-Methylphenol (P-Cresol) | 0.53 J |
| Benzo(A)Anthracene | 43 |
| Benzo(A)Pyrene | 37 |
| Benzo(B)Fluoranthene | 50 |
| Benzo(K)Fluoranthene | 23 |
| Chrysene | 43 |
| Dibenz(A,H)Anthracene | 5.2 |
| Dibenzofuran | 8.1 |
| Indeno(1,2,3-C,D)Pyrene | 21 |
| Phenol | 0.38 J |
| Metals | |
| Barium | 524 |
| Lead | 572 |
| Mercury | 0.43 |
| Zinc | 381 |
| Pesticides/Herbicides | |
| P,P'-DDT | 0.0061 J |

| RX-24-01 | 02/04/2025 |
|---|---|
| Depth (ft bls) | 0 - 2 |
| SVOCs | |
| Benzo(A)Anthracene | 1.3 |
| Benzo(A)Pyrene | 1.1 |
| Benzo(B)Fluoranthene | 1.4 |
| Chrysene | 1.2 |
| Metals | |
| Copper | 51.7 |
| Lead | 423 |
| Mercury | 0.41 |
| Zinc | 244 |

| RX-26-01 | 02/04/2025 |
|---|---|
| Depth (ft bls) | 0 - 2 |
| SVOCs | |
| Benzo(A)Anthracene | 3.5 |
| Benzo(A)Pyrene | 3.5 |
| Benzo(B)Fluoranthene | 4.5 |
| Benzo(K)Fluoranthene | 1.4 |
| Chrysene | 3.5 |
| Dibenz(A,H)Anthracene | 0.65 |
| Indeno(1,2,3-C,D)Pyrene | 1.9 |
| Metals | |
| Copper | 131 |
| Lead | 360 |
| Mercury | 0.6 |
| Zinc | 308 |
| Pesticides/Herbicides | |
| P,P'-DDT | 0.0039 J |

| RX-28-01 | 02/04/2025 |
|---|---|
| Depth (ft bls) | 0 - 2 |
| SVOCs | |
| Benzo(A)Anthracene | 1.7 |
| Benzo(A)Pyrene | 1.6 |
| Benzo(B)Fluoranthene | 2 |
| Chrysene | 1.7 |
| Indeno(1,2,3-C,D)Pyrene | 0.71 |
| Metals | |
| Copper | 82.6 |
| Lead | 308 |
| Mercury | 0.68 |
| Nickel | 36.7 |
| Zinc | 268 |
| Pesticides/Herbicides | |
| P,P'-DDT | 0.0035 J |

| RX-32-01 | 02/03/2025 |
|---|---|
| Depth (ft bls) | 0 - 2 |
| Metals | |
| Lead | 134 |
| Mercury | 0.2 |
| Zinc | 114 |

| RX-35-01 | 02/03/2025 |
|---|---|
| Depth (ft bls) | 0 - 2 |
| SVOCs | |
| Benzo(A)Anthracene | 4.8 |
| Benzo(A)Pyrene | 5.1 |
| Benzo(B)Fluoranthene | 6.7 |
| Benzo(K)Fluoranthene | 2.9 |
| Chrysene | 5.6 |
| Dibenz(A,H)Anthracene | 0.65 |
| Indeno(1,2,3-C,D)Pyrene | 2.8 |
| Metals | |
| Barium | 546 |
| Lead | 674 |
| Mercury | 0.19 |
| Zinc | 547 |
| Pesticides/Herbicides | |
| P,P'-DDT | 0.0069 P |

| RX-34-01 | 02/03/2025 | 02/03/2025 |
|---|---|---|
| Depth (ft bls) | 0 - 2 | 0 - 2 DUP |
| SVOCs | | |
| Benzo(A)Anthracene | 2.9 | 2.2 |
| Benzo(A)Pyrene | 3.3 | 2.4 |
| Benzo(B)Fluoranthene | 4.1 | 2.9 |
| Benzo(K)Fluoranthene | 1.7 | 1.3 |
| Chrysene | 3.1 | 2.3 |
| Dibenz(A,H)Anthracene | 0.57 | 0.39 |
| Indeno(1,2,3-C,D)Pyrene | 2.5 | 1.7 |
| Metals | | |
| Copper | 92.1 | 60.9 |
| Lead | 337 | 339 |
| Mercury | 0.19 | 0.26 |
| Zinc | 350 | 276 |
| Pesticides/Herbicides | | |
| P,P'-DDD | 0.008 | 0.007 |
| P,P'-DDT | 0.0041 J | 0.0042 J |

| Parameter | NYSDEC Part 375 Unrestricted Use Soil Cleanup Objectives | NYSDEC Part 375 Restricted Residential Soil Cleanup Objectives | NYSDEC Part 375 Protection of Groundwater Soil Cleanup Objectives | Units |
|---|---|---|---|---|
| VOCs | NE | NE | NE | mg/kg |
| SVOCs | | | | |
| Benzo(A)Anthracene | 1 | 1 | 1 | mg/kg |
| Benzo(A)Pyrene | 1 | 1 | 22 | mg/kg |
| Benzo(B)Fluoranthene | 1 | 1 | 1.7 | mg/kg |
| Benzo(K)Fluoranthene | 0.8 | 3.9 | 1.7 | mg/kg |
| Chrysene | 1 | 3.9 | 1 | mg/kg |
| Dibenz(A,H)Anthracene | 0.33 | 0.33 | 1000 | mg/kg |
| Indeno(1,2,3-C,D)Pyrene | 0.5 | 0.5 | 8.2 | mg/kg |
| Metals | | | | |
| Copper | 50 | 270 | 1720 | mg/kg |
| Lead | 63 | 400 | 450 | mg/kg |
| Mercury | 0.18 | 0.81 | 0.73 | mg/kg |
| Zinc | 109 | 10000 | 2480 | mg/kg |
| PCBs | ND | ND | ND | mg/kg |
| Pesticides/Herbicides | | | | |
| P,P'-DDT | 0.0033 | 7.9 | 136 | mg/kg |

## LEGEND

- LOCATION OF SOIL BORING
- PROPOSED BCP SITE BOUNDARY

## NOTES

1. AERIAL SOURCE: NYS OFFICE OF INFORMATION TECHNOLOGY SERVICES GIS PROGRAM OFFICE (GPO)

MG/KG - MILLIGRAMS PER KILOGRAM
NG/KG - NANOGRAMS PER KILOGRAM
NYSDEC - NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
-- NO NYSDEC PART 375 SOIL CLEANUP OBJECTIVES AVAILABLE
J - ESTIMATED VALUE
P - CONCENTRATION DIFFERENCE BETWEEN THE PRIMARY AND CONFIRMATION COLUMN >40%. THE LOWER RESULT IS REPORTED
DUP - DUPLICATE SAMPLE
VOCS - VOLATILE ORGANIC COMPOUNDS
SVOCS - SEMIVOLATILE ORGANIC COMPOUNDS
PCBS - POLYCHLORINATED BIPHENYLS
NE - NO EXCEEDANCE
ND - NO DETECTION
FT BLS - FEET BELOW LAND SURFACE

40 0 40'

Title:

# SOIL SAMPLE EXCEEDANCES

2030 FULTON STREET
BROOKLYN, NEW YORK

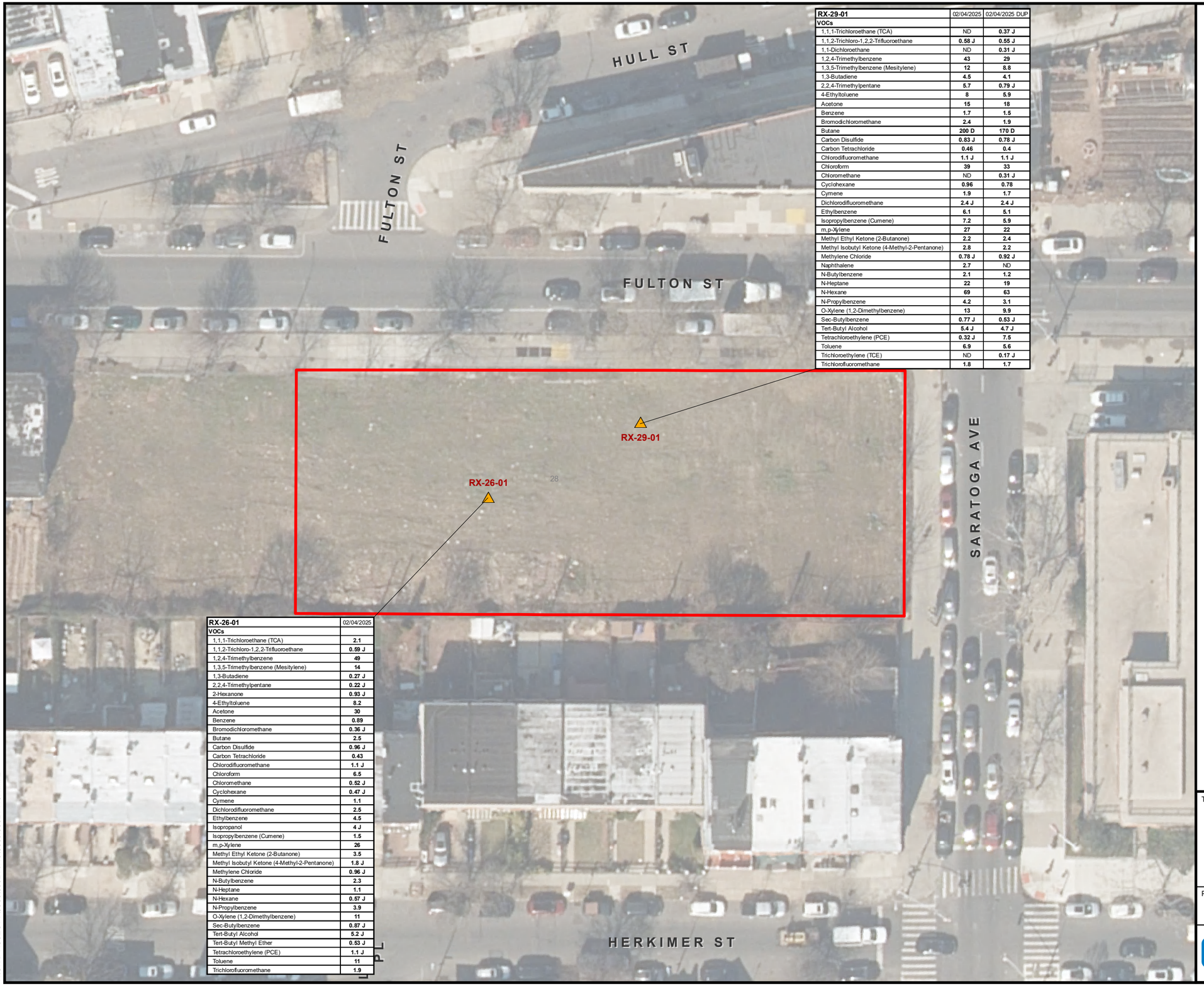
Prepared for:

STEPS FAMILY LLC

ROUX

| | | |
|---|---|---|
| Compiled by: B.H. | Date: 12/05/25 | FIGURE |
| Prepared by: M.S.R. | Scale: AS SHOWN | 3 |
| Project Mgr: B.H. | Project: 3805.0002Y003 | |
| File: 3805.0002Y113.3.mxd | | |

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| RX-29-01 | 02/04/2025 | 02/04/2025 DUP |
|---|---|---|
| VOCs | | |
| 1,1,1-Trichloroethane (TCA) | ND | 0.37 J |
| 1,1,2-Trichloro-1,2,2-Trifluoroethane | 0.58 J | 0.55 J |
| 1,1-Dichloroethane | ND | 0.31 J |
| 1,2,4-Trimethylbenzene | 43 | 29 |
| 1,3,5-Trimethylbenzene (Mesitylene) | 12 | 8.8 |
| 1,3-Butadiene | 4.5 | 4.1 |
| 2,2,4-Trimethylpentane | 5.7 | 0.79 J |
| 4-Ethyltoluene | 8 | 5.9 |
| Acetone | 15 | 18 |
| Benzene | 1.7 | 1.5 |
| Bromodichloromethane | 2.4 | 1.9 |
| Butane | 200 D | 170 D |
| Carbon Disulfide | 0.83 J | 0.78 J |
| Carbon Tetrachloride | 0.46 | 0.4 |
| Chlorodifluoromethane | 1.1 J | 1.1 J |
| Chloroform | 39 | 33 |
| Chloromethane | ND | 0.31 J |
| Cyclohexane | 0.96 | 0.78 |
| Cymene | 1.9 | 1.7 |
| Dichlorodifluoromethane | 2.4 J | 2.4 J |
| Ethylbenzene | 6.1 | 5.1 |
| Isopropylbenzene (Cumene) | 7.2 | 5.9 |
| m,p-Xylene | 27 | 22 |
| Methyl Ethyl Ketone (2-Butanone) | 2.2 | 2.4 |
| Methyl Isobutyl Ketone (4-Methyl-2-Pentanone) | 2.8 | 2.2 |
| Methylene Chloride | 0.78 J | 0.92 J |
| Naphthalene | 2.7 | ND |
| N-Butylbenzene | 2.1 | 1.2 |
| N-Heptane | 22 | 19 |
| N-Hexane | 69 | 63 |
| N-Propylbenzene | 4.2 | 3.1 |
| O-Xylene (1,2-Dimethylbenzene) | 13 | 9.9 |
| Sec-Butylbenzene | 0.77 J | 0.53 J |
| Tert-Butyl Alcohol | 5.4 J | 4.7 J |
| Tetrachloroethylene (PCE) | 0.32 J | 7.5 |
| Toluene | 6.9 | 5.6 |
| Trichloroethylene (TCE) | ND | 0.17 J |
| Trichlorofluoromethane | 1.8 | 1.7 |

| RX-26-01 | 02/04/2025 |
|---|---|
| VOCs | |
| 1,1,1-Trichloroethane (TCA) | 2.1 |
| 1,1,2-Trichloro-1,2,2-Trifluoroethane | 0.59 J |
| 1,2,4-Trimethylbenzene | 49 |
| 1,3,5-Trimethylbenzene (Mesitylene) | 14 |
| 1,3-Butadiene | 0.27 J |
| 2,2,4-Trimethylpentane | 0.22 J |
| 2-Hexanone | 0.93 J |
| 4-Ethyltoluene | 8.2 |
| Acetone | 30 |
| Benzene | 0.89 |
| Bromodichloromethane | 0.36 J |
| Butane | 2.5 |
| Carbon Disulfide | 0.96 J |
| Carbon Tetrachloride | 0.43 |
| Chlorodifluoromethane | 1.1 J |
| Chloroform | 6.5 |
| Chloromethane | 0.52 J |
| Cyclohexane | 0.47 J |
| Cymene | 1.1 |
| Dichlorodifluoromethane | 2.5 |
| Ethylbenzene | 4.5 |
| Isopropanol | 4 J |
| Isopropylbenzene (Cumene) | 1.5 |
| m,p-Xylene | 26 |
| Methyl Ethyl Ketone (2-Butanone) | 3.5 |
| Methyl Isobutyl Ketone (4-Methyl-2-Pentanone) | 1.8 J |
| Methylene Chloride | 0.96 J |
| N-Butylbenzene | 2.3 |
| N-Heptane | 1.1 |
| N-Hexane | 0.57 J |
| N-Propylbenzene | 3.9 |
| O-Xylene (1,2-Dimethylbenzene) | 11 |
| Sec-Butylbenzene | 0.87 J |
| Tert-Butyl Alcohol | 5.2 J |
| Tert-Butyl Methyl Ether | 0.53 J |
| Tetrachloroethylene (PCE) | 1.1 J |
| Toluene | 11 |
| Trichlorofluoromethane | 1.9 |

N

LEGEND

- LOCATION OF SOIL VAPOR POINT
- PROPOSED BCP SITE BOUNDARY

NOTES

1. AERIAL SOURCE: NYS OFFICE OF INFORMATION TECHNOLOGY SERVICES GIS PROGRAM OFFICE (GPO)
2. CONCENTRATIONS IN µG/M$^3$

µG/M$^3$ - MICROGRAMS PER CUBIC METER
VOCS - VOLATILE ORGANIC COMPOUNDS
ND - COMPOUND WAS ANALYZED FOR BUT NOT DETECTED
J - ESTIMATED VALUE
D - CONCENTRATION OF ANALYTE WAS QUANTIFIED FROM DILUTED ANALYSIS. FLAG ONLY APPLIES TO FIELD SAMPLES THAT HAVE DETECTABLE CONCENTRATIONS OF THE ANALYTE

40 0 40'

Title:

# SOIL VAPOR DETECTIONS

2030 FULTON STREET
BROOKLYN, NEW YORK

Prepared for:

STEPS FAMILY LLC

ROUX

| | | |
|---|---|---|
| Compiled by: B.H. | Date: 11/06/25 | FIGURE |
| Prepared by: M.S.R. | Scale: AS SHOWN | 4 |
| Project Mgr: B.H. | Project: 3805.0002Y003 | |
| File: 3805.0002Y113.4.mxd | | |

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N

LEGEND

- LOCATION OF PREVIOUS SOIL BORING
- LOCATION OF PREVIOUS SOIL VAPOR POINT
- PROPOSED BCP SITE BOUNDARY
- PARCEL BOUNDARY

NOTES

1. AERIAL SOURCE: NYS OFFICE OF INFORMATION TECHNOLOGY SERVICES GIS PROGRAM OFFICE (GPO)
2. PARCEL SOURCE: DEPARTMENT OF CITY PLANNING (DCP) PLUTO DATA SET

40 0 40'

Title:

# HISTORIC SAMPLE LOCATIONS

2030 FULTON STREET
BROOKLYN, NEW YORK

Prepared for:

STEPS FAMILY LLC

ROUX

| | | |
|---|---|---|
| Compiled by: B.H. | Date: 09/11/26 | FIGURE |
| Prepared by: M.S.R. | Scale: AS SHOWN | 5A |
| Project Mgr: B.H. | Project: 3805.0002Y003 | |
| File: 3805.0002Y113.5A.mxd | | |

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N

LEGEND

- LOCATION OF PROPOSED SOIL BORING
- LOCATION OF PROPOSED SOIL BORING AND SOIL VAPOR POINT
- LOCATION OF PROPOSED SOIL BORING AND GROUNDWATER MONITORING WELL
- PROPOSED BCP SITE BOUNDARY
- PARCEL BOUNDARY

NOTES

1. AERIAL SOURCE: NYS OFFICE OF INFORMATION TECHNOLOGY SERVICES GIS PROGRAM OFFICE (GPO)
2. PARCEL SOURCE: DEPARTMENT OF CITY PLANNING (DCP) PLUTO DATA SET

40 0 40'

Title:

# PROPOSED SAMPLE LOCATIONS

2030 FULTON STREET
BROOKLYN, NEW YORK

Prepared for:

STEPS FAMILY LLC

ROUX

| | | |
|---|---|---|
| Compiled by: B.H. | Date: 09/29/26 | FIGURE |
| Prepared by: M.S.R. | Scale: AS SHOWN | 5B |
| Project Mgr: B.H. | Project: 3805.0002Y003 | |
| File: 3805.0002Y113.5B.mxd | | |

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# APPENDICES

A. Historical Reports
B. Quality Assurance Project Plan/Field Sampling Plan
C. Site Specific Health and Safety Plan
D. Community Air Monitoring Plan

# APPENDIX A

Historical Reports
(Submitted Under Separate Cover)

# APPENDIX B

Quality Assurance Project Plan/Field Sampling Plan

# Quality Assurance Project Plan/Field Sampling Plan (QAPP/FSP)

2030 Fulton Street
Brooklyn, New York

September 30, 2026

Prepared for:

**Steps Family LLC**
**c/o Riseboro Community Partnership**
217 Wyckoff Avenue
Brooklyn, New York 11237

Prepared by:

**Roux Environmental Engineering and Geology, D.P.C.**
209 Shafter Street
Islandia, New York 11749

3805.0002Y114/CV

Environmental Consulting
& Management
+1.800.322.ROUX
rouxinc.com

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1. Professional Profiles
2. Laboratory's Standard Operating Procedures
3. Roux's Standard Operating Procedures
4. NYSDEC PFAS Sampling Guidance

# 1. Introduction

Roux Environmental Engineering and Geology, D.P.C. (Roux), on behalf of Steps Senior LLC (Steps Senior), has prepared this Quality Assurance Project Plan/Field Sampling Plan (QAPP/FSP) to describe the measures that will be taken to ensure that the data generated during performance of the Remedial Investigation (RI) for the property located at 2030 Fulton Street, Brooklyn, New York (Site) are of quality sufficient to meet project-specific data quality objectives (DQOs). This QAPP/FSP also includes field sampling procedures.

This QAPP/FSP was prepared in accordance with the guidance provided in NYSDEC Technical Guidance DER-10 Technical Guidance for Site Investigation and Remediation (DER-10), the NYSDEC Brownfield Cleanup Program (BCP) Guide, and the United States Environmental Protection Agency's (USEPA's) Guidance for the Data Quality Objectives Process (EPA QA/G 4).

## 1.1 Purpose

The QAPP/FSP describes in detail the field sampling and quality assurance/quality control (QA/QC) methods to be used during soil, soil vapor, and groundwater sampling tasks performed during the RI.

This QAPP/FSP provides guidelines and procedures to be followed by field personnel during performance of sampling during the RI. Information contained in this QAPP/FSP relates to:

- Sampling objectives (Section 2);
- Project organization (Section 3);
- Sample media, sampling locations, analytical suites, sampling frequencies and analytical laboratory (Section 4);
- Field sampling procedures (Section 5);
- Sample handling, sample analysis, and quality assurance/quality control (Section 6); and
- Site control procedures and decontamination (Section 7).

# 2. Sampling Objectives

The objective of the RI is to further assess the nature and extent of contamination associated with the following AOCs:

- Former Site and surrounding properties operations; and
- The presence of historical fill at the Site.

An inspection of the existing Site conditions will be conducted to determine final locations of soil borings, monitoring wells, and vapor sampling points based on actual field conditions.

Sampling procedures are discussed in Section 5 of this QAPP/FSP. A discussion of the DQOs and quality assurance/quality control is provided in Section 6.

# 3. Project Organization

A general and generic summary of the overall management structure and responsibilities of project team members are presented below. Professional profiles for the team are provided in Attachment 1.

**Project Principal**

Jessica Taylor, P.G. will serve as Project Principal. The Project Principal is responsible for defining project objectives and bears ultimate responsibility for the successful completion of the investigation.

**Project Manager**

Brooke Hildebrand will serve as Project Manager. The Project Manager will provide overall management for the implementation of the scope of work and will coordinate all field activities. The Project Manager is also responsible for data review/interpretation and report preparation.

**Field Team Leader**

Benjamin Weintraub will serve as the Field Team Leader. The Field Team Leader bears the responsibility for the successful execution of the field program. The Field Team Leader will direct the activities of the technical staff in the field, as well as all subcontractors. The Field Team Leader will also assist in the interpretation of data and in report preparation. The Field Team Leader reports to the Project Manager.

**Laboratory Project Manager**

Elizabeth Flannery will serve as the Laboratory Project Manager. The Laboratory Project Manager is responsible for sample container preparation, sample custody in the laboratory, and completion of the required analysis through oversight of the laboratory staff. The Laboratory Project Manager will ensure that quality assurance procedures are followed and that an acceptable laboratory report is prepared and submitted. The Laboratory Project Manager reports to the Project Principal and Project Manager.

**Quality Assurance Officer**

Brandon Vella will serve as the Quality Assurance Officer (QAO). The QAO is responsible for conducting reviews, inspections, and audits to ensure that the data collection is conducted in accordance with the FSP and QAPP. The QAO's responsibilities range from ensuring effective field equipment decontamination procedures and proper sample collection to the review of all laboratory analytical data for completeness and usefulness. The QAO reports to the Project Manager and makes independent recommendations to the Field Team Leader.

# 4. Sample Media, Locations, Analytical Suites, and Frequency

The media to be sampled during the RI include soil, groundwater, and soil vapor. Sampling locations, analytical suites, and frequency may vary by medium. The scope of the RI will include the collection of sufficient Site investigation data so that, together with the historical data, the entire Site will be sufficiently characterized to determine BCP eligibility as well as support the development of the Site-wide Remedial Action Work Plan (RAWP).

A discussion of the sampling schedule for each medium is provided below, while the assumed number of field samples to be collected for each medium, including quality control (QC) samples, is shown in Tables 1 and 2. Specifics regarding the collection of samples at each location and for each task are provided in Section 5 of this QAPP/FSP.

## 4.1 Soil Sampling

Soil samples are to be used to characterize the soil conditions for the AOCs at the Site, and to collect the data sufficient to define the nature and extent of impacted soils. A total of 44-55 soil samples from eleven soil borings will be collected in a manner consistent with this QAPP/FSP. As part of the RI Work Plan (RIWP), six soil borings are proposed to be advanced at the locations shown in Figure 1 (Figure 5 of the RIWP).

The summary table below provides details for the soil sampling locations that are proposed as part of the RIWP:

**Table 1 – Proposed Soil Sample Locations, Analyses, and Rationale**

| Soil Boring Location | Sample Depth Interval (ft bls) | Analytical Suites | Rationale |
|---|---|---|---|
| RXS-01<br>RXS-02<br>RXS-03<br>RXS-04 | ▪ 0-0.5 ft bls<br>▪ Most impacted 2 ft interval, if applicable*<br>▪ Bottom 2 ft of Fill<br>▪ Clean 2 ft Interval Beneath Impacts or Fill, whichever is deeper<br>▪ 2 ft above Groundwater Table | Full Part 375** and ECs*** | Evaluate soil conditions on Site and determine extent of contamination both vertically to the groundwater interface and horizontally across the Site. |
| RXS-05<br>RXS-06<br>RXS-07<br>RXS-08<br>RXS-09<br>RXS-10<br>RXS-11 | ▪ 0-0.5 ft bls<br>▪ Most impacted 2 ft interval, if applicable*<br>▪ Bottom 2 ft of Fill<br>▪ Clean 2ft Interval Beneath Impacts or Fill<br>▪ Soil Boring Termination Depth | Full Part 375 and ECs | Evaluate soil conditions on Site and determine extent of contamination both vertically to the historical fill/native soil interface and horizontally across the Site. |

* Note: If applicable, a sample will be collected from the most impacted interval as determined by field observations (e.g. staining and odors). If no impacts are observed, then a sample will not be collected.

** Note: Part 375/TCL plus 30/ TAL (TCL + 30/TAL) includes:

- Part 375/TCL VOCs + 10 TICs;
- Part 375/TCL Base Neutral Acids (BNA)/ SVOCs + 20 TICs;
- Part 375/TCL Pesticides;
- Part 375/TCL Herbicides;
- Part 375/TCL PCBs;
- Part 375/TAL Metals (including hexavalent chromium); and
- Total Cyanide.

***Note: Emerging Contaminants *(*ECs) list includes 1,4-Dioxane and the Per- and Polyfluoroalkyl Substances (PFAS) compounds listed in the NYSDEC April 2023 "Sampling, Analysis, and Assessment of Per-And Polyfluoroalkyl Substances (PFAS)". PFAS in soil will be analyzed by USEPA Method 1633A and 1,4-Dioxane in soil will be analyzed by USEPA Method 8270D.

If odor/ visual evidence of contamination or elevated photoionization detector (PID) readings are noted, additional samples will be collected from the interval that exhibits the highest contamination.

## 4.2 Groundwater Sampling

To evaluate groundwater quality conditions across the Site, four permanent monitoring wells (RXS-01 through RXS-04) will be installed in converted soil boring locations. Roux will collect a groundwater sample from each newly installed monitoring well location totaling four samples, as shown on Figure 1 (Figure 5 of the RIWP). All monitoring wells will be surveyed for horizontal coordinates, land surface elevation and measuring point elevation.

Monitoring wells will be constructed of 2-inch inside diameter, Schedule 40 polyvinyl chloride (PVC) casing and, 0.020 inch slot, machined screen. Well construction will be as follows:

- Well screens will be 10 feet long, and will be installed with three feet above and seven feet below the water table.
- A sand pack will be placed around the well screen, extending two feet above the top of the screened zone.
- A minimum two foot thick bentonite pellet seal will be placed above the sand pack. Once the pellets have been allowed to hydrate, cement bentonite grout will be pumped into the remaining annular space from the bottom up.
- The wells will be completed using locking well plugs, and flush mounted, bolt down, watertight, manhole covers cemented into place.

Each monitoring well to be sampled will be developed to ensure proper hydraulic connection with the aquifer and to reduce/eliminate turbidity of the groundwater. The wells will be developed using a submersible pump, which will be surged periodically until well yield is consistent and has turbidity below 50 nephelometric turbidity units (NTUs), if possible. Detailed procedures regarding well development can be found in the QAPP/FSP (Appendix B).

Prior to sampling, the depth to groundwater in each monitoring well will be measured using an electronic water level meter and a groundwater contour map will be developed using the survey data. At least one week following redevelopment, all on-Site wells will then be sampled consistent with USEPA low-flow sampling requirements using a peristaltic pump and one round of groundwater samples will be collected in accordance with the QAPP/FSP. Table 2 provides a listing of the groundwater sampling locations, screened intervals, analytical suites and rationale for sampling. Field parameters (e.g., pH, dissolved

oxygen, oxidation-reduction potential [ORP], etc.) will also be collected using a water quality meter during purging and prior to sampling.

The summary table below provides details for the groundwater sampling locations that are proposed as part of the RIWP:

**Table 2. Proposed Groundwater Sample Locations, Analyses and Rationale**

| Monitoring Well Location | Sample Depth Interval (ft bls) | Analytical Suites | Rationale |
|---|---|---|---|
| RXS-01 | 75-85 (dependent on where groundwater is observed) | Full Part 375* and ECs** | Evaluate groundwater conditions on Site |
| RXS-02 | 75-85 (dependent on where groundwater is observed) | Full Part 375 and ECs | Evaluate groundwater conditions on Site |
| RXS-03 | 75-85 (dependent on where groundwater is observed) | Full Part 375 and ECs | Evaluate groundwater conditions on Site |
| RXS-04 | 75-85 (dependent on where groundwater is observed) | Full Part 375 and ECs | Evaluate groundwater conditions on Site |

* Note: Part 375/TCL plus 30/ TAL (TCL + 30/TAL) includes:

- Part 375/TCL VOCs + 10 TICs;
- Part 375/TCL Base Neutral Acids (BNA)/ SVOCs + 20 TICs;
- Part 375/TCL Pesticides;
- Part 375/TCL Herbicides;
- Part 375/TCL PCBs;
- Part 375/TAL Metals (including hexavalent chromium); and
- Total Cyanide.

**Note: Emerging Contaminants (ECs) list includes 1,4-Dioxane and the Per- and Polyfluoroalkyl Substances (PFAS) compounds listed in the NYSDEC April 2023 "Sampling, Analysis, and Assessment of Per-And Polyfluoroalkyl Substances (PFAS)". PFAS in groundwater will be analyzed by USEPA Method 1633A and 1,4-Dioxane in groundwater will be analyzed by USEPA Method 8270D SIM.

## 4.3 Soil Vapor Sampling

To evaluate soil vapor and the potential for soil vapor intrusion (SVI) into the proposed building, Roux will install six soil vapor points (RXS-05 through RXS-10) in converted soil boring locations by hand or by using a Geoprobe® direct-push rig to the target depth of fifteen ft bls to represent the approximate depth of excavation for the building cellar. One soil vapor sample will be collected from each point. New Teflon®-lined tubing will be attached to an expendable soil vapor sampling point with a 6-inch stainless steel screen. The soil vapor points will be backfilled with #2 Morie sand to approximately one foot above the screen. The remainder of the borehole will be backfilled with a concrete/bentonite slurry to grade.

In accordance with the 2006 NYSDOH Guidance for Evaluating SVI and its updates, the integrity of each soil vapor sampling point seal will be checked following installation to verify that the soil vapor sample will not be compromised by inadvertent introduction of ambient air into the sample. The soil vapor samples will be collected using pre-cleaned (batch certified) 6-liter summa canisters with regulators calibrated to collect samples over a 2-hour period and analyzed using USEPA Method TO-15 for VOCs and methane. The proposed soil vapor sampling locations are shown on Figure 1 (Figure 5 of the RIWP. Additional details regarding soil vapor sampling methods are provided in the QAPP/FSP (Appendix B).

The summary table below provides details for the soil vapor sampling locations that are proposed as part of the RIWP:

**Table 3. Proposed Soil Vapor Sample Locations, Analyses and Rationale**

| Soil Vapor Point Location | Sample Depth Interval (ft bls) | Analytical Suites | Rationale |
|---|---|---|---|
| RXS-05 | 14.5-15 | VOCs by TO-15 and methane | Evaluate soil vapor conditions on Site |
| RXS-06 | 14.5-15 | VOCs by TO-15 and methane | Evaluate soil vapor conditions on Site |
| RXS-07 | 14.5-15 | VOCs by TO-15 and methane | Evaluate soil vapor conditions on Site |
| RXS-09 | 14.5-15 | VOCs by TO-15 and methane | Evaluate soil vapor conditions on Site |
| RXS-10 | 14.5-15 | VOCs by TO-15 and methane | Evaluate soil vapor conditions on Site |
| RXS-11 | 14.5-15 | VOCs by TO-15 and methane | Evaluate soil vapor conditions on Site |

# 5. Field Sampling Procedures

This section provides a detailed discussion of the field procedures to be used during sampling of the various media being evaluated as part of the RI (i.e., soil, groundwater, and soil vapor). As discussed, the sample locations are shown on Figure 1 and additional information including intervals to be sampled and sample rationale is provided in the RIWP. Additional details regarding sampling procedures and protocols are described in Roux's relevant Standard Operation Procedures (SOP)s, which are provided in Attachment 3.

## 5.1 Soil Sampling

Details for the collection of soil samples are provided below. At each borehole location utility clearances will be performed prior to advancement of soil borings using hand tools and/or vacuum excavation to a minimum of five feet below land surface (ft bls) at each location to confirm that no subsurface utilities were present. Should a utility or other feature be observed during pre-clearance activities, the sampling location will be relocated.

Soil borings will be advanced using a sonic drill rig or Geoprobe direct-push drill rig. Samples of the soil profile will be collected continuously from land surfance to a maximum depth of approximately ten feet below the water table for borings with colocated groundwater monitoring wells, or 17 bls for borings with colocated soil vapor points.

The soil from each five foot interval will be observed for lithology and evidence of contamination (e.g., staining, odors, and/or visible free product) and placed immediately thereafter into large Ziploc®bags for recording headspace using a PID. After a minimum of 15 minutes for equilibration with the headspace in the Ziploc® bag, each sample will be screened for organic vapors using a PID equipped with a 10.6 eV lamp. Samples for possible VOC analysis will be placed in a laboratory-supplied jar or encore sampler prior to screening, due to the potential for loss of VOCs through volatilization. Soil samples will be collected according to in Section 4. These samples will be placed in the laboratory-supplied containers and shipped to the laboratory under chain of custody procedures in accordance with Roux's SOPs in Attachment 3.

Soil samples will also be analyzed for the NYSDEC-required ECs. Additional necessary precautions will be taken when sampling for ECs in the field including, but not limited to:

- Using the proper field clothing or personal protective equipment (i.e., no materials will contain Gore-Tex or Tyvek);
- Avoid using sampling equipment components/containers making contact with aluminum foil, low density polyethylene (LDPE), glass, or polytetrafluoroethylene materials;
- Following PFAS field sampling guidelines (i.e., using sampling materials made from high density polyethylene [HDPE], silicon, or stainless steel and avoid using equipment containing Teflon and using sharpies, permanent markers, adhesives, and waterproof/plastic clipboards and notebooks); and
- Utilizing regular ice for sample presevation and only "PFAS-free" water and Alconox for decontamination.

Contaminated soil cuttings, if encountered, will be placed in sealed and labeled U.S. Department of Transportation (DOT) approved 55-gallon drums pending characterization and off-site disposal at a permitted facility.

## 5.2 Groundwater Sampling

Prior to sampling, depth to water will be measured at each well using an electronic water level meter with an accuracy of +/ 0.01 feet. All wells will then be purged and sampled using a submersible pump or low-flow method, or an alternative method, depending on the observed depth to groundwater and logistical issues. Purging and sampling will be performed consistent with USEPA low-flow sampling requirements. Field parameters (i.e., pH, dissolved oxygen, ORP, etc., as described in the USEPA low-flow sampling requirements) will be collected using a water quality meter with flow-through cell until parameters stabilize before samples are collected. Samples will be analyzed for TCL + 30/TAL as shown on Table 2.

Monitoring wells will also be analyzed for same parameters listed in Section 4.1. Similarly to the collection of soil samples for ECs, additional necessary precautions will be taken when groundwater sampling for ECs in the field including, but not limited to:

- Using the proper field clothing or personal protective equipment (i.e., no materials will contain Gore-Tex or Tyvek);
- Avoid using sampling equipment components/containers making contact with aluminum foil, low density polyethylene (LDPE), glass, or polytetrafluoroethylene materials;
- Following PFAS field sampling guidelines (i.e., using sampling materials made from high density polyethylene [HDPE], silicon, or stainless steel and avoid using equipment containing Teflon and using sharpies, permanent markers, adhesives, and waterproof/plastic clipboards and notebooks); and
- Utilizing regular ice for sample presevation and only “PFAS-free” water and Alconox for decontamination.

All groundwater samples will be collected and placed in the laboratory-supplied containers and shipped to the laboratory under chain of custody procedures in accordance with Roux’s field sampling SOPs included as Attachment 3.

## 5.3 Soil Vapor Sampling

Six soil vapor samples will be collected during the RI to evaluate soil vapor concentrations at the Site. The soil vapor samples will be collected from a depth of 15 ft bls, which is representative of the proposed building cellar excavation depth. New Teflon® lined tubing will be attached to an expendable soil vapor sampling point with a 6-inch stainless steel screen inside the rods, to prevent infiltration of ambient air. The soil vapor points will be backfilled with #2 Morie sand to approximately one foot above the screen. The remainder of the borehole will be backfilled with a cement/bentonite slurry to grade.

Prior to sample collection, the Teflon®-lined tubing will be purged of approximately two volumes of the tubing using a vacuum pump set at a rate of 0.2 liters per minute. A tracer gas (i.e., helium) will be used to enrich the atmosphere in the immediate vicinity of the sampling location in order to test the borehole seal and verify that ambient air is not being drawn into the sample in accordance with the procedures outlined in the 2006 NYSDOH Guidance for Evaluating SVI and its updates. Following purging and verification with the tracer gas, the tubing will be connected to the pre-cleaned (batch-certified) laboratory supplied summa canister. All soil vapor samples will be collected using the canisters with regulators calibrated to collect samples over a 2-hour period and analyzed using USEPA Method TO-15 for VOCs and USEPA Method 18 (Modified) for methane.

# 6. Sample Handling and Analysis

To ensure quality data acquisition and collection of representative samples, there are selective procedures to minimize sample degradation or contamination. These include procedures for preservation of the samples, as well as sample packaging, shipping procedures, and QA/QC.

## 6.1 Field Sample Handling

A discussion of the proposed number and types of samples to be collected during each task, as well as the analyses to be performed, can be found in Section 4 of this QAPP/FSP. The types of containers, volumes, and preservation techniques for the aforementioned testing parameters are presented in Table 3.

## 6.2 Sample Custody Documentation

The purpose of documenting sample custody is to ensure that the integrity and handling of the samples is not subject to question. Sample custody will be maintained from the point of sampling through the analysis (and return of unused sample portion, if applicable).

Each individual collecting samples is personally responsible for the care and custody of the samples. All sample labels should be pre printed or filled out using waterproof ink. The technical staff will review all field activities with the Field Team Leader to determine whether proper custody procedures were followed during the field work and to decide if additional samples are required.

All samples being shipped offsite for analysis must be accompanied by a properly completed chain of custody form. The sample numbers will be listed on the chain of custody form. When transferring the possession of samples, individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to/from a secure storage area, and to the laboratory.

Samples will be packaged for shipment and dispatched to the appropriate laboratory for analysis with a separate signed custody record enclosed in each sample box or cooler. Shipping containers will be locked and/or secured with strapping tape in at least two locations for shipment to the laboratory.

## 6.3 Sample Shipment

If sample shipment is necessary, sample packaging and shipping procedures are based upon USEPA specifications, as well as DOT regulations. The procedures vary according to potential sample analytes, concentration, and matrix and are designed to provide optimum protection for the samples and the public. Sample packaging and shipment must be performed using the general outline described below.

All samples will be shipped within 24 hours of collection and will be preserved appropriately from the time of sample collection. A description of the sample packing and shipping procedures is presented below:

1. Prepare cooler(s) for shipment:
    - tape drain(s) of cooler shut;
    - affix “This Side Up” arrow labels and “Fragile” labels on each cooler; and
    - place mailing label with laboratory address on top of cooler(s).
2. Arrange sample containers in groups by sample number.

3. Ensure that all bottle labels are completed correctly. Place clear tape over bottle labels to prevent moisture accumulation from causing the label to peel off.
4. Arrange containers in front of assigned coolers.
5. Place packaging material approximately at the bottom of the cooler to act as a cushion for the sample containers.
6. Arrange containers in the cooler so that they are not in contact with the cooler or other samples.
7. Fill remaining spaces with packaging material.
8. Ensure all containers are firmly packed in packaging material.
9. If ice is required to preserve the samples, ice cubes should be repackaged in Ziploc® bags and placed on top of the packaging material.
10. Sign chain of custody form (or obtain signature) and indicate the time and date it was relinquished to courier as appropriate.
11. Separate chain of custody forms. Seal proper copies within a large Ziploc® bag and tape to inside cover of cooler. Retain copies of all forms.
12. Close lid and latch.
13. Secure each cooler using custody seals.
14. Tape cooler shut on both ends.
15. Relinquish to overnight delivery service as appropriate. Retain air bill receipt for project records. (Note: All samples will be shipped for "NEXT A.M." delivery).

## 6.4 Quality Assurance/Quality Control

The primary intended use for the RI data is to characterize Site conditions, determine BCP eligibility, and aid in the preparation of the RAWP. The primary DQO of the soil, groundwater, and soil vapor programs, therefore, is that data be accurate and precise, and hence representative of the actual Site conditions. Accuracy refers to the ability of the laboratory to obtain a true value (i.e., compared to a standard) and is assessed through the use of laboratory quality control (QC) samples, including laboratory control samples and matrix spike samples, as well as through the use of surrogates, which are compounds not typically found in the environment that are injected into the samples prior to analysis. Precision refers to the ability to replicate a value and is assessed through both field and laboratory duplicate samples.

Sensitivity is also a critical issue in generating representative data. Laboratory equipment must be of sufficient sensitivity to detect target compounds and analytes at levels below NYSDEC standards and guidelines whenever possible. Equipment sensitivity can be decreased by field or laboratory contamination of samples, and by sample matrix effects. Assessment of instrument sensitivity is performed through the analysis of reagent blanks, near-detection-limit standards, and response factors. Potential field and/or laboratory contamination is assessed through use of trip blanks, method blanks, and equipment rinse blanks (also called "field blanks"). Field blanks for PFAS will be collected at a minimum frequency of one per day. A laboratory SOP for analysis of PFAS is included in Attachment 2.

Table 1 lists the requirements for field and laboratory QC samples that will be analyzed to assess data accuracy and precision, as well as to determine if equipment sensitivity has been compromised. Table 2 lists the number/type of field and QA/QC samples that will be collected during the RI. Table 3 lists the preservation, holding times and sample container information.

All RI “assessment” analyses (i.e., TCL plus 30/ TAL and TO-15) will be performed in accordance with the NYSDEC Analytical Services Protocol (ASP), using USEPA SW 846 methods.

All laboratory data are to be reported in NYSDEC ASP Category B deliverables and will be delivered to NYSDEC in electronic data deliverable (EDD) format as described on NYSDEC’s website (http://www.dec.ny.gov/chemical/62440.html). A Data Usability Summary Report (DUSR) will be prepared meeting the requirements in Section 2.2(a)1.ii and Appendix 2B of DER-10 for all data packages generated for the RI. Judy Harry of Data Validation Services will review the analytical data for quality assurance and quality control in accordance with NYSDEC standards. The professional profile for Judy Harry is provided in Attachment 1.

# 7. Site Control Procedures

Site control procedures, including decontamination and waste handling and disposal, are discussed below. Site control procedures have been developed to minimize both the risk of exposure to contamination and the spread of contamination during field activities at the Site. All personnel who come into designated work areas, including contractors and observers, will be required to adhere strictly to the conditions imposed herein and to the provisions of a Site-Specific Health and Safety Plan (HASP). The HASP is included as Appendix D to the RIWP.

## 7.1 Decontamination

In an attempt to avoid the spread of contamination, all drilling and sampling equipment must be decontaminated at a reasonable frequency in a properly designed and located decontamination area. Detailed procedures for the decontamination of field and sampling equipment are included in Roux's SOPs for the Decontamination of Field Equipment located in Attachment 3. The location of the decontamination area will be determined prior to the start of field operations. The decontamination area will be constructed to ensure that all wash water generated during decontamination can be collected and containerized for proper disposal. Only "PFAS-free" water will be used for decontamination of sample equipment onsite. Only Alconox will be used as decontamination detergent (Liquinox shall not be used).

## 7.2 Waste Handling and Disposal

All waste materials (drill cuttings, decontamination water, etc.) generated during the RI will be consolidated, and stored in appropriate labeled bulk containers (drums, etc.) with labels indicating pending analysis as a hazardous waste material. The drums will be temporarily staged on protective sheeting in an investigation derived waste storage area onsite that will be covered for protection from the elements. Roux will then coordinate waste characterization and disposal by appropriate means.

# TABLES

1. Field and Laboratory QC Summary
2. Remedial Investigation Sampling Summary
3. Preservation, Holding Times, and Sample Containers

**Table 1. Field and Laboratory QC Summary**

| QC Check Type | Minimum Frequency | Use |
|---|---|---|
| Field QC | | |
| Duplicate | 1 per matrix per 20 samples or SDG* | Precision |
| Trip Blank | 1 per VOC cooler | Sensitivity |
| Field Blank | 1 per matrix per 20 samples, 1 per day when sampling for PFAS | Sensitivity |
| Equipment Blank | 1 per day when sampling for PFAS | Sensitivity |
| Laboratory QC | | |
| Laboratory Control Sample | 1 per matrix per SDG | Accuracy |
| Matrix Spike/Matrix Spike Duplicate/Matrix Duplicat | 1 per matrix per SDG | Accuracy/Precision |
| Surrogate Spike | All organics samples | Accuracy |
| Laboratory Duplicate | 1 per matrix per SDG | Precision |
| Method Blank | 1 per matrix per SDG | Sensitivity |

* SDG - Sample Delivery Group - Assumes a single extraction or preparation
** Provided to lab by field sampling personnel
PFAS - Per- and Polyfluoroalkyl Substances

## Table 2. Remedial Investigation Sampling Summary

| Sample Medium | Target Analytes | Field Samples | Replicates[1] | Trip Blanks[2] | Field Blanks[1] | Equipment Blanks[3] | Matrix Spikes[1] | Spike Duplicates[1] | Total No. of Samples |
|---|---|---|---|---|---|---|---|---|---|
| Soil | TCL VOCs +10 | 28 | 2 | 14 | 2 | - | 2 | 2 | 50 |
| | TCL SVOCs +20 | 28 | 2 | - | 2 | - | 2 | 2 | 36 |
| | TAL Metals | 28 | 2 | - | 2 | - | 2 | 2 | 36 |
| | Mercury | 28 | 2 | - | 2 | - | 2 | 2 | 36 |
| | PCBs | 28 | 2 | - | 2 | - | 2 | 2 | 36 |
| | Herbicides | 28 | 2 | - | 2 | - | 2 | 2 | 36 |
| | Pesticides | 28 | 2 | - | 2 | - | 2 | 2 | 36 |
| | PFAS | 28 | 2 | - | 2 | - | 2 | 2 | 36 |
| | 1,4-Dioxane | 28 | 2 | - | 2 | - | 2 | 2 | 36 |
| | Total Cyanide | 28 | 2 | - | 2 | - | 2 | 2 | 36 |
| | Hexavalent Chromium | 28 | 2 | - | 2 | - | 2 | 2 | 36 |
| Groundwater | TCL VOCs +10 | 4 | 1 | 4 | 1 | - | 1 | 1 | 12 |
| | TCL SVOCs +20 | 4 | 1 | - | 1 | - | 1 | 1 | 8 |
| | TAL Metals (Total) | 4 | 1 | - | 1 | - | 1 | 1 | 8 |
| | TAL Metals (Dissolved) | 4 | 1 | - | 1 | - | 1 | 1 | 8 |
| | Mercury | 4 | 1 | - | 1 | - | 1 | 1 | 8 |
| | PCBs | 4 | 1 | - | 1 | - | 1 | 1 | 8 |
| | Herbicides | 4 | 1 | - | 1 | - | 1 | 1 | 8 |
| | Pesticides | 4 | 1 | - | 1 | - | 1 | 1 | 8 |
| | PFAS | 4 | 1 | - | 4 | 4 | 1 | 1 | 15 |
| | 1,4-Dioxane | 4 | 1 | - | 1 | - | 1 | 1 | 8 |
| | Total Cyanide | 4 | 1 | - | 1 | - | 1 | 1 | 8 |
| | Hexavalent Chromium | 4 | 1 | - | 1 | - | 1 | 1 | 8 |
| Soil Vapor | TO-15 VOCs | 6 | 1 | - | - | - | - | - | 7 |

Totals are estimated based on scope of work as written, actual sample quantities may vary based on field conditions. QA/QC sample quantities will be adjusted accordingly.

[1] Based on 1 per 20 samples or 1 per Sample Delivery Group (3 days max)

[2] Based on 1 cooler per day

[3] Based on 1 per day PFAS sampling occurs

TCL - USEPA Contract Laboratory Program Target Compound List
USEPA - United States Environmental Protection Agency
VOCs - Volatile Organic Compounds
SVOCs - Semivolatile Organic Compounds
PCBs - Polychlorinated Biphenyls
EPH - Extractable Petroleum Hydrocarbons
RCRA - Resource Conservation and Recovery Act
TCLP - Toxicity Characteristic Leaching Procedure
PFAS - Per- and Polyfluoroalkyl Substances
RIWP- Remedial Investigation Work Plan
ESA - Environmental Site Assessment
NYSDEC - New York State Department of Environmental Conservation
RRSCO - Restricted Residential Use Soil Cleanup Objectives
UUSCO - Unrestricted Use Soil Cleanup Objectives
TAL - USEPA Contract Laboratory Program Target Analyte List

## Table 3. Preservation, Holding Times and Sample Containers

| Analysis | Matrix | Bottle Type | Preservation(a) | Holding Time(b) |
|---|---|---|---|---|
| TAL Metals (total & dissolved)<br>SW-846 6020B/7471B | Soil<br>Water | 8 oz wide mouth glass, teflon lined cap<br>250 mL plastic, teflon lined cap | Cool to 4°C<br>Nitric acid | 180 days, Hg 28 days |
| Mercury<br>SW-846 7470A | Soil<br>Water | 5 gram Plastic<br>250 mL Plastic | Cool to 4°C<br>Nitric acid | 28 days from sample collection<br>28 days from sample collection |
| Hexavalent Chromium\Trivalent Chromium(calculate<br>SW-846 7196A\6010C | Soil<br>Water | 2 oz wide mouth glass, teflon lined cap<br>500 mL Plastic | None | 180 days<br>24 hours from sample collection |
| Total Cyanide<br>SW-846 9012B | Soil<br>Water | 4 oz wide mouth glass, teflon lined cap<br>250 mL Plastic | Cool to 4°C<br>NaOH | 14 days from sample collection<br>14 days from sample collection |
| 1,4-Dioxane<br>SW-846 8270D GS/MS SIM/Isotope Dilution | Soil<br>Water | 8 oz wide mouth glass, teflon lined cap<br>2 x 500 mL amber wide mouth glass | Cool to 4°C<br>Cool to 4°C | 14 days from sample collection<br>7 days from sample collection |
| Per- and Polyfluoroalkyl Substances (PFAS)<br>EPA 1633 | Soil<br>Water | 8 oz plastic container (non teflon lined)<br>2 - 250 mL plastic | Cool to 4°C<br>Trizma | 28 days from sample collection<br>14 days from sample collection |
| Volatile Organic Compounds (VOCs)<br>TO-15 | Air | 6 liter Summa Canister for 2-hr sampling | None | 14 days from sample collection |
| Methane<br>EPA 18 Modified | Air | 6 liter Summa Canister for 2-hr sampling | None | 30 days from sample collection |
| **Target Compound List (TCL)** | | | | |
| TCL Volatile Organic Compounds (VOCs) + 10 TICS<br>SW-846 8260C | Soil<br>Water | Encore<br>40mL voa vial, teflon lined cap | Cool to 4°C<br>Hydrochloric Acid | 24 hours from sample collection<br>14 days from sample collection |
| TCL Semivolatile Organic Compounds (SVOCs) +20<br>SW-846 8270D | Soil<br>Water | 8 oz wide mouth glass, teflon lined cap<br>1 liter amber glass, teflon lined cap | Cool to 4°C | 14 days to extract, 40 days to analysis<br>7 days to extract, 40 days to analysis |
| TCL Polychlorinated biphenyls (PCBs)<br>SW-846 8082A | Soil<br>Water | 8 oz wide mouth glass, teflon lined cap<br>1 liter amber glass, teflon lined cap | Cool to 4°C | 14 days to extract, 40 days to analysis<br>7 days to extract, 40 days to analysis |
| TCL Pesticides<br>SW-846 8081 | Soil<br>Water | 4 oz wide mouth glass, teflon lined cap<br>2 x 8 oz amber glass or plastic, teflon lin | Cool to 6°C<br>Cool to 6°C | 14 days to extract, 40 days to analysis<br>7 days to extract, 40 days to analysis |

(a) All soil and groundwater samples to be preserved in ice during collection and transport

(b) Days from date of sample collection.

TAL - Target Analyte List

TCL - USEPA Contract Laboratory Program Target Compound List

Gas chromatography-mass spectrometry (GC/MS) in selected ion monitoring (SIM) mode

# FIGURES

1. Proposed Sample Locations




N

LEGEND

- LOCATION OF PROPOSED SOIL BORING AND SOIL VAPOR POINT
- LOCATION OF PROPOSED SOIL BORING AND GROUNDWATER MONITORING WELL
- LOCATION OF PREVIOUS SOIL BORING
- LOCATION OF PREVIOUS SOIL VAPOR POINT
- PROPOSED BCP SITE BOUNDARY
- PARCEL BOUNDARY

NOTES

1. AERIAL SOURCE: NYS OFFICE OF INFORMATION TECHNOLOGY SERVICES GIS PROGRAM OFFICE (GPO)
2. PARCEL SOURCE: DEPARTMENT OF CITY PLANNING (DCP) PLUTO DATA SET

30 0 30'

Title:

# PROPOSED SAMPLE LOCATIONS

2030 FULTON STREET
BROOKLYN, NEW YORK

Prepared for:

STEPS FAMILY LLC

ROUX

| | | |
|---|---|---|
| Compiled by: B.H. | Date: 11/06/25 | FIGURE |
| Prepared by: M.S.R. | Scale: AS SHOWN | 1 |
| Project Mgr: B.H. | Project: 3805.0002Y003 | |
| File: 3805.0002Y113.5.mxd | | |

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# ATTACHMENTS

1. Professional Profiles
2. Laboratory's Standard Operating Procedures
3. Roux's Standard Operating Procedures
4. NYSDEC PFAS Sampling Guidance

# ATTACHMENT 1

Professional Profiles

# PROFESSIONAL PROFILE




## CONTACT INFORMATION

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## EDUCATION

BS, Geology, Binghamton University, 2005

## PROFESSIONAL LICENSES

Professional Geologist, New York, 2017

# Jessica L. Taylor, PG

## Principal Hydrogeologist

### EXPERIENCE SUMMARY

Nineteen years of experience: Principal, Senior, Project, and Staff Hydrogeologist, Roux, Islandia, New York; Staff Hydrogeologist and Intern at GSC | Kleinfelder.

### TECHNICAL SPECIALTIES

Project Management and Field Management for large-scale soil excavation and remediation projects, including site assessment, remediation implementation, and construction activities. Negotiation with NYSDEC Brownfield Clean-up Program (BCP) and NYCOER E-Designation/Voluntary Clean-up Program. Coordination and management of large-scale demolition and renovation support. Performance of sampling and direction of field sampling teams for the following media: soil, groundwater, surface water, soil vapor, sludge, and sediment. Soil characterization, coordination with construction management teams, disposal brokers, and facilities for cost-effective disposal of contaminated and clean soils from complex, large-scale projects.

### REPRESENTATIVE PROJECTS

- Project Principal for remediation of two parcels in Inwood, Manhattan, New York, as part of the NYSDEC Brownfield Cleanup Program. This project included due diligence environmental assessment and investigation of a former grocery store and retail gasoline filling station/auto repair, development of NYSDEC-approved Remedial Investigation Work Plan and Remedial Action Work Plan, and remediation during construction an affordable housing development. Acted on behalf of developer to provide soil classification data and vet disposal facilities and manage registration and disposal of unknown USTs.
- Project Principal for a large redevelopment project in Brooklyn, New York, including four buildings with NYCOER E-Designations for Hazardous Materials. The project encompasses 22 acres including the Barclays Center. Project responsibilities include coordination and oversight of in situ waste characterization sampling, excavation, and proper disposal of 500,000 CY of soil. Included sampling, classification, and coordination with construction managers and soil disposal facilities for disposal of soil impacted by petroleum contamination and TCLP hazardous levels of lead and corrosivity. Designed soil delineation sampling programs to limit boundaries of more contaminated soils to reduce disposal costs. Responsible for implementing and managing remediation work at several NYSDEC spill sites within the project footprint, including in situ chemical oxidation, UST removal, and soil excavation. Agency support for NYSDEC, NYCDEP, NYCOER, MTA (LIRR/NYCT), and ESDC.
- Project Principal providing technical guidance to construction management team during the excavation of NYSDEC Brownfield Cleanup Program project in Yonkers, New York. Coordinated with development team, their consultant, soil brokers, and disposal facilities to determine appropriate facilities and manage costs during disposal of approximately 30,000 tons of soil contaminated with TCSA-level PCBs, hazardous lead, and elevated VOCs triggering UHCs.
- Project Principal for remediation of two parcels in Hunter's Point South, Queens, New York, as part of the NYSDEC Brownfield Cleanup Program. This project included due diligence environmental assessment and investigation, development of NYSDEC-approved Remedial Investigation Work Plan and Remedial Action Work Plan, and remediation during construction of two mixed-use, affordable housing developments. Acted on behalf of developer to evaluate Change Order Request by contractor for disposal of soil with elevated PAHs and hazardous levels of lead. Designed delineation sampling program to limit costs associated with disposal of contaminated soil.

- Project Principal for remediation of a 0.66-acre parcel in Brooklyn, New York, as part of a NYSDEC Brownfield Cleanup Program. This project included due diligence environmental assessment and investigation, development of NYSDEC-approved RIWP and RAWP including an active sub-slab depressurization system, and remediation during construction of mixed- use affordable housing developments including full cellar. Also required coordination with NYCHPD and NYCDEP to meet regulatory requirements for funding. Continued support during the Site Management phase includes preparation of Periodic Review Reports and quarterly sampling and monitoring of an active sub-slab depressurization system.
- Project Principal for management of NYCOER E-Designation for Hazardous Materials during excavation and construction of a hotel/residential building in Manhattanville. Responsibilities included negotiation with NYCOER to limit additional sampling required for Remedial Investigation, and soil management including performing in situ waste characterization, coordination with soil brokers, and oversight of disposal of 16,000 CY of soil.
- Project Principal for real estate acquisition due diligence for industrial properties across Queens and the Bronx. Prepared Phase I ESAs and designed and implemented Phase II ESAs to meet financing requirements while coordinating with selling parties. Provided technical and strategic support, including remedial cost estimates during short due diligence periods.
- Project Principal for redevelopment of four properties for a supportive housing development in Brooklyn, New York, with NYCOER to address NYCDEP E-Designations. Coordination with NYCOER to implement remedial investigation and develop RAP as part of the NYC VCP.
- Senior Project Manager for the environmental management of asbestos remediation during the renovation of Nassau Coliseum, Uniondale, New York. Responsible for coordinating inspections and delineation of ACM, preparing budgetary estimates, and bid support for full abatement. Also includes management of decommissioning and replacement of existing emergency generator UST.
- Project Manager for commercial redevelopment site in the Bronx, including in situ waste characterization, management and coordination of excavation, community air monitoring, and development of NYCDEP-approved RAP.
- Client liaison and full-time on-site construction manager at redevelopment site in Rego Park, New York. Collection of 500 in situ waste characterization soil samples, oversight of 250,000 cubic yards of soil excavation and remediation, development of post-remediation sampling plan, and organization of waste manifests and hazardous waste documents to ensure proper disposal. Coordination of daily site activities with multiple construction contractors and other involved parties on behalf of client. Oversight and confirmatory soil sampling for on-site treatment of 75,000 cubic yards of hazardous lead-contaminated soil.
- Project and Field Manager for Phase I and Phase II ESAs on a portfolio of retail gasoline stations in New York and New Jersey. Responsibilities included drilling and sampling oversight and health and safety management, as well as writing Phase II ESA reports for over 40 sites.
- Field Manager for remediation system installation in Brooklyn, New York. Responsibilities included daily oversight of contractors excavating soil and installing piping within streets and sidewalks. Ensured compliance with client-specific Health and Safety Completed daily health and safety meeting and reports and attended weekly construction progress meetings.
- Field manager for quarterly groundwater sampling rounds at a former pesticides manufacturing facility in Dayton, New Jersey. Responsibilities include direction of multiple sampling teams and coordination with subcontractors.
- Field manager for site investigation activities at a redevelopment site in Staten Island. Responsible for oversight of subcontractor performing soil borings and soil and groundwater sampling.
- Field manager during groundwater monitoring rounds at former oil and fuel distribution terminals in Staten Island, Westchester, Glen Cove, and Oceanside, New York. Activities include system operations and maintenance, supervision of multiple field personnel, performing water levels gauging, and collection of groundwater samples from site monitoring wells using a variety of equipment.
- Field manager during a site investigation at a former chemical manufacturer in Brooklyn, New York. Responsibilities included oversight of subcontractor installation of monitoring wells, collection of soil and groundwater samples, and logging soil lithology.
- Field manager for annual groundwater sampling at a chemical manufacturing Superfund site in Delaware. Involves the oversight and supervision of multiple teams of personnel for a weeklong gauging and sampling round.
- Field manager at an underground storage tank closure in Queens, New York. Provided health and safety supervision, collection of soil and groundwater samples, and oversight of subcontractor closure activities on behalf of client.
- Field manager for a long-term remediation project for the City of New York. Includes quarterly groundwater sampling and soil vapor sampling as part of an NYSDEC-approved work plan, as well as oversight of soil sampling as part of subsurface investigations. Creation of tables and figures based on investigation results and as part of periodic reports submitted to NYSDEC.
- Field manager during off-site investigation at a former chemical production facility in Rensselaer, New York. Investigation

included the installation of piezometers, monitoring wells, and soil gas and environmental health and safety oversight.

- Project and Field Manager for implementation of a Phase II ESA at one of the largest on-going redevelopment projects in New York. Responsibilities included coordination and oversight of drilling, sampling, and excavation of soil. Drilling methods included sonic rotary, Geoprobe, hollow-stem auger, and air hammer/split spoon sampling. Includes oversight of Phase II ESA and waste characterization activities, coordination with laboratory contacts, as well as synthesis of lithology logs for over 100 soil borings. Project includes completion of Phase II ESAs (sampling of soil, groundwater, and soil gas) for over 50 properties.
- Project and field manager responsible for implementing and managing remediation work at several NYSDEC spill sites within the footprint of a large redevelopment project in Brooklyn, New York. Responsibilities included management and oversight of in situ chemical oxidation for treatment of petroleum-impacted groundwater.
- Field manager for redevelopment site in New Jersey. Collection of waste characterization soil samples, post excavation confirmatory sampling, and groundwater off-site discharge sampling.

**PROFESSIONAL TRAININGS**

OSHA 40-Hour HAZWOPER Training, 2005

OSHA 10-Hour Construction Safety Training, 2008

PROFESSIONAL PROFILE




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**EDUCATION**
BS, Environmental Science,
Minor in Engineering,
Binghamton University, 2019
AS, Environmental Science,
Suffolk County Community
College, 2017

# Brooke Hildebrand

## Project Scientist

**EXPERIENCE SUMMARY**
Six years of professional experience: Project Scientist, Roux, Islandia, New York.

**TECHNICAL SPECIALTIES**
Phase I and II environmental site assessments; Remedial construction and soil excavation oversight; Hazardous and non-hazardous waste disposal oversight; New York State Pollutant Discharge Elimination System (SPDES) monitoring and Stormwater Pollution Prevention Plan (SWPPP) compliance. Soil import oversight, Waste characterization and delineation oversight; Environmental site assessments focusing on soil, groundwater, and soil vapor investigations using multiple sampling techniques; Drilling oversight; Implementation of Community Air Monitoring Programs (CAMP); Insurance litigation support.

**REPRESENTATIVE PROJECTS**

**Industrial Site Assessment and Remediation**

- Authored and assisted with Phase I Environmental Site Assessment portfolios for 13 parcels of land located in the southern portion of Brooklyn, New York. Responsible for maintaining report integrity and compliance with ASTM E1527-13 standards. Maintained client expectations and delivered accurate results in a timely manner to satisfy tight due diligence deadlines. Provided recommendations to the client where Phase II investigations were warranted.
- Staff on an insurance litigation case regarding the evaluation of past and future costs for the investigation and remediation of a site with past railroad, wood treating, and drum reconditioning operations. Responsibilities included the review and organization of over 20,000 documents, past cost categorization, and report writing assistance.
- Main author of various technical reports submitted to the NYSDEC for Sites located throughout the boroughs including remedial action report, construction completion report, periodic review reports, phase II reports, daily construction reports, daily CAMP reports, final engineering report, site management plan, remedial investigation report, etc.
- Field manager for remedial excavation activities, CAMP implementation, and endpoint soil sampling at an affordable housing Site located in Harlem, New York. Main author of the Site's final engineering report (FER) submitted to the New York State Department of Environmental Conservation (NYSDEC) following completion of the environmental role onsite.
- Field manager for the implementation of the On-Site Environmental Monitoring (OEM) Program at a 43-acre redevelopment project in Belmont, New York. Responsibilities included ensuring environmental compliance with regards to air, stormwater, noise, traffic, and other relevant environmental concerns during the performance of any construction-related activity across the Site.
- Field manager for a Site in Sleepy Hollow, New York performing soil importation and fill operation activities. Responsibilities include implementation of CAMP; management of soil importation; preparation of daily progress reports submitted to the NYSDEC; onsite soil tracking; and oversight of fill operations using onsite and imported fill.
- Field Staff for groundwater and soil vapor sampling monitoring programs at a former petroleum refinery and terminal in Brooklyn, New York. This work was done to monitor the largest subsurface free-product plume in North America. Field work responsibilities included

the sampling of over fifty wells and monitoring points for petroleum contaminated groundwater and soil vapor using multiple sampling methods.

- Field Manager responsible for the implementation of groundwater sampling activities at multiple locations including a high-end shopping mall site in Norwalk, Connecticut, and a Site in Brooklyn, New York.
- Field manager for NYSDEC BCP Site located in Gowanus neighborhood in Brooklyn, NY. Industrial Site history and proximity to Superfund Site Gowanus Canal requires a USEPA-approved remediation. Responsibilities included construction oversight, CAMP implementation, SWPPP, and groundwater sampling.
- Field manager responsible for air monitoring to collect interior air quality data during construction activities at a site in Hudson Yards, New York enrolled the New York State Department of Environmental Conservation (NYSDEC) Brownfield Cleanup Program (BCP). Roux implemented monitoring by utilizing 13 stationary 5-gas meters with telemetry and 2 roaming 5-gas meters, capable of measuring the following air quality parameters: oxygen, carbon monoxide, hydrogen sulfide, methane (as LEL) and organic vapors (VOCs). Responsibilities also included construction oversight, analyzing air monitoring data and preparing associated reporting daily.
- Field Manager responsible for environmental oversight of geotechnical work at a generating station in Astoria, New York. Responsibilities included monitoring and enforcing the subcontractor's adherence to specific procedures to prevent vertical migration of NAPL, logging and screening soil with a photo ionization detector (PID), recording lithology of soil cores, and confirming depths of clay layers and bedrock.
- Field Manager responsible for the implementation of CAMP during excavation, disposal, and import activities at multiple locations in Queens, Brooklyn and Upstate, New York. Monitored airborne dust and VOCs that are potentially generated by remedial action work activities, reviewing the collected data for exceedances of the New York State Department of Health (NYSDOH) guidelines.
- Site Safety Officer for various remedial investigation sites. Responsibilities include preparation of health and safety plans (HASPs); development and review of job safety analysis (JSA) documents; on-site safety meeting management; safety document preparation (Lessons Learned, Near Loss, Field Audits, etc.); and planning/ execution of corrective actions.
- Project Manager for multiple public sector sites located in Queens and Manhattan with petroleum contamination. Responsibilities include project management, coordination of groundwater monitoring and product recovery events, data evaluation and reporting and client communications.
- Project Manager for NYSDEC BCP Site located Upstate, New York. Responsibilities included project management of the remedial investigation which included coordination of day and night shifts, work within an active commercial building, data evaluation, preparation of a remedial investigation report and remedial action work plan, client communications, and NYSDEC communications.
- Project Manager for multiple phase II investigations conducted at sites located in Brooklyn, Queens, and Manhattan. Responsibilities included subcontractor coordination, project management, data evaluation, reporting, and client communications.

**JUDY V. HARRY**
**P. O. Box 208**
**120 Cobble Creek Rd.**
**North Creek, NY 12853**

***Occupation:*** Data Validator/Environmental Technical Consultant

***Years Experience:*** 45

***Education:*** B.S., Chemistry, Magna cum laude, 1976, Phi Beta Kappa

***Certifications:*** New York State Woman-Owned Business Enterprise (WBE)

***Relevant Work History:***

**Data Validation Services: September 1989 - present**

Sole proprietor of Data Validation Services, a woman-owned small business registered with SAM, providing consultation/validation services to regulatory and commercial clients.

These services include the review of analytical laboratory data for compliance with respect to specific protocols, accuracy and defensibility of data, verification of reported values, and evaluation of quality parameters for analytical usability of results. Approved by USEPA, NYSDEC, NJDEP, NYSERDA, and NYCDEP as a data validator for projects, including USEPA Superfund, Brownfield, and lead sites, and those contracted through the NYSDEC Division of Hazardous Waste Remediation, Division of Solid Waste, and Division of Water Quality.

Performed validation for compliance with laboratory analytical protocols including USEPA OLM, USEPA OLC, USEPA ILM, USEPA DFLM, USEPA SOW3/90, USEPA SOW 7/87 CLP, USEPA SOW 2/88 CLP, USEPA SW846, RCRA, AFCEE, NYS 6 NYCRR Part 360, 40 CFR, Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, including TO-15, 1989/1991/1995/2000/2005 NYSDEC ASPs, and 1987 NYSDEC CLP.

Performed validation according to the USEPA National and Regional SOPs and Functional Guidelines, AFCEE requirements, NYSDEC Validation Scope of Work, NYS DUSR, and NJDEP Division of Hazardous Site Mitigation/Publicly Funded Site Remediation SOPs.

Performed validation for USEPA Superfund Sites including Salem Acres, York Oil, Port Washington L-4 Landfill, Bridgeport Rental and Oil Services, GE-MRFA, MMR/ OTIS AFB, LCP, and Peter Cooper site; and for USEPA lead sites including SJ&J Piconne, Maska, Bowe System, Jones Sanitation, and Syossett Landfill, involving CLP, RAS, and SAS protocols.

Contracted for NYSDEC Superfund Standby Contracts with LMS Engineers/HDR, CDM Smith, Malcolm-Pirnie/ARCADIS, Ecology & Environment, Shaw Environmental/APTIM, CG&I, O'Brien & Gere Engineers, and EC Jordan, involving samples collected at NYS Superfund Sites and analyzed under the NYSDEC ASP.

Performed validation services for NYSDEC Phase II remedial investigations, RI/FS projects, Brownfield sites, and PRP over-site projects for hazardous waste sites.

Performed validation services for clients conducting RI/FS activities involving samples of many matrices, including waste, air, sludges, leachates, solids/sediments, aqueous, and biota.

Clients have included AECOM, ARCADIS, Barton & Loguidice, Benchmark Engineering, Bergmann Associates, Blasland, Bouck & Lee, Brown and Caldwell, CDM Smith, CB&I Shaw Environmental, C&S Consulting Engineers, Chazen Companies, Clough Harbour & Associates, Columbia Analytical Services, C.T. Male, Dames & Moore, Day Engineering, EA Engineering, EcolSciences, Ecology & Environment, Ecosystems, EC Jordan, Environmental Chemical Corporation, EHRT, ENSR Consulting, ELM, ERM-Northeast, Fagan Engineers, Fanning Phillips & Molnar, FluorDaniel GTI, Frontier, Foster Wheeler Environmental Corp, Frontier Technical, Galson Consultants, GEI, GE&R, Geomatrix Consultants, GZA Environmental, Handex of N, Hazard Evaluations, H2M Group, HDR, HRP, IT Corp, Jacques Whitford, JTM Associates, JMT, Labella Associates, Langan Engineers, Leader Environmental, Lockwood, Kessler & Bartlett, LMS Engineers, Malcolm-Pirnie, Metcalf & Eddy, NWEC&C, O'Brien & Gere Engineers, Pace, Parsons Engineering-Science, Plumley Engineering, Prescott Environmental, P. W. Grosser, Rizzo Associates, Roux Associates, Sear Brown Group, SECOR, Shaw Environmental, Stantec, ThermoRemediation Inc., TRC Environmental, Turnkey Environmental Restoration, TVGA Engineering, URS Consultants, Wehran Emcon, Weston, Wittman Geosciences, YEC, and private firms.

Provided consultation services to laboratories regarding analytical procedures and protocol interpretation, and to law firms for litigation support.

Provided services to firms involving audits of environmental analytical laboratories to determine analytical capability, particularly for compliance with NYSDEC ASP and AFCEE requirements.

Guest speaker on a panel discussing Data Review/Compliance and Usability, for an analysis workshop for the New York Association of Approved Environmental Laboratories, 1993.

## Adirondack Environmental Services: June 1987 - August 1989

Senior mass spectroscopist for AES. Responsible for GC/MS analyses of environmental samples by USEPA and NYSDEC protocols, development of the GC/MS laboratory, initiating the instrumental and computer operations from the point of installation, and for implementing the procedures and methodologies for Contract Laboratory Protocol.

## CompuChem Laboratories: May 1982 - January 1987

Managed a GC/MS production laboratory; developed, implemented, and supervised QA/QC criteria at three different levels of review; and was responsible for the development and production of the analysis of environmental and clinical samples. Directed a staff of 23 technical and clerical personnel, and managed the extraction and GC/MS labs and data review operations.

## Research Triangle Institute (RTI): December 1979 - May 1982

Worked as an analytical research chemist responsible for development of analytical methods for the EPA Federal Register at RTI. This involved analysis of biological and environmental samples for priority pollutants, primarily relating to wastewaters and to human sampling studies. Method development included modification and interfacing of the initially developed Tekmar volatile purge apparatus to GC/MS, development and refinement of methods for entrapment and concentration of the air medium for subsequent volatile analysis, and the analysis and resolution/ identification of individual PCB congeners within Aroclor mixtures by capillary column and mass spectra.

## Guardsman Chemical Company: February 1977 - November 1979

Performed all quality control functions for the manufacturing plant. Performed research and development on coatings and dyes.

## Almay Cosmetics: May 1976 - December 1976

Product evaluation chemist. Responsible for analytical QC of manufactured products.

## Publication

Pellizzzari, E.D., Moseley, M.A., Cooper, S.D., Harry, J.V., Demian, B., & Mullin, M. D. (1985). Recent Advances in the Analysis of Polychlorinated Biphenyls in Environmental and Biological Media. *Journal of Chromatography*, 334(3) 277-314.

# ATTACHMENT 2

Laboratory's Standard Operating Procedures

# 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

# Method 1633 Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids and Tissue Samples by LC-MS/MS

**References:** Method 1633 - Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, 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, Biosolids and Soil 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 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 Soil and Sediment 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** 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.

**6.3.2** 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) 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 – (Waters Oasis WAX 150 mg, Cat # 186002493 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.

**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.

**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 l 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 ACETATE – (Caledon Ultra LC/MS grade or equivalent

**8.1.11** AMMONIUM HYDROXIDE ($NH_3$, CAS#: 1336-21-6) – High purity, demonstrated to be free of analytes and interferences.

**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).

**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

**8.1.24** CARBON - – EnviCarb® 1-M-USP or equivalent, verified by lot number before use, store 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.

**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%

**9.10.3**

## 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 to 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 Soils, Solids and Sediments.

**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 Tissues.

**10.4.1** 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.

**10.4.2** Add 20 µL of the EIS PDS 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** 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.

**10.4.5** 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.

**10.4.6** 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.

**10.4.7** 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.

**10.4.8** 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.

**10.4.9** 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.

## 10.5 SPE Extract: All matrices

**10.5.1** Pack clean silanized glass wool to half the height of the WAX SPE cartridge barrel.

**10.5.2** Pre-condition the cartridges by washing them with 3 X 5 mL of 1% methanolic ammonium hydroxide, discarding the wash volumes.

**10.5.3** 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

**10.5.4** 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.

**10.5.5** 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.

**10.5.6** 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.

**10.5.7** 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.

**10.5.8** Let the wetted sorbent bed and wool soak for 1 minute.

**10.5.9** Open the cartridge valve and collect the eluate into a 15 ml polypropylene collection tube.

**10.5.10** Add 25 µL of concentrated acetic acid to each sample eluted in the collection tubes and vortex to mix.

**10.5.11** 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.

**10.5.12** 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.

**10.5.13** 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

## 10.6 Sample Volume Determination

**10.6.1** 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.

**10.7 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.

**10.7.1** ESI-MS/MS TUNE

**10.7.1.1** Calibrate the mass scale of the MS with the calibration compounds and procedures prescribed by the manufacturer.

**10.7.1.2** 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.

**10.7.1.3** 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.

**10.7.2** 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.

**10.7.3** 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.

**10.7.4** 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.

**10.7.5** Prepare a set of CAL standards as outlined in table 5. The lowest concentration CAL standard must be at or below the LOQ.

**10.7.6** 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.

**10.7.7 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.

**10.7.8 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.

**10.8 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. Subsequent CCVs should alternate between a medium and Low concentration CAL standard.

**10.8.1** Inject an aliquot of the appropriate concentration CAL standard and analyze with the same conditions used during the initial calibration.

**10.8.2** 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.

**10.8.3** 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 and verification of sensitivity by analyzing a CCV at or below the LOQ.

## 10.9 EXTRACT ANALYSIS

**10.9.1** The same operating conditions used for the initial calibration and summarized in Tables 6 and 7 are to be used.

**10.9.2** 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.

**10.9.3** 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.

**10.9.4** Calibrate the system by either the analysis of a calibration curve or by confirming the initial calibration is still valid by analyzing a CCV.

**10.9.5** 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.

**10.9.6** 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.

**10.9.7** 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.

**10.9.8** 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

**10.9.9** 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 |
| Perfluoorotetradecanoic 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 |

| Isotope Labeled Standard | Conc. of EIS Stock (ng/mL) | Nominal amount of EIS added to extracts (ng) |
|---|---|---|
| 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 ® BEH$C_{18}$ 2.1 x 50 mm packed with 1.7 µm BEH $C_{18}$ stationary phase<br>Flow rate of 0.4 mL/min<br>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 |

# 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°C) 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<br>Re-extract if still failing |
| IS Failure | IS not present (less than 10% recovery) | Add IS<br>Rerun |
| | IS out high or low | Revial/rerun<br>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<br>Re-extract if contamination still present |
| | Above RL, sample(s) concentration less than 10 times above the blank and no volume available | Revial/rerun<br>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<br>Rerun |
| | IS out high or low | Revial/rerun<br>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<br>Re-extract if still high |
| | Spike compounds out low | Revial/rerun<br>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<br>Re-extract if still failing |
| IS Failure | IS not present (less than 10% recovery) | Add IS<br>Rerun |
| | IS out high or low | Revial/rerun<br>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<br>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

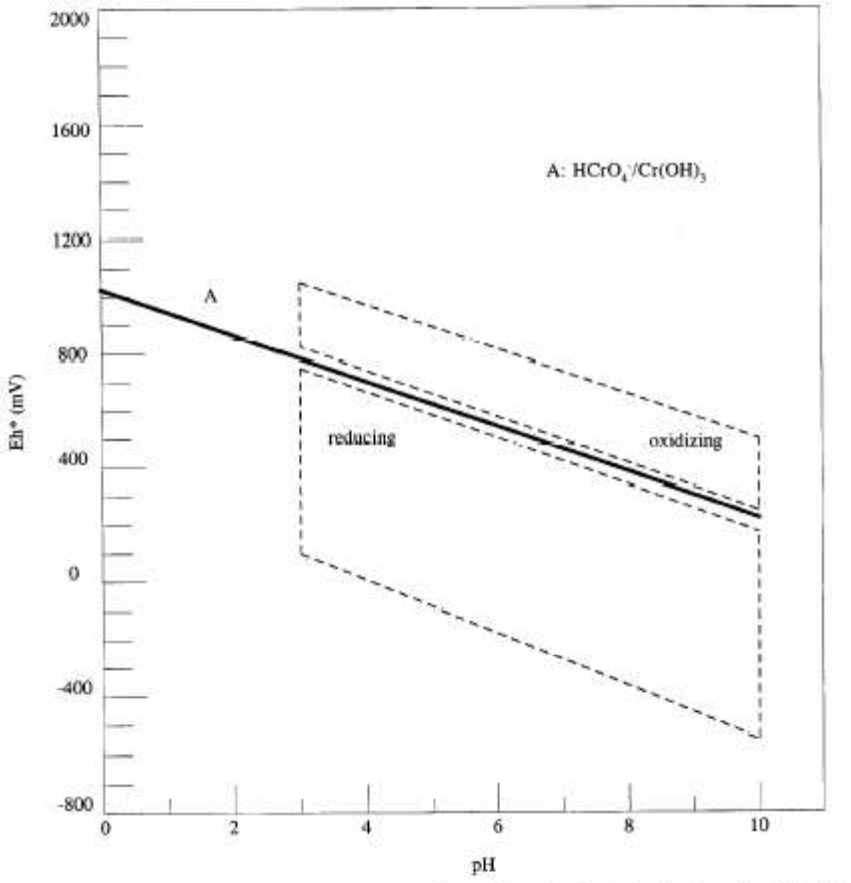
SOP/1728 Waste Management and Disposal SOP

# 16. Attachments

Figure 1: Eh/pH Phase Diagram

FIGURE 1
Eh/pH PHASE DIAGRAM

The dashed lines define Eh-pH boundaries commonly encountered in soils and sediments.




* Note the Eh values plotted on this diagram are corrected for the reference electrode voltage: 244 mV units must be added to the measured value when a separate calomel electrode is used, or 199 mV units must be added if a combination platinum electrode is used.

# 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. $Hg2{+}{+}SnCl2 \rightarrow Hg0 \uparrow$

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.

**7.1 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

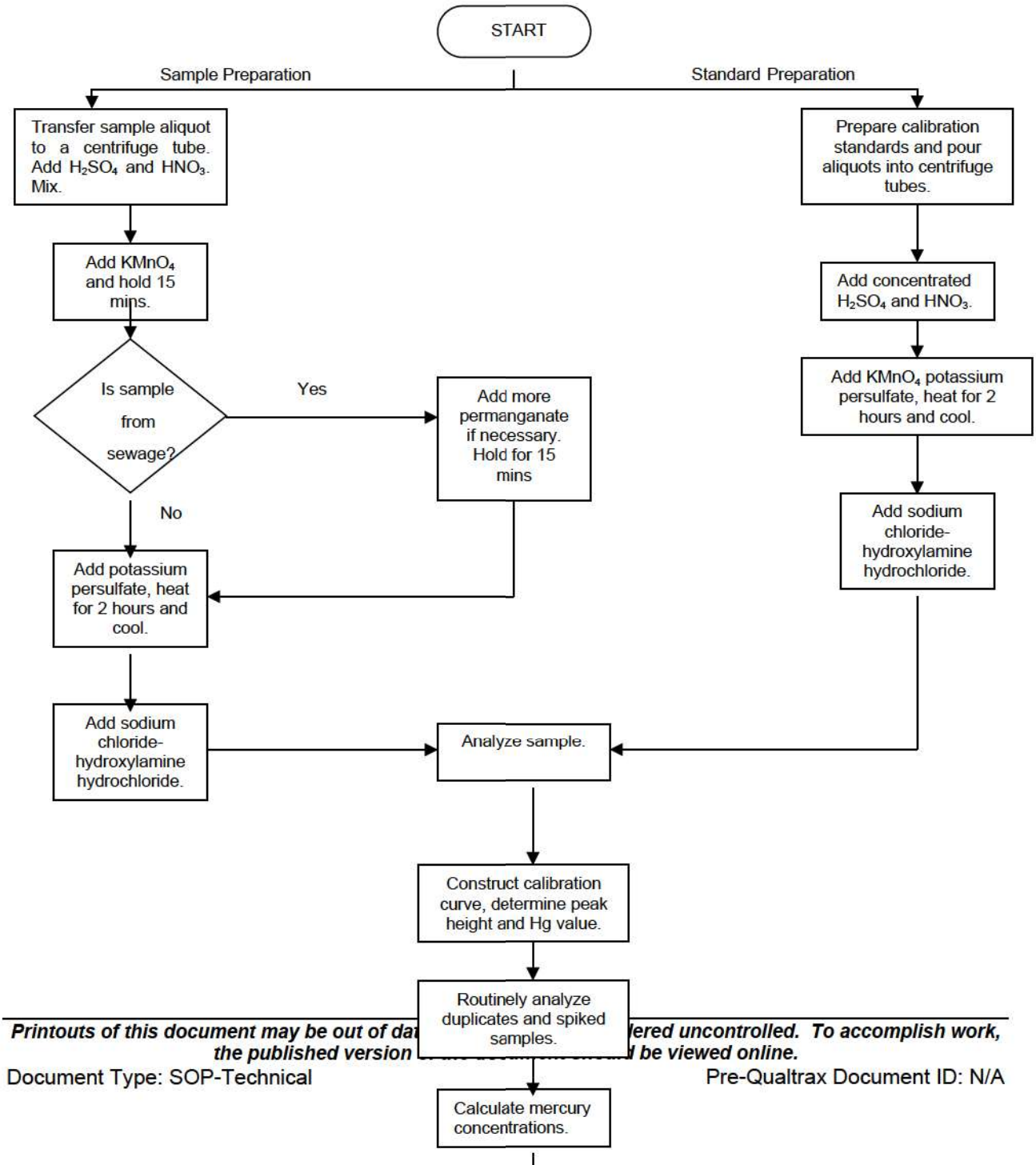
SOP/1797 Waste Management and Disposal SOP

# 16. Attachments

Figure 1: Method 7470A Flow Chart

**Figure 1**

**Method 7470A Flow Chart**



END

# Mercury 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

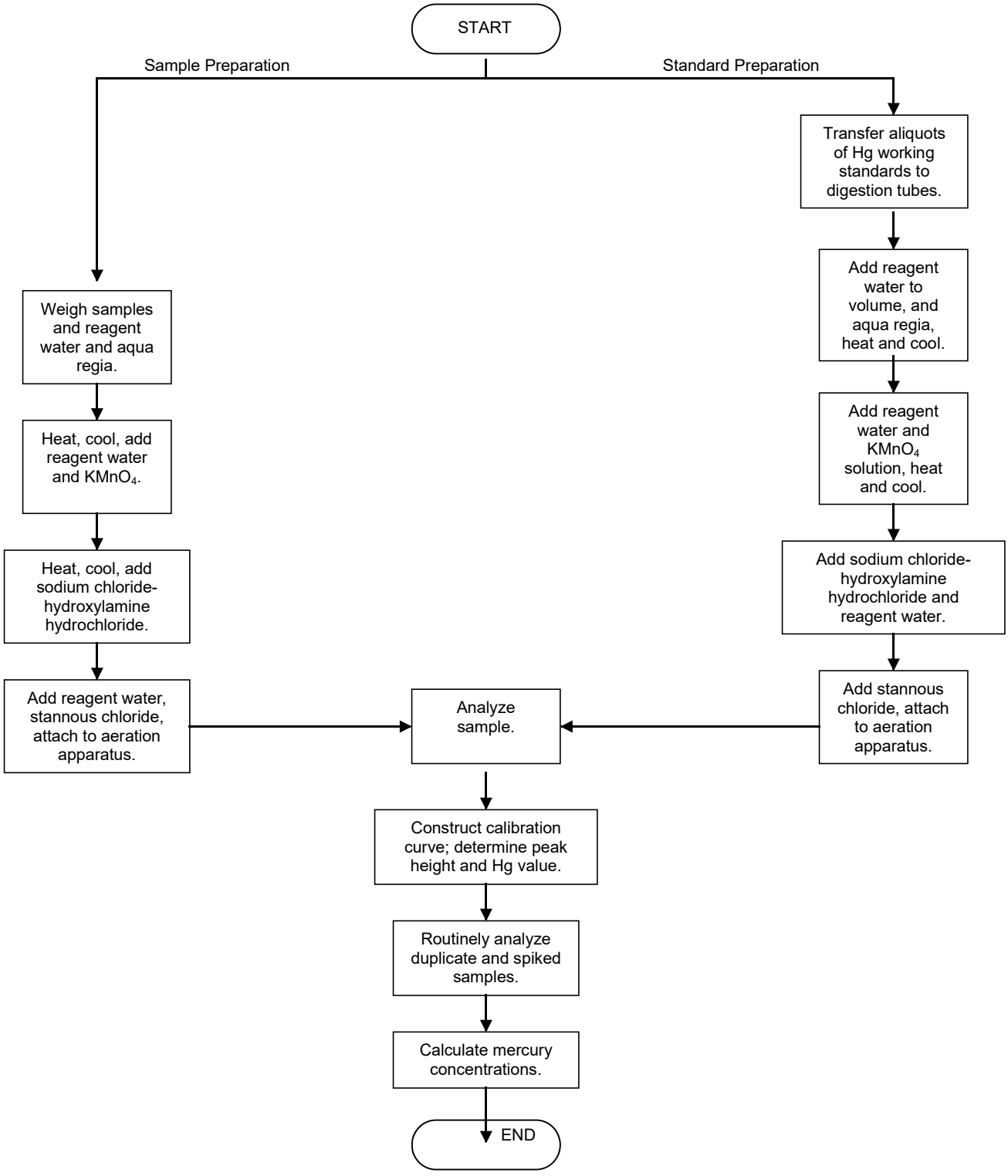
SOP/1797 Waste Management and Disposal SOP

## 16. Attachments

FIGURE 1: Flow Chart for Method 7471B

## Figure 1

## Method 7471B Flow Chart



# 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.

# 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 1, July 1, 2010.

SW-846 Pesticides by 8081, State of Connecticut Department of Environmental Protection Reasonable Confidence Protocol (RCP), Version 1.0, July 2005

## 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 Alpha 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 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, 500 mls or 1L, for solids, 15g to 30g) 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 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.

**7.1.1 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:** 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.

**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 Volumetric Flasks:** 10mL, 25mL, 50mL, 100mL, 200mL, and 2000mL, for the preparation of standards.

**7.4 Microsyringes/Wiretrol syringes:** 10 µL – 1000 µL

**7.5 Disposable Borosilicate Pipets**

**7.6 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 1 year 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 performed at 10 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<br>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 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.

**8.8.1** Preparation of surrogate standard:

(syringe needed: *1000μL [A]*)
(solvent: *Acetone)*
(glassware: *1x2000mL, volumetric flasks*)

**SURROGATES:** *2000μg/L; 2000mL*
20mL[A] of AccuStandard CLP-032-R (Deca. & TCMX; 0.2mg/mL)

**8.9 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.

**8.9.1** 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 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 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 is 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 Method-specific Quality Control Samples

### 9.7.1 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

**Initial calibration:**

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:**

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 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 in 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** 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 Turbochrom or Agilent Chemstation data acquisition software.

**10.2.2.1** Record the calibration standard, unique lab identifier code (lot), 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** 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.

**10.2.5 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 $\leq$ 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 $\geq$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.

If samples are to be analyzed per the Massachusetts Contingency Plan (MCP) Method, averaged, linear or non-linear regression may be used. When a linear calibration is used,

the required calibration coefficient needs to be > 0.99. In addition, when using linear or non-linear regression, the low standard must be processed against the curve and fall within 70-130% of the curve.

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 Retention Time Windows**

**10.2.7.1** 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.

**10.2.7.2** 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.

**10.2.8 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.

**10.2.8.1** CT-RCP requires that %D for each analyte to be quantitated must not exceed a ± 15% difference when compared to the initial calibration curve.

## 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 ≤ 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 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.

**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, 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.

**10.3.4.1** CT-RCP requires a closing calibration verification standard with a %D that is ± 15% for each of the target analytes.

**10.3.5** Sample injections may continue for 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 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 samples and at the end of the analysis sequence for CT-RCP 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 Alpha's Quality Manual. The Internal Standard area of the CCV must be within 50%-150% of the Initial Calibration Internal Standard area.

CT-RCP samples require that the response factor (for internal standard compounds) for each analyte to be quantitated must not exceed a ± 15% difference when compared to the initial calibration curve.

**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** GC injector ports are of critical concern, especially in the analysis of DDT and Endrin.

**10.6.1.1.1** 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. 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 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 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 | 2500 | 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<br>Re-extract if still failing |
| IS Failure | IS not present (less than 10% recovery) | Add IS<br>Rerun |
| | IS out high or low | Revial/rerun<br>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<br>Re-extract if contamination still present |
| | Above RL, sample(s) concentration less than 10 times above the blank and no volume available | Revial/rerun<br>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<br>Rerun |
| | IS out high or low | Revial/rerun<br>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<br>Re-extract if still high |
| | Spike compounds out low | Revial/rerun<br>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<br>Re-extract if still failing |
| IS Failure | IS not present (less than 10% recovery) | Add IS<br>Rerun |
| | IS out high or low | Revial/rerun<br>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<br>Re-extract if still failing |
| | RDP failure | Report as is |

# 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.1, July 1, 2010.

State of Connecticut, Department of Environmental Protection, RRCP, Version 2.0, July 2006.

# 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 (volumes and weights can vary but approximately 1L or 140-272 mls (LVI – Low Volume Initiative) 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**: 0.25 ug/L / Aroclor (based on a 1L extraction or 140 ml LVI 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 of 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's cabinet.

# 6. Sample Collection, Preservation, Shipping and Handling

## 6.1 Sample Collection

Aqueous samples are collected in two 1L or two 140 ml (LVI) amber glass jars with Teflon-lined lids. Solid samples are collected in one 250 mL 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 and RCP CT aqueous samples must be extracted within 7 days of sample collection, solid and oil samples within 14 days of collection (NJ DKQP allows 365 days for solids if frozen). All other samples, both aqueous and solid, 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 are 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:

- **1660 L6**: (mix of Aroclors 1016 and 1260) is prepared by diluting
  - 2.5uL 1016 Standard (Agilent, Cat. #PP-282)
  - 2.5uL 1260 Standard (Agilent, Cat. #PP-361)
  - 1.6mL of PCB ICAL Surrogate Stock

  to 25ml of Hexane to achieve concentration of
  - 1016 and 1260 at 10ug/mL
  - TCMX at 0.64ug/mL and DCB at 1.28ug/mL.
- **For LVI**, this solution is diluted 10X more, achieving a concentration of
  - 1016 and 1260 at 1ug/mL
  - TCMX at 0.064ug/mL and DCB at 0.128ug/mL
- **2154 L6:** (mix of Aroclors 1221 and 1254) is prepared by diluting
  - 2.5uL 1221 Standard (Agilent, Cat. #PP-292)
  - 2.5uL 1254 Standard (Agilent, Cat. #PP-351)

  to 25ml of Hexane to achieve concentration of
  - 1221 and 1254 at 10ug/mL
- **For LVI**, this solution is diluted 10X more, achieving a concentration of
  - 1221 and 1254 at 1ug/mL
- **4268 L6** (mix of Aroclors 1242 and 1268), **3262 L6** (mix of Aroclors 1232 and 1262) and **1248 L6;** all are prepared from their respective Stock Standards according to the formula used for L6 of 2154.
- Levels 1-5 are prepared by serial dilutions of L6s and formula with volumes needed can be found in Table 2.

**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 are 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
- **For LVI**, this solution is diluted 10X more, achieving a concentration of
  - 1016 and 1260 at 0.25ug/mL
  - TCMX at 0.016ug/mL and DCB at 0.032ug/mL
- **2154, 4268, 3262 and 1248 ICVs** are prepared from their respective Stock Standards according to the formula used for 1660 ICV.

**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 LVI, 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 of 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. For LVI, this solution is diluted 10X more, achieving a concentration of 0.5ug/mL for TCMX and 0.1ug/mL for DCB.
- **CCAL Surrogates Stock:** is prepared by diluting of 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. For LVI, this solution is diluted 10X more, achieving a concentration of 1 ug/ml for TCMX and 2 ug/mL for DCB.
- **Extraction Surrogates Stock:** is prepared by diluting of 10mL of TCMX&DCB (Accustandard, Cat. #CLP-032-R) to 1000mL of Acetone to achieve concentration of TCMX and DCB at 2ug/mL. For LVI, this solution is diluted 10X more, achieving a concentration of 0.2 ug/mL for both TCMX and DCB.
- **PCB Caulking Surrogates Stock:** is prepared by diluting of 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 of 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 of 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 LVI, 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 of 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 of 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 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 Method Blank failures are listed in Table 5.

## 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 4. The recovery acceptance criteria are listed in Table 3. 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 5.

## 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 4. Upon client request, a 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 3. 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.

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 3. If the RPD criteria are not met, the failure may be attributed to matrix effect. Actions associated with DUP failures are listed in Table 5.

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 4. 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 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 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. 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
4. Matrix Spike / Matrix Spike Duplicate (if requested by Client)
5. Duplicate (if included with batch QC)
6. Samples up to 16
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.

| | 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 |

| | INSTRUMENT CONDITIONS |
|---|---|
| Injector temperature | 250$^0$C |
| Injector mode | Split |
| 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 |

## 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. Alternatively, a standard containing a mixture of Aroclor 1016 and Aroclor 1260 will include many of the peaks represented in the other five Aroclor mixtures. As a result, a multi-point initial calibration employing a mixture of Aroclors 1016 and 1260 (1660) at five concentrations should be sufficient to demonstrate the linearity of the detector response without the necessity of performing multi-point initial calibrations for each of the seven Aroclors. In addition, such a mixture can be used as a standard to demonstrate that a sample does not contain peaks that represent any one of the Aroclors. Single standards of each of the other seven Aroclors are required to aid the analyst in pattern recognition. Assuming that the Aroclor 1016/1260 standards have been used to demonstrate the linearity of the detector, these single standards of the remaining seven Aroclors also may be used to determine the calibration factor for each Aroclor when a linear calibration model through the origin is chosen. Prepare a standard for each of the other Aroclors. The concentrations 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.

**10.2.5.1 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 formula listed in Alpha's 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 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.
- **MCP (Massachusetts Contingency Plan) requirement**: minimum of five unique peaks must be evaluated for Aroclors 1016 and 1260.
- **MCP requirement:** Minimum of five standards (or six if non-linear regression used) must be used.
- **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%** (**15% for CT RCP**) when compared to the mean response factor from the initial calibration.

**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.

## 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 (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 (**± 15% for CT RCP**). The Enviroquant data processing software automatically calculates the **%D** for all analytes according to the formulae in Alpha's Quality Manual.

**10.4.3** **A Retention Time shift >30 seconds** for the internal standard necessitates reanalysis of all affected 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 (10.4.3).

**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 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

**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 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/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: QC ACCEPTANCE CRITERIA

Table 4: SPIKING LEVEL

Table 5: 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) |
|---|---|---|---|---|---|---|---|
| **PCB** | | | | | | | |
| Aroclor 1016/1260 | 100 | 0.1 | 0.5 | 1 | 2.5 | 5 | 10 |
| Aroclors 1221, 1232, 1242, 1254, 1262, 1268 | 100 | 0.1 | 0.5 | 1 | 2.5 | 5 | 10 |
| LVI | | 0.01 | 0.05 | 0.1 | 0.25 | 0.5 | 1 |
| **Internal Standard** | | | | | | | |
| 1-Bromo-2-Nitrobenzene | 5000 | 0.25 | 0.25 | 0.25 | 0.25 | 0.25 | 0.25 |
| LVI | | 0.025 | 0.025 | 0.025 | 0.025 | 0.025 | 0.025 |
| **Surrogates:** | | | | | | | |
| Tetrachloro-m-xylene | 2.0 | 0.0064 | 0.032 | 0.064 | 0.16 | 0.32 | 0.64 |
| Decachlorobiphenyl | 2.0 | 0.0126 | 0.064 | 0.128 | 0.32 | 0.64 | 1.28 |
| LVI – 10X less | | | | | | | |

**LVI is spiked 10X lower**

## TABLE 2

## PCB ICAL PREPARATION:

| Aroclors: 1660, 2154, 4268, 3262, 1248 | | | | |
|---|---|---|---|---|
| **Level** | **Conc [ug/L]** | **Spike** | **Spike [uL]** | **Hexane [mL]** |
| **6** | 10000 | L6 | 1000 | 0 |
| **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

## QC ACCEPTANCE CRITERIA

| Aqueous, Soils, Oils | | |
|---|---|---|
| **Surrogate % Recovery** | **Lower Control Limit** | **Upper Control Limit** |
| 2,4,5,6-Tetrachloro-m-xylene | 30% | 150% |
| Decachlorobiphenyl | 30% | 150% |

| | **Aqueous, Soils % Recovery** | | **Duplicate and/or MSD** | |
|---|---|---|---|---|
| **MS/MSD and LCS** | **Lower Control Limit** | **Upper Control Limit** | **Aqueous RPD** | **Soil RPD** |
| Aroclor 1016, 1260 | 40% | 140% | 30% | 50% |

## TABLE 4

## SPIKING LEVEL

| | LCS\MS\Surr Spiking Level | | | |
|---|---|---|---|---|
| | **Spike Solution [ug/mL]** | **Surr. Solution [ug/mL]** | **Spike Conc [ug/L]** | **Surr Conc [ug/L]** |
| **LVI** | 0.2 | 0.2 | 250 | 200 |
| **Soil/Water** | 12.5 | 2 | 3125 | 500 |
| **Oil** | 12.5 | 2 | 1250 | 200 |
| **Caulking** | 125 | 20 | 3125 | 200 |

## 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<br>Re-extract if still failing |
| IS Failure | IS not present (less than 10% recovery) | Add IS<br>Rerun |
| | IS out high or low | Revial/rerun<br>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<br>Re-extract if contamination still present |
| | Above RL, sample(s) concentration less than 10 times above the blank and no volume available | Revial/rerun<br>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<br>Rerun |
| | IS out high or low | Revial/rerun<br>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<br>Re-extract if still high |
| | Spike compounds out low | Revial/rerun<br>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<br>Re-extract if still failing |
| IS Failure | IS not present (less than 10% recovery) | Add IS<br>Rerun |
| | IS out high or low | Revial/rerun<br>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<br>Re-extract if still failing |
| | RDP failure | Report as is |

# 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

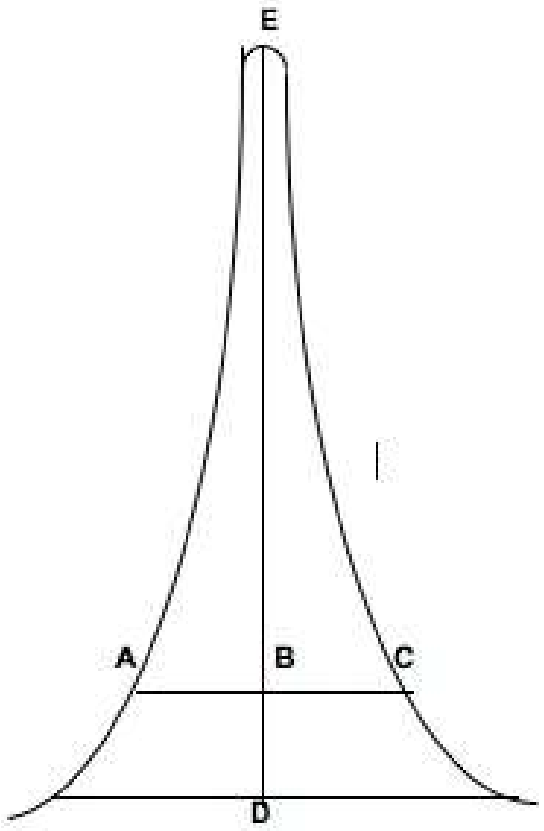
## ACCEPTABLE SOIL QC LIMITS

| | 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<br>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 |

# 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

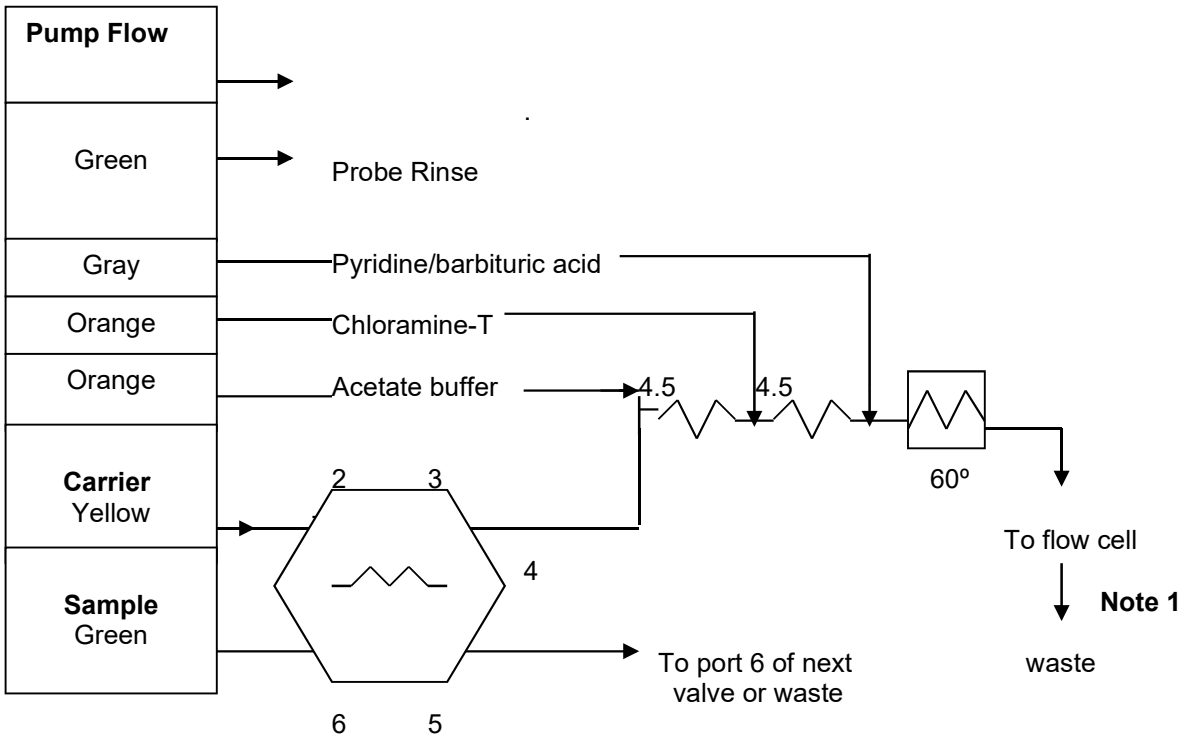
1728 Hazardous Waste Management and Disposal SOP

# 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

# 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:

$$\textit{Concentration (ug/Kg)} = \frac{\textit{(Conc.) (Vp) (5000) (DF)}}{\textit{(W) (Ve) (\%S)}}$$

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.

$$\textit{\% moisture} = \frac{\textit{g of sample} - \textit{g of dry sample}}{\textit{g of sample}} \times 100$$

$$V_t = \frac{\textit{[mL of solvent + (\%moisture x g of sample)]}}{100} \times 1000\text{mL/mL}$$

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:

$$\textit{Corrected concentration (mg/Kg)} = \frac{\textit{(Conc.) (}V_t\textit{ + methanol vol.) (Vp) (DF)}}{\textit{(W) (Ve) (\%S)}}$$

# 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 |

# Determination of Volatile Organic Compounds in Air Using Specially-Prepared Canisters and Analyzed by GC/MS

References: **Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air - Second Edition.** U.S. Environmental Protection Agency. EPA/625/R-96/010b. Office of Research and Development National Risk Management Research Laboratory. Center for Environmental Research Information. Cincinnati, Ohio. January 1999.

**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).** U.S. Environmental Protection Agency. EPA/625/R-96/010b. Office of Research and Development National Risk Management Research Laboratory. Center for Environmental Research Information. Cincinnati, Ohio. January 1999.

## 1. Scope and Application

**Matrices:** Ambient Air, Soil Gas, Soil Vapor

**Definitions:** Refer to Section 16 and Alpha Analytical Quality Systems Manual

This SOP describes the procedure for the analysis of volatile organic compounds (VOCS) in ambient air. The whole air samples are collected in fused-silica lined (FSL) stainless steel canisters, or Tedlar® bags. The VOCs are subsequently separated by gas chromatography (GC) and measured by mass selective detector (MSD).

The organic compounds that are amenable to this method are listed in Table 9. Other compounds may also be amenable provided they meet the QA/QC requirements of the method.

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.

This SOP contains addendums for various state-specific requirements. The criteria in these addenda (Addendum C-F) must be adhered to for projects conducted under the state programs.

## 2. Summary of Method

Samples are collected in precleaned, evacuated FSL canisters or Tedlar® bags.

Samples are pre-concentrated using the Entech 7200 or 7200A Cryogenic Concentrator. A specified volume of sample is pulled using a vacuum pump through a mass flow controller. The sample is cryogenically concentrated to a volume of less than one mL on a Tenax® trap.

Following pre-concentration, the sample is refocused on the GC transfer line. This step further reduces the sample volume to less than one microliter for injection.

The sample is then injected into the GC, which is used to separate the compounds of interest. All compounds are detected using an MSD.

### 2.1 Method Modifications from Reference

Initial Calibration modifications: If a target analyte cannot meet the %RSD criteria for relative response factor calibration, then linear regression may be used. A minimum of five calibration points must be incorporated and a correlation coefficient of 0.995 or greater must be achieved. The calibration plot must be printed and approval by a supervisor must be obtained prior to calibration acceptance. If any compound is calibrated using linear regression then after the ICV and prior to any sample analysis, a low point standard must be analyzed to confirm there is no bias resulting from the linear regression calibration used. Recovery of the low point standard must be 60-140% using the linear regression curve.

Continuing calibration and laboratory controlled spike (LCS) modifications: The recoveries of all analytes must be within 70% to 130% of the true value. If more than 10% of the compounds fail these criteria, or if one compound has a recovery less than 50% or greater than 150% the LCS must be re-analyzed. If failure occurs a second time, the instrument must be re-calibrated. Recoveries greater than 150% may be acceptable, provided analytes are not detected in the samples.

Initial Calibration Verification modification: Two analytes are allowed to be greater than 30% RSD, but less than 40%.

Sample Duplicate modifications: Up to 10% of the target analyte detections may exceed acceptance criteria. If more variation occurs, the sample analysis must be repeated. If an analyte detected in one of the analysis at >5x the reporting limit, and not detected in the duplicate analysis, the analysis must be repeated. If an analyte is detected in one analysis at <5x the reporting limit and not detected in the duplicate analysis, the RPD is not calculable (NC) and the analysis does not have to be repeated. If an analyte is not detected in both the original and duplicate analysis, the RPD is NC.

Section 8.4.1.2 of the TO-15 method requires all canisters to be leak checked for a period of 24 hr via pressurization of the canister. The laboratory conducts the leak check by measuring the vacuum of the canister after a minimum of a 24 hr. period has elapsed, not by pressurizing the canister as per the method.

The % RSD for any analyte must be < 30%, as outlined in Section 10.2.2.7 of this SOP.

Humidified nitrogen is used in place of zero air due to the frequency of detection of VOCs in zero air, particularly at SIM detection limits.

There is no NIST-traceable second source standard currently available for the analytes listed in Table 1, Table 3B, and Table A-7.

## 3. Reporting Limits

Table 9 lists target analytes and Reported Detection Limit information.

## 4. Interferences

**4.1** Contamination may occur in the sampling system if canisters are not properly cleaned before use. Additionally, all other sampling equipment (e.g., pump and flow controllers)

must be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples.

**4.2** System carryover can be a potential problem, particularly for the heavier molecular weight hydrocarbons. Carryover can occur after the analysis of standards or high-level samples. Measures that must be taken to remove this contamination can include the analysis of multiple blanks, lab air, and the purging of the autosampler with nitrogen.

**4.3** High moisture content, methane levels and/or carbon dioxide levels may interfere with the chromatography and trapping of target analytes. Dilutions may be performed on these samples; however, the reporting limits will then be elevated.

# 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 employees performing laboratory procedures must have read and understood the Alpha Analytical Chemical Hygiene Plan. All laboratory procedures must be performed in accordance with the provisions and policies of the manual. All accidents, injuries, spills, or unsafe conditions must be reported immediately to the laboratory manager, and such occurrences must be thoroughly documented.

The analyst must wear a lab coat, gloves, and safety glasses while preparing solutions or handling samples.

Preparation of liquid standards must be performed under a properly functioning fume hood. Preparation and venting of gaseous standards must also be performed under a properly functioning fume hood.

# 6. Sample Collection, Preservation, Shipping and Handling

## 6.1 Sample Collection

**6.1.1** FSL canister samples can be collected as grab samples or as time-integrated samples. Time-integrated samples can be collected for a maximum of 12 hours using 2.7-liter canisters, or a maximum of 24 hours to 7 days using 6-liter canisters. One liter canisters are typically used for soil vapor sampling with a sampling flowrate of 100-200 ml/min.

**6.1.1.1** Grab samples are collected by opening the canister valve and allowing the canister to fill to ambient pressure. This process takes approximately one minute.

**6.1.1.2** Time-integrated samples require the use of a properly calibrated flow controller. The flow controller, if provided by Alpha, is calibrated prior to

sample collection and is documented in the Alpha ACS LIMs (Refer to Alpha SOP # 2190 for Canister and Flow Controller Preparation).

**6.1.2** Tedlar® bag samples typically can be collected as grab or composite samples and may require a pumping system or evacuated box.

**6.1.3** Upon receipt at the laboratory, all samples are assigned unique laboratory identification numbers, checked for possible discrepancies, etc. (See SOP # 1559.)

## 6.2 Sample Preservation

Canisters-None. Tedlar® bags-should be protected from light.

## 6.3 Sample Shipping

All samples must be accompanied by a chain of custody form, which documents the date, and time of sample collection.

## 6.4 Sample Handling

The pressure of all FSL canister samples is measured upon receipt at the laboratory and documented in the ACS LIMs (See Alpha SOP #2190). A pressure gauge is attached to the canister inlet, the canister valve is briefly opened and the pressure is recorded. The gauge apparatus used to measure ambient air samples must be separate from that used to measure soil vapor or other matrices known to have elevated levels of VOCs to avoid cross-contamination.

Samples with pressures greater than -15 inches Hg are considered acceptable for analysis.

Samples with less than -15 inches Hg should be pressurized to > -15 inches Hg in order for the concentrator system to accurately draw the correct volume, resulting in a dilution of the sample. For ambient air samples, the client must be notified prior to sample analysis since this dilution may cause reporting limits to be elevated above project action levels.

Any samples that undergo pressurization prior to analysis are documented in the instrument software. Refer to Section 10.3.3.6 for the calculation of dilution factors due to pressurization of samples.

Refer to SOP # 1559 for Sample Management information.

FSL canister and Tedlar® bag samples are stored in the Volatiles Laboratory until analysis has been completed. Tedlar® bag samples are stored in opaque containers.

The recommended holding time for the analysis of FSL canister samples for TO-15 is 30 days from date of collection. The recommended holding time for the analysis of Tedlar® bag samples for TO-15 is 48-72 hours from date of collection. Tedlar® bag samples requiring TO-15 analysis may be transferred into canisters upon receipt at the laboratory in order to extend the holding time of the sample to 30 days.

Samples designated by client to be held for subsequent analyses or are “on hold” are to be kept in a designated area in the laboratory. “Hold” samples are discarded upon client authorization or after holding time expiration date.

# 7. Equipment and Supplies

**7.1 Microliter syringes:** 10, 25,and 500 µL

**7.2 Gas tight syringes:** 1 mL, 5 mL, 25 mL, 50 mL, and 100 mL

**7.3 FSL canisters**: 1.0, 2.7, 6.0 and 15 Liter

**7.4 Tedlar® bags:** Various sizes. Alpha supplies 5-Liter sizes. All bags must have polypropylene fittings which are recommended for the analysis of Sulfides and Mercaptans (see App. A).

**7.5 Stop watch**

**7.6 Sample Concentrator**

**7.6.1** The concentrator system consists of two separate pieces of equipment: (1) Entech Model 7016D VOC Autosampler, and (2) Entech Model 7200 or 7200A Cryogenic Concentrator using liquid nitrogen.

**7.6.2** A vacuum pump (Vaccubrand Model ME2 or similar) delivers the sample from the autosampler to the cryogenic concentrator FSL-lined steel tubing.

**7.7 Gas Chromatograph System**

**7.7.1** Gas chromatograph - Shimadzu 2010, 2030

**7.7.2** Chromatographic column: Restek RTX-1; 60 meters, 0.25 mm or 0.32 mm ID, 1 micron film thickness

**7.7.3** Transfer line from column to GC injection port: Hydroguard ™ 0.32 mm capillary tubing connected to column with Restek Vu-Union connector.

**7.8 Mass Spectrometer System**

**7.8.1** Mass spectrometer - Shimadzu 2010, 2020

**7.8.2** The mass spectrometer must be capable of scanning from 29 to 270 amu every 3 seconds or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum that meets all the criteria in Table 5 when 50 ng of 4- bromofluorobenzene is injected. For SIM (selective ion monitoring) analysis, the system must be capable of simultaneous SIM/full scan acquisition.

**7.8.3** Data System - Shimadzu GC/MS Solutions software for data acquisition and Agilent Enviroquant version E.02.00 for data processing.

**7.9 Dilution Systems**

**7.9.1** Entech 4600A Dynamic Dilution System- for performing sample dilutions in canisters and Entech 4700 Precision Diluter preparing calibration standards in canisters. .

**7.10 Primary flow measurement device:** BIOS Cell Defender 510 or equivalent

# 8. Reagents and Standards

## 8.1 DI Water or Carbon-filtered tap water

## 8.2 High purity purge and trap grade methanol (Fisher part # A453-1 or equivalent) for MS source cleaning

## 8.3 Ultra high purity (UHP) helium for the GC/MS system

## 8.4 Ultra high purity (UHP) nitrogen for standard preparation

## 8.5 NIST certified TO-15 gas standards, purchased from Linde (formerly Spectra Gases). Standards are stored at room temperature and expire per vendor's expiration date, unless re-certified. Recertified standards are received with an updated certificate of analysis which includes a new expiration date.

## 8.6 Neat chemicals: Listed in Table 1, Table A-1, and Table 3B, ≥ 98% purity.

## 8.7 Liquid nitrogen: For the concentrator system and/or GC cooling

## 8.8 Primary Standards

**8.8.1** Primary standard mixtures of TO-15 analytes are purchased certified gaseous standards already prepared as well as gaseous standards prepared in the laboratory by injecting neat chemicals into Tedlar® bags (See Table 1).

**8.8.2** Table 1 indicates volumes of neat chemicals that are injected into 20 L of zero air or UHP nitrogen to obtain primary standard concentrations for all analytes.

**8.8.3** Purchased primary standards are assigned a CSS # (commercially supplied standard) upon receipt for tracking purposes. Preparation of primary standards must be entered into the primary standard preparation logbook (Form No.: 117-11).

**8.8.4** Standards are valid per the manufacturer's expiration date as noted.

## 8.9 Secondary Standards

**8.9.1** Prepare secondary standards in canisters using the Entech 4700 Precision Diluter at a minimum of two concentration levels. Table 3A and 3B outlines the preparation steps for each secondary standard.

**8.9.2** Prior to preparation of the standards, verify that an appropriate vacuum exists in the canister (>0.5 psia). Figure 3 demonstrates the standard preparation system. Primary standards prepared in Tedlar bags are injected into a canister (typically

15 L) using an injection tee with a septum or transferred from purchased and / or prepared cylinders via the precision diluter.

**8.9.3** Attach the transfer lines from the primary standards to the dynamic diluter.

**8.9.4** Prior to the injection of the gaseous standards, allow the dynamic diluter to equilibrate for a approximately 25 minutes by allowing each standard channel to flush for a minimum of 5 min. Be sure the vent line is attached to the outlet.

**8.9.5** After equilibrating the system, attach the canister to the outlet of the diluter. Flush line with nitrogen for 20 sec prior to attaching canister. Load in the appropriate configuration and standard preparation method (TO15 ICAL, DIAZ, or TO15 LCS) into the precision dilution software for the corresponding target concentration (TO15 ICAL 100 ppbV, TO15 ICAL 10 ppbV).

**Equation 1:** Flow rate calculation:

$$T_f = V_{std} / F_{std}$$

Where:

$T_f$ = standard transfer time, minutes
$V_{std}$ = standard volume, mL
$F_{std}$ = standard flowrate, mL/min

**8.9.6** Inject the appropriate amount of Tedlar bag primary standard and/or the low vapor pressure compounds listed in Table 3B into the injection port tee. This injection must be done while the canister is below atmospheric pressure.

**8.9.7** When all the primary standards have been added to the canister, the canister will be pressurized to the programmed final pressure using the precision diluter.

**NOTE:** Standard canisters prepared for analysis using the autosampler must have a maximum pressure of 30 psia to ensure proper and consistent sampling by the instrument.

**8.9.8** Label the canister accordingly and record the standard preparation in the secondary standard (SS) preparation logbook (Form No.: 12925).

**8.9.9** The ICV/LCS standard is prepared in the same manner, using primary standards of differing lot #s, at a concentration of 10 ppbV

Secondary standards are valid for 6 months.

## 8.10 Internal Standard and BFB Tuning Standard/Surrogate Standard

An internal standard (Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-D5) and tuning/surrogate standard containing Toluene-D8, 1,2-Dichloroethane-D4, and Bromofluorobenzene (BFB) are prepared as two separate gas standards at 25 ppbV (2

year expiration date). The internal standards and BFB / surrogates are loaded onto the sample trap prior to the calibration standard(s), sample, or QC sample(s) via a mass flow controller. The concentration of the internal standard and BFB / surrogates added is based upon the nominal concentration of sample that is analyzed. If the nominal volume of sample is 250 mL, then 100 mL of the 25 ppbV internal standard mix will yield a true value of 10 ppbV for the internal standards and BFB. Using equation 7, the ug/$m^3$ equivalent of BFB injected 179 ug/$m^3$ or 179 ng/L (MW of BFB = 175). Thus, the total ng injected is:

Total ng = 179 ng/L x 0.100 L = 17.9 ng

### 8.11 Instrument Calibration Standards

Calibration standards are prepared by injecting different volumes of the secondary standards into the concentrator/GC/MS system. The low standard will be used to establish the reporting limit for sample analyses. These are described in more detail in Section 10.

## 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 limits may also be found in the Laboratory Information Management System(LIMs)

At a minimum, for each day of analysis, a Continuing Calibration standard, Laboratory Method Blank, Laboratory Control Spike and Laboratory Duplicate must be analyzed. Laboratory Control Spike Duplicate (LCSD) will be analyzed only upon client request.

### 9.1 Laboratory Method Blank(s)

A FSL canister pressurized to 30 psia with humidified nitrogen is utilized as the Laboratory Method Blank. This method blank must be free of target analyte contamination at or above the reporting limit. If it is not, the system must be evaluated for possible sources of contamination. Once the source is determined and eliminated, the Blank must be reanalyzed.

A Laboratory Method Blank must be run after samples suspected of being highly contaminated to determine if sample carryover has occurred. If samples have been analyzed using an autosampler, data must be evaluated for potential carryover and re-analyses conducted, as appropriate.

### 9.2 Laboratory Control Sample (LCS) / Laboratory Control Spike Duplicate (LCSD)

**NOTE:** A Laboratory Control Spike Duplicate is only performed when specified by the project requirements and/or upon Client request.

**Laboratory Control Spike -** A Laboratory Control Spike (LCS) is prepared by spiking an evacuated FSL canister with a different primary standard solution than that used for the calibration or a purchased gaseous standard with the components of interest may be used. If the recovery is not within acceptance criteria, the LCS may be analyzed a second time. If the LCS failure continues, the instrument must be recalibrated. Refer to Section 12 for appropriate corrective actions to be taken. QC limits are subject to change for any particular analyte, if deemed necessary after review of QC control limits.

### 9.3 Initial Calibration Verification (ICV)

A mid-range calibration standard must be analyzed after the initial calibration and prior to sample analysis, and must be a different source than that used for the initial calibration. See section 10.4.1 for additional criteria. Otherwise, sample analysis may proceed.

## 9.4 Continuing Calibration Verification (CCV)

A mid-range calibration standard must be analyzed prior to sample analysis. This standard is of a different source than that used for the LCS/LCSD pair, typically the same source as the initial calibration standards. See section 10.4. or additional criteria. If repeated failure of the CCV occurs, the instrument must be recalibrated. Otherwise, sample analysis may proceed. The LCS may also be utilized as the continuing calibration unless otherwise defined in an addendum.

## 9.5 Matrix Spike

Not applicable.

## 9.6 Laboratory Duplicate

Laboratory Duplicate is a replicate analysis of a sample. The RPD of duplicate analyses must not exceed 25. Up to 10% of the target analyte detections may exceed acceptance criteria. The criteria does not need to be applied to concentrations less than 5X the reporting limit. If more variation occurs, the sample analysis must be repeated. If an analyte is detected in one analysis at >5x the reporting limit and not detected in the duplicate analysis, the analysis must be repeated. If an analyte is detected in one analysis at <5x the reporting limit and not detected in the duplicate analysis, the RPD is not calculable (NC) and the analysis does not have to be repeated. If an analyte is not detected in both the original and duplicate analyses, the RPD is NC. Equation 9 is used to calculate the RPD.
The sample chosen for duplicate analysis should not be a trip blank, field blank, or equipment blank. The sample chosen for duplicate analysis must be rotated among clients and/or sites. If possible, field duplicates should not be chosen for duplicate analysis, nor should outside air samples if indoor air samples are also included in the analytical batch. Refer to Appendices D - F for agency –specific criteria on laboratory duplicate criteria.

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**Equation 9**: RPD Calculation

$$\mathbf{RPD = ABS(C_s - C_d) / [(C_s + C_d)/2]*100}$$

where:

RPD = relative percent difference

$C_s$ = concentration in original sample analysis
$C_d$ = concentration in duplicate sample analysis

## 9.7 Method-specific Quality Control Samples

**9.7.1 BFB Tune -** A successful BFB spectrum must meet the criteria in Table 5 prior to sample analysis. If a successful BFB spectrum is not obtained, the MS must be retuned and the BFB spectrum re-evaluated prior to analyzing samples.

**9.7.2 Internal Standards -** The internal standard area counts of each sample, blank, and Laboratory Control Sample are evaluated against the corresponding

continuing calibration standard. The internal standard area counts must be within 60-140% of the continuing calibration standard area counts. If the retention times of the internal standards must be within +/- 0.33 min. If the internal standards fall outside this range, the sample, blank, or Laboratory Control Sample must be reanalyzed. In addition, area counts for internal standards for continuing calibration must be within 60-140% recovery of initial calibration. Refer to Sect. 12 for contingencies on samples exhibiting internal standard recovery failures.

**9.7.3 TIC Internal Standards -** Internal standards used for the quantitation of TICs must be evaluated by comparing the total ion area counts of the internal standards in the samples to the total ion area counts of the internal standards in the blanks. The internal standard area counts must be within 50-200% of the blank area counts. If the internal standards fall outside this range, a different internal standard or an estimated internal standard total ion area must be used to quantitate the TIC. This estimate can be done by using the total ion area from a blank or a clean sample within the analytical batch.

### 9.8 Method Sequence

- BFB Tune Check
- Calibration Standards (initial) or Continuing Calibration
- Laboratory Control Sample (may be used as the ICV or CCV)
- Laboratory Control Sample Duplicate (if needed)
- Laboratory Method Blank
- Samples
- Laboratory Duplicate

Injections may be made until 24 hours after the injection used to check the BFB tune.

All analytical sequences must be recorded in the instrument software and documented in the instrument logbook (Form 117-09).

## 10. Procedure

### 10.1 Equipment Set-up

#### 10.1.1 Canister Cleaning and Certification

Refer to Alpha SOP #2190 for canister and flow controller preparation.

#### 10.1.2 Sample Preparation and Concentration

Ensure the integrity of the canister sample as described in Section 6.4. General description of procedure: A 3-stage concentration technique called Cold-Trap Dehydration is used to analyze VOC's in air. The air sample is first concentrated to about a 0.5cc volume by drawing an aliquot of sample simultaneously through a cold trap (no packing material) and then through a Tenax trap. The cold trap is then heated to 10 °C and is held there while slowly passing helium through it to transfer these compounds to the Tenax trap, leaving most of the moisture in the cold trap. Sweeping the VOC's from the first to the second trap with only 20cc of helium results in a transfer of less than 0.5 µL of water (40 mL @ 100% RH @ 10 °C) which can be easily handled by benchtop mass spectrometers. The 20 cc transfer volume also serves to flush the $CO_2$ through the Tenax trap. After transfer to the second trap, the VOC's are back-flushed while heating to be further focused on an open-tubular focusing trap (cryofocuser) for rapid injection onto the analytical column. Internal standard is added directly to the first stage cryogenic trap prior to the sample by a mass flow controller (MFC). MFC controlled introduction is advantageous over loop injection as it remains consistent with the mechanism used to measure the sample volume.

Connect the canisters or Tedlar® bag(s) to the Entech 7016D Autosampler. For FSL canisters: Align the tubing from one of the 16 positions to the canister inlet position. Push the inlet line into the orifice of the canister and hold in place while tightening the fitting finger tight. Turn the stainless steel nut ¼ turn more with a wrench. The canister valves must be closed at this point. For Tedlar® bags: Connect the valve of the Tedlar® bag with the autosampler line using an adapter fitting.

For canister samples, leak check all inlet connections using the leak check procedure included with the Entech software. The software will indicate if there is a change in vacuum over a period of 30 sec. The vacuum must not increase more than 2 psia. Analysis cannot begin until the leak check has passed for each canister being tested and/or the source of the leak has been determined..

Open the canister or bag valves.

Set up the sequence of the Entech system to withdraw 250 mL from each sample. If high concentrations are expected, lower volumes can be used ( minimum of 25 mL).. Samples suspected to contain elevated concentrations (i.e. soil vapor, sub-slab, landfill gas) should be pre-screened prior to analysis to obtain more precise dilution information. If screening results indicate elevated concentrations of non-target analytes, the sample should be diluted such that the peak height of the non-target analyte is approximately 10X greater than the peak height of the first internal standard. Recommended concentrator operating parameters are provided in Table 6.

## 10.2 Initial Calibration

### 10.2.1 GC Conditions (Shimadzu 2010, 2030)

**Oven program:** 25° C, hold for 5.0 minutes, then:

Ramp 1: 100° C at 8.0° C / min.; hold for 0.0 min

Ramp 2: 220° C at 25° C / min.; hold for 4.0 min

**Gas Flows**

Helium carrier gas flow program: 2.0 mL/min for entire run (23.18 min)

**Sample Injection**

| | |
|---|---|
| Injection mode: | split |
| Injection port temperature: | 250$^0$ C |
| Inlet pressure: | 27.3 psi |
| Total flow: | 39.3 mL/min |
| Split ratio: | 17.3 |
| Split flow: | 34.6 mL/min |
| Gas saver flow: | OFF |

### 10.2.2 MS Conditions

Temperature of MSD transfer line: 250$^0$ C

Temperature of MS Quad: 150$^0$ C

Temperature of MS Source: 230$^0$ C

Solvent Delay: 3.0 minutes

Scanning Parameters: 29-270 amu until 10 min, scan rate = 5.52 scans/sec; then 35-270 amu, scan rate = 3.1 scans/sec. Threshold = 150. Sampling rate = 2. EM offset-variable to achieve response of 200K area counts (+/- 25K) for the internal standard bromochloromethane.

### 10.2.3 Daily GC/MS Performance Check

**10.2.3.1** The first analysis of the day is typically a tune evaluation. The GC/MS system is checked to confirm that acceptable performance criteria for bromofluorobenzene (BFB), which is in surrogate mixture, are achieved. These criteria must be met prior to analyzing further standards, blanks and samples.

**10.2.3.2** A maximum injection of 50 ng must successfully meet the BFB spectrum criteria in Table 5.

**10.2.3.3** If the spectrum of BFB does not meet the above stated criteria, the analysis must be repeated. If the spectrum of BFB still does not meet these criteria, the GC/MS instrument must be re-tuned.

**10.2.3.4** The Daily GC/MS Performance Check must be analyzed every 24 hours or less.

### 10.2.4 Initial Calibration

**10.2.4.1** Analyze a minimum of five different levels by analyzing various volumes of the secondary standards prepared in Table 3 (Table A-3 for sulfide/mercaptan analysis). The lowest standard will be at or below the

reporting limit. If the response is not linear at the lowest level for the higher molecular weight compounds, this point must not be included in the calibration curve for these compounds. As a result, the analysis of more than five levels may be required in order to ensure a minimum of five calibration points for each analyte.

Table 4 lists the calibration standard levels and the volumes of the secondary standards needed to achieve these levels.

**10.2.4.2** The true value of each of these calibration points is determined by applying a dilution factor that is based on the volume of sample extracted from the canister for each calibration point. Assuming that a volume of 250 mL will be the maximum volume extracted from the samples; this will be the "1X" volume. A dilution factor can be calculated using Equation 2.

**Equation 2:** Calculation of Instrument Dilution Factor

$$\mathbf{DF = V_{1X} / V_{actual}}$$

where:

DF = dilution factor
$V_{1X}$ = maximum volume sampled, mL
$V_{actual}$ = actual volume sampled for samples and standards

**10.2.4.3** Analyze each calibration standard according to the procedures specified in Section 10. The true value of each calibration point is determined by dividing the concentration of the canister by the dilution factor determined using Equation 2.

**10.2.4.4** Tabulate the area response of the characteristic ions against the amount for each analyte and internal standard and calculate relative response factors (RRF) for each compound using Equation 3. Perform this calculation for each calibration standard.

**Equation 3:** Relative Response Factor for Individual Target Analytes

$$\mathbf{RRF = [(A_{EC}) * (C_i)] / [(A_{EI}) * (C_c)]}$$

where:

RRF = relative response factor
$A_{EC}$ = area count of the extracted ion for the analyte of interest
$C_i$= amount of internal standard (ppbV)
$A_{EI}$ = area count of the extracted ion for the associated internal standard

$C_c$ = amount of analyte of interest (ppbV)

Table 7 lists all TO-15 analytes, internal standards and the associated quantitation ion.

Table 8 lists the internal standards and the associated TO-15 analytes.

**10.2.4.5** Calculate the average response factor for each of the target analytes by the following equation ($AVG_x$ = SUM(RFs) / total # of RFs).

**10.2.4.6** Calculate the percent relative standard deviation (%RSD) of the response factors over the secondary range of the curve for each of the target analytes using Equation 4.

**Equation 4:** Percent Relative Standard Deviation

$$\%RSD = [(SD_n - 1) / (AVG_x)] * 100]$$

where:

%RSD = percent relative standard deviation
$SD_n$ -1 = standard deviation (n-1 degrees of freedom)
$AVG_x$ = average response factor from the initial calibration curve

This task can also be accomplished using the quantitation software provided by the instrument manufacturer.

**10.2.4.7** If the %RSD is <30 for each analyte, linearity can be assumed for the associated target analyte and sample analysis may proceed.

If the %RSD is >30 for any analyte, the integrations must be evaluated and the calculations verified. If a %RSD <30 cannot be achieved, it is acceptable for two (2) of the analytes to be above 30%, but below 40% RSD (applies to analytes flagged with a “C” on the Enviroquant initial calibration summary table).

Alpha may use the following modified acceptance criteria only for projects that have documentation and approval within the QAPP by the quality assurance project planners, and also for analytes not listed in EPA Method TO-15:

**10.2.4.8** If the %RSD is >30 for any analyte, the integrations must be evaluated and the calculations verified. If a %RSD <30 cannot be achieved, it is acceptable for 10% of the total analytes to be above 30%, but below 50% RSD. Before acceptance of such a Calibration Curve, it must be confirmed with the approval of the Section Supervisor and/or the Project Manager that these analytes are typically and historically "trouble" analytes or "poor performers" (typically compounds listed in Table 3B), and that all Client and Project Data Quality Objectives (DQOs) will still be met when analyzing samples using this calibration. Refer to Appendices D - F for agency –specific criteria regarding initial calibration acceptance criteria.

Calibration points may be removed from the calibration curve to meet the 30% RSD criteria, so long as five consecutive points remain in the calibration curve, and the following procedure is followed:

- Remove high level calibration points
- Remove low-level calibration points; reporting limits will need to be elevated, however.

**10.2.4.9** If calibration points in the mid-level range need to be removed due to a sequence error or instrument malfunction, the entire calibration level must be removed from the calibration curve. If the %RSD >30, a calibration curve is generated using the EnviroQuant quantitation software.

Correlation Coefficient calculation is performed by the Enviroquant software as follows;

$$r = \frac{n(\Sigma xy) - (\Sigma x)(\Sigma y)}{\sqrt{[\, n\Sigma x^2 - (\Sigma x)^2 \,][\, n\Sigma y^2 - (\Sigma y)^2 \,]}}$$

Where:

r = Correlation Coefficient

n = number of standards measured

x = true concentration of the standard

y = observed concentration of the standard

The correlation coefficient (linear) for the calibration curve must be greater than 0.995. If these criteria cannot be met, prepare a new set of calibration standards and recalibrate the instrument. NOTE: Quadratic calibration in any form is not acceptable.

Authorization from the department supervisor is required prior to using linear regression calibration. Linear regression is only allowed if certain criteria listed below are met:

- The minimum number of points for a linear regression curve is five points.
- The curve must be plotted and printed and turned in with the raw data.
- A calibration standard must be analyzed at the low point of the curve. Recovery of the low point standard must be 60-140% using the linear regression curve.

  The recovery of the compound for the continuing calibration / LCS must be within 70-130%.

**10.2.4.10** The reference spectra for all target analytes are reviewed for both assignments and purity for all instruments. In addition, this process of reviewing all spectra continues whenever a new calibration is completed.

Reference spectra should be updated with each initial calibration performed with the midpoint standard of the calibration.

**10.2.4.11 Internal Standard Criteria for Initial Calibration Levels and ICV.**

The mean response for each internal standard compound is calculated over the initial calibration range. The area response at each calibration level must be within 60-140% of the midpoint area response over the initial calibration range for each internal standard. If recovery is outside the range, re-analyze calibration level. This criteria must be met prior to sample analysis.

All of these criteria must be met prior to sample analysis.

## 10.3 Equipment Operation and Sample Processing

### 10.3.1 GC/MS ANALYSIS

**10.3.1.1** The Entech 7200 or 7200A Concentrator is programmed to the specific analytical conditions listed in Table 6 (Entech method Alpha_TO15.CTD) and the GC/MS parameters are set to those listed in Sections 10.3.1 and 10.3.2. (Enviroquant method TO15-SFS.M (SIM and full scan) or TO15_FS_35C.M (full scan only, for sulfide & mercaptan analysis in App. A)).

**10.3.1.2** The BFB spectrum is evaluated by analyzing a Laboratory Method Blank and adding 100 mL of the BFB/surrogate mix.

**10.3.1.3** A continuing calibration and/or a laboratory control spike is analyzed. See sect. 9.2 for acceptable criteria, and refer to Appendices D - F for agency – specific criteria on continuing calibration and laboratory control spike criteria.

**10.3.1.4** A Laboratory Method Blank is analyzed. The Laboratory Method Blank consists of the analysis of 250 mL from a canister of humidified nitrogen. The method blank must be free of target analyte contamination at or above the reporting limit.

**10.3.1.5** A 250-mL aliquot of sample is preconcentrated on the Entech 7200 or 7200A concentrator and injected onto the GC column. For soil vapor samples, or other samples that may contain elevated levels, the aliquot amount must be determined using the results from a pre-screening analysis. Sample concentrations are determined using the following equation:

$$ppbV = [(A_{EC}) * (C_i)] / [(A_{EI}) * (RRF)]$$

**10.3.1.6** Instrument Dilutions and Sub-Atmospheric Sample dilutions

**10.3.1.6.1** For dilutions, smaller sample volumes (<250 mL) are analyzed. The smallest volume that can be analyzed with accuracy using the Entech concentrator is 10 mL. The dilution factor is accounted for by entering the volume analyzed in the sample calculation discussed in Section 10.2.2.2 (Equation 2).

**10.3.1.6.2** Samples that arrive at the laboratory with pressures below -15 inches Hg should be pressurized with nitrogen to greater than -15 inches Hg, as discussed in Section 6.4. This pressurization results in a dilution factor. The dilution factor is calculated using Equation 6, and the canister dilution spreadsheet (Form No.: 117-05). Attach a green tag to the canister with the pressurization information (initial pressure and final pressure) recorded on the tag.

**Equation 6**: Dilution Factor for Pressurization of Subatmospheric Samples: Three Steps

Step 1: Calculate the volume in the canister prior to pressurization (Assume a 2.7-liter canister is used).

$$\mathbf{V_{ci} = 2.7 * P_I / 14.696}$$

Step 2: Calculate the volume in the canister after pressurization.

$$\mathbf{V_{cf} = 2.7 * P_F / 14.696}$$

Step 3: Calculate the dilution factor.

$$\mathbf{DF = V_{cf} / V_{ci}}$$

where:

$V_{ci}$= volume of air in canister prior to pressurization, L

$P_I$ = pressure reading of canister prior to pressurization (psia)

$V_{cf}$ = volume of air in canister after pressurization, L

$P_F$ = pressure reading of canister after pressurization (psia)

DF = dilution factor

14.696 = atmospheric pressure (psia)

**10.3.1.6.3** If samples require larger dilutions than pressurization and instrument dilutions, a syringe dilution into an additional canister or Tedlar bag (typically used only for App. A analytes) with a known volume of nitrogen is required.

**10.3.1.6.4** Fit a VCO® adapter with a septa to the pressurized sample canister. With a gastight syringe remove appropriate sample size for dilution. Allow sample to flow through syringe for 1 – 2 seconds to flush syringe prior to volumizing. Inject the sample aliquot into a Tedlar bag. If using an evacuated canister, connect the canister to an injection port tee (see Figure 3) attached to the dynamic diluter. Inject the aliquot of sample while a steady stream of Nitrogen is flowing into the dilution canister. Pressurize this canister to 30 psia. Attach a green tag to the canister with dilution information recorded on the tag. Use the dilution calculation worksheet (Form No.: 117-05) to calculate resulting dilutions.

### 10.3.2 Qualitative Identifications

**10.3.2.1** An analyst competent in the interpretation of mass spectra must identify the target analytes by comparison of the sample mass spectrum to the mass spectrum of the standard. Two criteria must be satisfied to verify the identification: (1) elution of the component in the sample at the same GC relative retention time (RRT) as the component in the standard, and (2) agreement of the sample component and standard component mass spectra.

**10.3.2.2** For establishing correspondence of the GC RRT, the RRT of the component in the sample must compare within ± 0.06 RRT units of the RRT of the component in the standard. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT must be assigned using extracted ion current profiles for the ion unique to the component of interest.

**10.3.2.3** For comparison of the standard and sample component mass spectra, mass spectra of standards obtained on the GC/MS under the same instrument conditions are required. Reference spectra should be updated for each initial calibration performed, using the mid-level standard. Once obtained, these standard spectra may be used for identification and reference purposes.

**10.3.2.4** The requirements for qualitative verification by comparison of mass spectra are as follows:

- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) must be present in the sample spectrum.
- The relative intensities of ions specified must agree within ± 20% between the standard and sample spectra.
- Ions greater than 10% in the sample spectrum must be considered and accounted for by the analyst making the comparison.

Table 7 lists the primary and secondary ions for all analytes.

**10.3.2.5** Manual integrations: for peaks that are observed to be not integrated correctly by the quantitation software, manual integrations must be performed. Please refer to the manual integration SOP for further instruction on how to properly perform and document manual integrations (Alpha SOP # 1731).

**10.3.2.6** **Tentatively identified compounds (TICs)-**A library search may be performed for non-target sample components for the purpose of tentative identification, as requested by the client. Mass spectra are compared to the National Institute of Standards and Technology Mass Spectral Library (2002 version), and a qualitative match is determined by the analyst. Computer generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when

compared to each other. Refer to Appendices D - F for agency –specific criteria on TIC reporting.

**10.3.2.7** Guidelines for making tentative identification:

- Relative intensities of major ions in the reference spectrum (ions greater than 20% of the most abundant ion) must be present in the sample spectrum.
- The relative intensities of the major ions must agree within ± 30%.
- Molecular ions present in the reference spectrum must be present in the sample spectrum.
- Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of coeluting compounds.
- If, in the technical judgment of the mass spectral interpretation specialist, no valid tentative identification can be made, the compound will be reported as "Unknown". The mass spectral interpretation specialist should give additional classification of the unknown compound, if possible (i.e., unknown aromatic, unknown hydrocarbon, unknown acid, unknown chlorinated compound)..

## 10.4 Continuing Calibration

### 10.4.1 Calibration Verification

**10.4.1.1** The initial calibration must be verified through the analysis of an Initial Calibration Verification (ICV) sample. (The ICV may also be used to satisfy LCS requirements.) This analysis must be performed every time an initial calibration is performed.

**10.4.1.2** The ICV must be prepared using a purchased gaseous standard (from a different lot # or separate vendor) with the components of interest in an evacuated FSL canister. Follow the standard preparation procedure for the calibration standards outlined in Section 8.0. The standard must be prepared at or below the midpoint of the calibration curve.

See section 10.4.2.6 for acceptable criteria and Section 12 for corrective actions.

### 10.4.2 Continuing Calibration

**10.4.2.1** A continuing calibration check must be performed daily prior to sample analysis. The continuing calibration standard must be one of the initial calibration levels.

**10.4.2.2** Analyze a calibration standard that is at the midpoint of the calibration curve.

**10.4.2.3** The LCS standard may be utilized as the continuing calibration check, provided that all target analytes of interest are present in the LCS standard.. Refer to Appendices D - F for agency –specific criteria on LCS criteria.

**10.4.2.4** Evaluate continuing calibration for internal standard recoveries. Recoveries must be between 60-140%. If not, the CCV will be re-analyzed.

**10.4.2.5** Calculate the percent difference (%D) of the continuing calibration response factor from the initial calibration average response factor using Equation 5.

**Equation 5:** Percent Difference

$$\%\ D = [(C_{found})-(C_{true}) / (C_{true})] * 100$$

where:

%D = percent difference

$C_{found}$ = amount of the analyte detected in the standard (ppbV)
$C_{true}$ = true amount of the analyte in the standard (ppbV)

This task can also be accomplished using the quantitation software provided by the instrument manufacturer.

**10.4.2.6 Acceptance Criteria**

The acceptance criteria is less than 30% RSD for any analyte, with an allowance of two analytes to be greater than 30%, but less than 40%.

Alpha may use the following modified acceptance criteria only for projects that have documentation and approval within the QAPP by the quality assurance project planners, and also for analytes not listed in EPA Method TO-15:

If the %RSD is <30 for each analyte, linearity can be assumed for the associated target analyte and sample analysis may proceed.

**10.4.2.7** If the %RSD is >30 for any analyte, the integrations must be evaluated and the calculations verified. If a %RSD <30 cannot be achieved, it is acceptable for 10% of the total analytes to be above 30%, but below 50% RSD. Before acceptance of such a Calibration Curve, it must be confirmed with the approval of the Section Supervisor and/or the Project Manager that these analytes are typically and historically "trouble" analytes or "poor performers" (typically compounds listed in Table 3B), and that all Client and Project Data Quality Objectives (DQOs) will still be met when

analyzing samples using this calibration. . Refer to Appendices D - F for agency –specific criteria on initial calibration acceptance criteria.

**10.4.2.8** Refer to Sect. 12 for additional procedures regarding continuing calibration acceptance criteria and corrective actions if criteria are not met.

### 10.5 Preventive Maintenance

Mass flow controllers should be checked annually for flowrate accuracy using a BIOS cell or other primary flow measurement device.

Ion source cleaning – typically prior to initial calibration.

Electron Multiplier (EM)-changed when the voltage setting required to achieve adequate response approaches 1900.

Rough pump oil changed annually.

Transfer lines, concentrator traps, and the GC guard column should be changed semi-annually, or when system repeatedly fails initial calibration.

## 11. Data Evaluation, Calculations and Reporting

### 11.1 Calculations

**11.1.1 Individual Target Analytes:** The average response factor from the initial calibration is used to calculate the amount of analyte detected in the sample analyses. Standards are prepared on a ppbV basis, so if no dilution is performed, values can be reported from the quantitation report without any calculations. Dilution factors are calculated using Equation 2. Equation 7 shows the conversion of ppbV to µg/$m^3$ .

**Equation 7**: Conversion of ppbV to µg/$m^3$

**µg/$m^3$ = (ppbV) * MW / 24.45**

where:

24.45 = molar gas constant (g/g-mole)
MW = molecular weight of the compound of interest (Table 1 and 2 lists the molecular weights of the target analytes)

**11.1.2 TICS:** An estimated amount for the TIC is calculated using the total area of the TIC, the total area of the internal standard assigned by the quantitation software, and a response factor of 1.000 (Equation 8). If the internal standard assigned by the quantitation software exhibits significant interference from other analytes, the next closest eluting internal standard will be utilized. Refer to Appendices D – F for agency-specific criteria on reporting TICS.

**Equation 8:** Calculation of TIC Results in ppbV

**$ppbV = [(A_T) * (C_{IS})] / [(A_{IS-T}) * (1.000)]$**

where:

$A_T$ = total ion area of the TIC to be measured

$C_{IS}$ = amount of the internal standard

$A_{IS-T}$ = total ion area of the closest eluting internal standard

The integration of target analytes and internal standards must be performed from valley to valley.

#### 11.1.3 Percent Recovery

$$\% \text{ Recovery} = \frac{C_x}{C_t} \text{ X } 100$$

where:
$C_x$ = measured concentration of compound
$C_t$ = true concentration of compound

## 11.2 Data Package

#### 11.2.1 Canister Cleaning Information

*A copy of the data for the batch certification analysis associated with the FSL canisters must be on file. The raw data must include a sample chromatogram, quantitation report, and spectra of all positive results.*

#### 11.2.2 BFB Tune Checks

Tune checks must be included for all days of analysis, including initial calibration. Raw data must include the chromatogram, mass spectra, and summary of relative abundances of the BFB ions.

#### 11.2.3 Calibration Data

- Initial calibration summary (including average response factors, %RSDs, and copies of calibration curves, if appropriate) for target analytes and all calibration chromatograms must be on file.
- Continuing calibration summaries (including %Ds) for individual analytes.
- Chromatograms and quantitation reports associated with all standards used, in the initial and continuing calibrations.

#### 11.2.4 QA/QC

- Internal standard responses and % recoveries vs. the continuing calibration.
- Quantitation report and chromatogram for laboratory control spike (and laboratory control spike duplicate, if requested).

- Quantitation reports, chromatograms, and spectra of positive results for all blanks.
- Copy of the instrument runlog.

#### 11.2.5 Sample Data

- Quantitation reports, chromatograms, spectra of positive results, negative proofs, and pre- and post-manual integrations for all LCSs, samples, and duplicates.
- A copy of the canister dilution worksheet, Form No.: 117-05 (if any canister pressurizations or canister dilutions are performed).

# 12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

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 indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation.

Holding time exceedence and/or container damage is noted on the Sample Delivery Group 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. The method blank must be free of target analyte contamination at or above the reporting limit. If it is not, the system must be evaluated for possible sources of contamination. Once the source is determined and eliminated, the method blank must be reanalyzed.

If the ICV, CCV, LCS or LCSD 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 Narratives. Immediate corrective action includes reanalyzing all affected samples (provided sufficient volume of sample remains) by using any retained sample before the expiration of the holding time. If sufficient volume is not available, Alpha will narrate accordingly. Analytes that fail ICV, CCV and LCS criteria due to elevated recoveries may be reported, provided there are no detections in the associated samples.

Samples exhibiting internal standard recovery failures must be re-analyzed at the same dilution level if instrument malfunction is suspected to be the cause, or at a lesser dilution if sample matrix or concentration levels of target and/or non-target analytes are suspected of being the cause. If recovery failures are observed upon re-analysis, narrate bias accordingly (negative if high recovery, positive if low recovery).

When applicable criteria is not met (ICV, CCV, LCS, IS), Alpha will narrate bias which will be included in the opening narrative of 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 # 1797 Hazardous Waste & Sample Disposal

SOP # 1731 Manual Integration

Form 117-05: Canister Dilution Worksheet Template

Form 117-09: Instrument Run Log

Form 117-11: Primary Standard Preparation Log

Form 12925: Secondary Standard Preparation Log

## 16. Attachments

**Table 1** TO-15 Tedlar® Bag Stock Standard Preparation for LCS

**Table 2A** TO-14 Primary Mix #1 & TO-15 Mix #2 Stock Standard Cylinder

**Table 2B** TO-15 Custom Mix #3 Stock Standard Cylinder

**Table 2C** TO-15 Custom Mix #4 Stock Standard Cylinder

**Table 2D** TO-15 Custom Mix #5 Stock Standard Cylinder

**Table 3A** Summary of Working Standard Preparation

**Table 3B** Preparation of Calibration Standards for Low Vapor Pressure Compounds

**Table 4** Calibration Standard Levels

**Table 5** BFB Key Ions and Abundance Criteria

**Table 6** Entech 7200 or 7200A /7016D Operating Parameters

**Table 7** Quantitation and Secondary Ions for TO-15 Analytes and Internal Standards

**Table 8** Internal Standards and the Associated Target Analytes

**Table 9A** TO-15 Target Analytes and Reporting Limits-Standard List

**Table 9B** TO-15 Target Analytes and Reporting Limits-Additional Analytes

**Appendix A** Cold Trap Dehydration technique (CTD) for Analysis of Sulifides and Mercaptans

**Appendix B** Data Acquisition Parameters and Analysis Modifications for Conducting SIM Analysis

**Appendix C** Modifications to Data Review and Case Narrative to Comply with MADEP MCP-TO-15 Method

**Appendix E** Modifications to Data Review and Case Narrative to Comply with 2014 NJDEP Technical Guidance for EPA Method TO-15

**Appendix F** Modifications to Data Review and Case Narrative to Comply with Ohio EPA Voluntary Action Program (VAP) Requirements for EPA Method TO-15

**DEFINITIONS**

**Absolute canister pressure -** Pg + Pa, where Pg = gauge pressure in the canister (psig) and Pa = barometric pressure.

**Absolute pressure -** Pressure measured with reference to absolute zero pressure (as opposed to atmospheric pressure), usually expressed as kPA, mm Hg, or psia (pounds per square inch absolute).

**Cryogen -** The refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen (bp = -196$^0$C).

**Gauge pressure -** Pressure measured above atmospheric pressure (as opposed to absolute pressure). Zero gauge is equal to ambient atmospheric (barometric) pressure. Units = psig (pounds per square inch gauge).

**ppmV –** parts per million on a volume basis.

**ppbV** – parts per billion on a volume basis

**psia –** pounds per square inch absolute

**Relative retention time (RRT)–** retention time (RT) ratio of the target analyte and the internal standard used to quantitate (RT target / RT internal standard).

## Table 1

## TO-15 Tedlar® Bag Stock Standard Preparation- LCS/ICV Standard

| ICV / LCS Standard | | | | |
|---|---|---|---|---|
| **COMPOUND (liquids)** | **MOL WGT** | **Density ug/uL** | **uL injected*** | **FINAL ppmV** |
| Acetone | 58.1 | 791 | 12.0 | 200 |
| Isopropyl alcohol | 60.1 | 785 | 4.8 | 76.7 |

All neat chemicals are injected into a Tedlar® bag containing 20 Liters of zero air or UHP nitrogen.

See Table A-1 & Table A-4 for sulfide/mercaptan stock standard preparation

## Table 2A

## TO-15 Purchased Primary Standard Mix

### TO-14 Primary Standard Mix #1

| COMPOUND | MOL WGT | Conc. ppmV | COMPOUND | MOL WGT | Conc. ppmV |
|---|---|---|---|---|---|
| dichlorodifluoromethane | 120.92 | 1.0 | cis-1,3-dichloropropene | 110.97 | 1.0 |
| chloromethane | 50.49 | 1.0 | trans-1,3-dichloropropene | 110.97 | 1.0 |
| Freon-114 | 170.92 | 1.0 | 1,1,2-trichloroethane | 133.41 | 1.0 |
| vinyl chloride | 62.5 | 1.0 | toluene | 92.14 | 1.0 |
| bromomethane | 94.94 | 1.0 | 1,2-dibromoethane | 187.87 | 1.0 |
| chloroethane | 64.52 | 1.0 | tetrachloroethene | 165.83 | 1.0 |
| trichlorofluoromethane | 137.37 | 1.0 | chlorobenzene | 112.56 | 1.0 |
| 1,1-dichloroethene | 96.94 | 1.0 | ethylbenzene | 106.17 | 1.0 |
| methylene chloride | 84.93 | 1.0 | m-xylene | 106.17 | 1.0 |
| Freon-113 | 187.38 | 1.0 | p-xylene | 106.17 | 1.0 |
| trans-1,2-dichloroethene | 98.96 | 1.0 | styrene | 104.15 | 1.0 |
| 1,1-dichloroethane | 98.96 | 1.0 | 1,1,2,2-tetrachloroethane | 167.85 | 1.0 |
| cis-1,2-dichloroethene | 96.94 | 1.0 | o-xylene | 106.17 | 1.0 |
| chloroform | 119.38 | 1.0 | 1,3,5-trimethylbenzene | 120.2 | 1.0 |
| 1,2-dichloroethane | 98.96 | 1.0 | 1,2,4-trimethylbenzene | 120.2 | 1.0 |
| 1,1,1-trichloroethane | 133.41 | 1.0 | 1,3-dichlorobenzene | 147.0 | 1.0 |
| benzene | 78.11 | 1.0 | 1,4-dichlorobenzene | 147.0 | 1.0 |
| carbon tetrachloride | 153.82 | 1.0 | 1,2-dichlorobenzene | 147.0 | 1.0 |
| 1,2-dichloropropane | 113 | 1.0 | 1,2,4-trichlorobenzene | 181.45 | 1.0 |
| trichloroethene | 131.38 | 1.0 | hexachlorobutadiene | 260.76 | 1.0 |

### TO-15 Primary Standard Mix #2

| COMPOUND | MOL WGT | Conc. ppmV |
|---|---|---|
| Propylene | 42.08 | 1.0 |
| 1,3-butadiene | 54.09 | 1.0 |
| Vinyl bromide | 106.96 | 1.0 |
| Acetone | 58.08 | 1.0 |
| Isopropyl alcohol | 60.1 | 1.0 |
| Carbon disulfide | 76.14 | 1.0 |
| 3-chloropropene | 76.53 | 1.0 |
| Trans-1,2-dichloroethene | 96.94 | 1.0 |
| Methyl-tert butyl ether | 88.15 | 1.0 |
| Vinyl acetate | 86.09 | 1.0 |
| 2-butanone (MEK) | 72.11 | 1.0 |
| Hexane | 86.18 | 1.0 |
| Ethyl acetate | 88.11 | 1.0 |
| Tetrahydrofuran | 72.11 | 1.0 |
| Cyclohexane | 84.16 | 1.0 |
| Bromodichloromethane | 163.83 | 1.0 |
| 1,4-dioxane | 88.11 | 1.0 |
| 2,2,4-trimethylpentane | 114.23 | 1.0 |
| Heptane | 100.21 | 1.0 |
| 4-methyl-2-pentanone (MIBK) | 100.16 | 1.0 |
| 2-hexanone | 100.16 | 1.0 |
| Dibromochloromethane | 208.29 | 1.0 |
| Bromoform | 252.75 | 1.0 |
| Benzyl chloride | 126.59 | 1.0 |
| 4-ethyl toluene | 120.2 | 1.0 |

## Table 2B

## TO-15 Purchased Custom Mix #3

| TO-15 Custom Standard Mix | | | | | |
|---|---|---|---|---|---|
| **COMPOUND** | **MOL WGT** | **Conc. ppmV** | **COMPOUND** | **MOL WGT** | **Conc. ppmV** |
| Propane | 44.10 | 1.0 | n-Octane | 114.23 | 1.0 |
| Chlorodifluoromethane | 86.47 | 1.0 | 1,1,1,2-Tetrachloroethane | 167.85 | 1.0 |
| Methanol | 32.04 | 5.0 | 1,2,3-Trichloropropane | 147.43 | 1.0 |
| n-Butane | 58.12 | 1.0 | Nonane | 128.26 | 1.0 |
| Dichlorofluoromethane | 102.92 | 1.0 | Isopropylbenzene | 120.19 | 1.0 |
| Ethanol | 46.07 | 5.0 | Bromobenzene | 157.01 | 1.0 |
| Acetonitrile | 41.05 | 1.0 | 2-Chlorotoluene | 126.58 | 1.0 |
| Acrolein | 56.10 | 1.0 | n-Propylbenzene | 120.19 | 1.0 |
| n-Pentane | 72.20 | 1.0 | 4-Chlorotoluene | 126.58 | 1.0 |
| Acrylonitrile | 53.10 | 1.0 | tert-Butylbenzene | 134.20 | 1.0 |
| Ethyl Ether | 74.12 | 1.0 | n-Decane | 142.28 | 1.0 |
| tert-Butyl Alcohol | 74.12 | 1.0 | sec-Butylbenzene | 134.20 | 1.0 |
| 2,2-Dichloropropane | 112.99 | 1.0 | p-Isopropyltoluene | 134.22 | 1.0 |
| Di-Isopropyl Ether | 102.17 | 1.0 | n-Butylbenzene | 134.20 | 1.0 |
| Tert-Butyl Ethyl Ether | 102.20 | 1.0 | 1,2-Dibromo-3-chloropropane | 236.33 | 1.0 |
| 1,1-Dichloropropene | 110.97 | 1.0 | n-Undecane | 156.31 | 1.0 |
| Tert Amyl Methyl Ether | 102.17 | 1.0 | Naphthalene | 128.17 | 1.0 |
| Dibromomethane | 173.83 | 1.0 | n-Dodecane | 170.33 | 1.0 |
| 1,3-Dichloropropane | 112.99 | 1.0 | 1,2,3-Trichlorobenzene | 181.45 | 1.0 |
| n-Butyl Acetate | 116.16 | 1.0 | | | |

All mixes currently purchased fromPraxair (formerly Linde and Spectra Gases)

## Table 2C

## TO-15 / PIANO Custom Mix #4

| COMPOUND (liquids) | Boiling Pt, deg C | Molecular Weight | Density ug/uL | uL injected* | FINAL ppmV |
|---|---|---|---|---|---|
| 1,1-difluoroethane (gas) | -24.7 | 66.05 | NA | 10 mL | 5.0 |
| 1-ethyl-1-methylcyclopentane | 121.55 | 112.21 | 785.4 | 11.9 | 1.0 |
| Indan | 177 | 118.18 | 965.0 | 10.2 | 1.0 |
| Indene | 182 | 116.16 | 996.0 | 9.7 | 1.0 |
| 1,2,3-trimethylbenzene | 176 | 120.19 | 890.0 | 11.2 | 1.0 |
| Acetaldehyde | 20.2 | 44.05 | 785 | 23.3 | 5.0 |
| Acetone | 56.1 | 58.08 | 784.5 | 24.6 | 4.0 |
| isopropyl alcohol | 82.6 | 60.1 | 786 | 9.5 | 1.5 |
| methyl methacrylate | 101 | 100.1 | 940 | 8.8 | 1.0 |

## Table 2D

## TO-15 / PIANO Custom Mix #5

| COMPOUND (liquids) | Boiling Pt, deg C | Molecular Weight | Density ug/uL | uL injected* | FINAL ppmV |
|---|---|---|---|---|---|
| Thiophene | 83 | 84.14 | 1060 | 6.6 | 1.0 |
| 2-Ethylthiophene | 133 | 112.19 | 990 | 9.4 | 1.0 |
| 2-methyllthiophene | 114 | 98.17 | 1016 | 8.0 | 1.0 |
| 3-Methylthiophene | 113 | 98.17 | 1017 | 8.0 | 1.0 |

* both mixes #4 and #5 prepared in 29.5 L high pressure cylinders, pressurized to 995 psia with UHP nitrogen. Total volume of standard is 1997 L. Two year expiration date.

## Table 3A

## Summary of Secondary Standards Preparation

| Primary Standard | Primary Standard Conc. ppmV | Volume of Primary Standard Injected into canister | Primary Standard Transfer Method | Final Volume canister (L) | Final Concentration ppbV ** |
|---|---|---|---|---|---|
| **Secondary standards prepared using precision dilution system (Entech 4700)** | | | | | |
| | | | | | |
| TO-15 Mixes #1 - #5 | 1.0 | 3000 mL | Pressure differential | 30 | 100 |
| | | | | | |
| TO-15 Mixes #1 - #5 | 1.0 | 300 mL | Pressure differential | 51 | 10 *** |
| **Secondary standards prepared via serial dilution** | | | | | |
| 100 ppbV ICAL mix | 1.0 | 300 mL | Syringe Injection | 30 | 1.0 |
| 100 ppbV ICAL mix | 0.1 * | 30 mL | Syringe Injection | 30 | 0.1 |

All standards prepared using humidified nitrogen.

* This calibration standard is used for TO-15 SIM analysis only (see Appendix B).

** The following analytes have concentrations that are greater than the listed concentration

Methanol, acetylaldehyde, and ethanol – 5 times greater

m&p-xylene- 2 times greater

Isopropyl alcohol-2.5 times greater

*** 10 ppbV standards that are prepared at a pressure of 50 psia are split out into additional empty 15L canisters to reduce the pressure in the standard and allow for additional canisters to be prepared for continuing calibration usage.

## Table 3B

## Preparation of Calibration Standards for Low Vapor Pressure Compounds

| COMPOUND (solids) | Vapor Pressure* (P), atm | Molecular Weight | Volume (V) extracted, mL | Gas Constant (R) (L atm/gm mol K) | T, °K | n** | Final Volume, L | mg | ug/m³ | ppbV |
|---|---|---|---|---|---|---|---|---|---|---|
| 1-methylnaphthalene | 7.11E-05 | 142.20 | 7.1 | 0.082057 | 298.1 | 2.44E-08 | 51 | 0.00347 | 115.7 | 10 |
| 1-methylnaphthalene | 7.11E-05 | 142.00 | 42 | 0.082057 | 298.1 | 1.22E-07 | 30 | 0.0173255 | 577.5 | 100 |
| | | | | | | | | | | |
| 2-methylnaphthalene | 8.96E-05 | 142.00 | 5.8 | 0.082057 | 298.1 | 2.491E-08 | 51 | 0.0035373 | 117.9 | 10.2 |
| 2-methylnaphthalene | 8.96E-05 | 142.00 | 34 | 0.082057 | 298.1 | 1.246E-07 | 30 | 0.0176867 | 589.6 | 101.6 |
| | | | | | | | | | | |
| benzothiophene | 7.70E-05 | 134.20 | 6.5 | 0.082057 | 291.5 | 2.446E-08 | 51 | 0.0032824 | 109.4 | 10.0 |
| benzothiophene | 7.70E-05 | 134.20 | 38 | 0.082057 | 291.5 | 1.223E-07 | 30 | 0.016412 | 547.1 | 100 |

Approximately 5.0 g of solid material was allowed to stand in a 250 mL jar w/ septa cap for 30 min prior to removal of vapor phase aliquot. The aliquot was then spiked directly into secondary standard.

All vapor pressure values from Lange's Handbook of Chemistry & Physics

## Table 4

## Calibration Standard Levels

| Calibration Level | Amount (ppbV) | Volume / Secondary Standard |
|---|---|---|
| 1 | 0.20 | 50 mL of 1.0 ppbV sec. standard |
| 2 | 0.50 | 125 mL of 1.0 ppbV sec. standard |
| 3 | 1.0 | 250 mL of 1.0 ppbV sec. standard |
| | | |
| 4 | 5.0 | 125 mL of 10 ppbV sec. standard |
| 5 | 10 | 250 mL of 10 ppbV sec. standard |
| 6 | 20 | 50 mL of 100 ppbV sec. standard |
| 7 | 50 | 125 mL of 100 ppbV sec. standard |
| 8 | 100 | 250 mL of 100 ppbV sec. standard |

## Table 5

## BFB Key Ions and Abundance Criteria

| Mass | Ion Abundance Criteria |
|---|---|
| 50 | 8.0-40.0 percent of the base peak |
| 75 | 30.0-66.0 percent of the base peak |
| 95 | Base peak, 100 percent relative abundance |
| 96 | 5.0-9.0 percent of the base peak |
| 173 | Less than 2.0% of mass 174 |
| 174 | 50.0 to 120.0% of mass 95 |
| 175 | 4.0-9.0 percent of mass 174 |
| 176 | Greater than 93.0 percent but less than 101.0 percent of mass 174 |
| 177 | 5.0-9.0 percent of mass 176 |

## Table 6

## ENTECH 7016D/ 7200 or 7200A Operating Parameters

| Module 1 (Cold Trap) | |
|---|---|
| **Parameter** | **Setting** |
| Trapping Temperature | -40° C |
| Internal standard / surrogate volume | 100 mL |
| Internal standard / surrogate flow rate | 100 mL / min |
| Nominal Sample volume (may vary depending on sample concentrations) | 250 mL |
| Sample flow rate, mL / min | 100 mL / min |
| Preheat Temperature | 10° C |
| Desorb Temperature | 10° C |
| Bake Temperature | 220° C |
| Bake Time | 10 min |
| Module 1 to Module 2 transfer volume / rate | 20 cc @ 5.0 cc/min |
| **Module 2 (Tenax trap)** | |
| **Parameter** | **Setting** |
| Trapping Temperature | -65° C to -75° C |
| Desorb Temperature | 220° C |
| Bake Temperature | 220° C |
| Module 2 to Module 3 desorb time | 3.5 min |
| **Module 3 (Cryofocusser)** | |
| **Parameter** | **Setting** |
| Cryofocusing Temperature | -130° C to -190° C |
| Desorb Temperature | Approx. 90° C |
| Module 3 to GC desorb time | 3.0-3.5 min |
| Bake temperature / event | Approx. 90° C / event 3 |
| Delay time | 13-17 min |
| | |

**Table 7**
**Quantitation and Secondary Ions for TO-15 Analytes and Internal Standards**

| Compound | Quant. Ion | Sec. Ion(s) | Compound | Quant. Ion | Sec. Ion(s) | Compound | Quant. Ion | Sec. Ion(s) |
|---|---|---|---|---|---|---|---|---|
| bromochloromethane | 49 | 130 | 1,4-difluorobenzene | 114 | 63 | bromoform | 173 | 171, 175 |
| chlorodifluoromethane | 51 | 67 | n-hexane | 57 | 43, 86 | styrene | 104 | 103, 78 |
| propylene | 41 | 39, 42 | diisopropyl ether | 87 | 45, 59 | 1,1,2,2-tetrachloroethane | 83 | 85 |
| propane | 29 | 43,39 | ethyl acetate | 61 | 43, 70 | o-xylene | 91 | 106 |
| dichlorodifluoromethane | 85 | 87 | 2,2-dichloropropane | 77 | 41, 97 | 1,2,3-trichloropropane | 75 | 39, 110 |
| chloromethane | 50 | 52 | tetrahydrofuran | 42 | 71, 72 | nonane | 43 | 57, 128 |
| Freon-114 | 85 | 87, 135 | tert-butyl ethyl ether | 59 | 87, 57 | bromofluorobenzene | 95 | 75, 174 |
| methanol | 31 | 32,29 | 1,2-dichloroethane-D4 | 65 | 67, 102 | isopropylbenzene | 105 | 120 |
| vinyl chloride | 62 | 64, | 1,1,1-trichloroethane | 97 | 61, 119 | bromobenzene | 77 | 156 |
| 1,3-butadiene | 54 | 39 | 1,1-dichloropropene | 75 | 39,110 | 2-chlorotoluene | 126 | 91 |
| butane | 43 | 41,58 | benzene | 78 | 52 | n-propylbenzene | 120 | 91 |
| bromomethane | 94 | 96 | carbon tetrachloride | 117 | 119, 82 | 4-chlorotoluene | 91 | 126 |
| chloroethane | 64 | 66 | cyclohexane | 56 | 84, 41 | 4-ethyl toluene | 105 | 91, 120 |
| dichlorofluoromethane | 67 | 69, 47 | tert-amyl methyl ether | 73 | 43, 87 | 1,3,5-trimethylbenzene | 105 | 91, 120 |
| ethanol | 31 | 45 | dibromomethane | 93 | 95, 174 | tert-butlybenzene | 119 | 134 |
| acetonitrile | 41 | 40 | 1,2-dichloropropane | 63 | 39, 62 | 1,2,4-trimethylbenzene | 105 | 91, 120 |
| vinyl bromide | 106 | 108 | bromodichloromethane | 83 | 85, 129 | decane | 57 | 43, 142 |
| acrolein | 56 | 55,29 | trichloroethene | 130 | 132, 97 | benzyl chloride | 91 | 126 |
| acetone | 43 | 58 | 1,4-dioxane | 88 | 58 | 1,3-dichlorobenzene | 146 | 75, 111 |
| trichlorofluoromethane | 101 | 103 | 2,2,4-trimethylpentane | 57 | 41, 99 | 1,4-dichlorobenzene | 146 | 75, 111 |
| isopropyl alcohol | 45 | 59 | n-heptane | 43 | 57, 100 | sec-butylbenzene | 105 | 134 |
| acrylonitrile | 53 | 52,51 | cis-1,3-dichloropropene | 75 | 39, 77 | p-isopropyltoluene | 119 | 134 |
| pentane | 43 | 57,72 | 4-methyl-2-pentanone | 43 | 58, 100 | 1,2-dichlorobenzene | 146 | 75, 111 |
| ethyl ether | 31 | 59,45 | methyl methacrylate | 41 | 69, 100 | n-butylbenzene | 91 | 134 |
| 1,1-dichloroethene | 61 | 96, 63 | trans-1,3-dichloropropene | 75 | 39, 77 | 1,2-dibromo-3-chloropropane | 75 | 39, 157 |
| Tertiary butyl Alcohol | 59 | 41, 43 | 1,1,2-trichloroethane | 97 | 61, 83 | undecane | 57 | 43, 71, 156 |
| methylene chloride | 49 | 84 | Thiophene | 84 | 45, 58 | dodecane | 57 | 43 |
| 3-chloropropene | 41 | 39, 76 | chlorobenzene-D5 | 54 | 82, 117 | 1,2,4-trichlorobenzene | 180 | 109, 145 |
| carbon disulfide | 76 | 44 | toluene | 91 | 92 | naphthalene | 128 | 102 |
| Freon 113 | 101 | 85, 151 | toluene-D8 | 98 | 100 | 1,2,3-trichlorobenzene | 180 | 109, 145 |
| trans-1,2-dichloroethene | 61 | 96, 98 | 1,3-dichloropropane | 76 | 41,49 | hexachlorobutadiene | 225 | 118, 260 |
| 1,1-dichloroethane | 63 | 65 | 2-hexanone | 43 | 58, 100 | 2-methylthiophene | 97 | 45, 98 |
| MTBE | 73 | 57, 43 | dibromochloromethane | 129 | 127, 131 | 3-methylthiophene | 97 | 45, 98 |
| vinyl acetate | 43 | 86 | 1,2-dibromoethane | 107 | 109 | 2-ethylthiophene | 97 | 45, 112 |
| 2-butanone | 43 | 72 | butyl acetate | 73 | 43, 56 | 1,2,3-trimethylbenzene | 105 | 120 |
| cis-1,2-dichloroethene | 61 | 96, 98 | octane | 85 | 43, 57, 114 | indan | 117 | 91, 118 |
| chloroform | 83 | 85, 47 | tetrachloroethene | 166 | 94, 131 | indene | 115 | 89, 116 |
| 1,2-dichloroethane | 62 | 49, 63, 64 | 1,1,1,2-tetrachloroethane | 131 | 95, 133 | 1,2,4,5-tetramethylbenzene | 119 | 91, 134 |
| acetaldehyde | 29 | 43, 44 | chlorobenzene | 112 | 77, 114 | benzothiophene | 134 | 63, 89 |
| | | | ethylbenzene | 91 | 106 | 2-methylnaphthalene | 142 | 115, 141 |
| | | | m+p-xylene | 91 | 106 | 1-methylnaphthalene | 142 | 115, 141 |
| | | | | | | | | |

# Table 8

## Internal Standards and the Associated Target Analytes

| bromochloromethane | | 1,4-difluorobenzene | chlorobenzene-D5 | |
|---|---|---|---|---|
| chlorodifluoromethane | trans-1,2-dichloroethene | hexane | toluene | decane |
| propylene | 1,1-dichloroethane | diisopropyl ether | toluene-D8 | benzyl chloride |
| propane | MTBE | tert-butyl ethyl ether | 1,3-dichloropropane | 1,3-dichlorobenzene |
| dichlorodifluoromethane | vinyl acetate | 1,2-dichloroethane-D4 | 2-hexanone | 1,4-dichlorobenzene |
| chloromethane | 2-butanone | 1,1,1-trichloroethane | dibromochloromethane | sec-butylbenzene |
| Freon-114 | cis-1,2-dichloroethene | 1,1-dichloropropene | 1,2-dibromoethane | p-isopropyltoluene |
| methanol | chloroform | benzene | butyl acetate | 1,2-dichlorobenzene |
| vinyl chloride | 1,2-dichloroethane | carbon tetrachloride | Octane | n-butylbenzene |
| 1,3-butadiene | trans-1,2-dichloroethene | cyclohexane | tetrachloroethene | 1,2-dibromo-3-chloropropane |
| butane | 1,1-dichloroethane | tert-amyl methyl ether | 1,1,1,2-tetrachloroethane | undecane |
| bromomethane | acetaldehyde | dibromomethane | Chlorobenzene | dodecane |
| chloroethane | ethyl acetate | 1,2-dichloropropane | Ethylbenzene | 1,2,4-trichlorobenzene |
| dichlorofluoromethane | 2,2-dichloropropane | bromodichloromethane | m+p-xylene | naphthalene |
| ethanol | tetrahydrofuran | trichloroethene | Bromoform | 1,2,3-trichlorobenzene |
| acetonitrile | | 1,4-dioxane | Styrene | hexachlorobutadiene |
| vinyl bromide | | 2,2,4-trimethylpentane | 1,1,2,2-tetrachloroethane | 2-methylthiophene |
| acrolein | | heptane | o-xylene | 3-methylthiophene |
| acetone | | cis-1,3-dichloropropene | 1,2,3-trichloropropane | 2-ethylthiophene |
| trichlorofluoromethane | | 4-methyl-2-pentanone | Nonane | 1,2,3-trimethylbenzene |
| isopropyl alcohol | | methyl methacrylate | bromofluorobenzene | indan |
| acrylonitrile | | trans-1,3-dichloropropene | isopropylbenzene | indene |
| pentane | | 1,1,2-trichloroethane | bromobenzene | 1,2,4,5-tetramethylbenzene |
| ethyl ether | | thiophene | 2-chlorotoluene | benzothiophene |
| 1,1-dichloroethene | | | n-propylbenzene | 2-methylnaphthalene |
| Tertiary butyl Alcohol | | | 4-chlorotoluene | 1-methylnaphthalene |
| methylene chloride | | | 4-ethyl toluene | |
| 3-chloropropene | | | 1,3,5-trimethylbenzene | |
| carbon disulfide | | | tert-butlybenzene | |
| Freon 113 | | | 1,2,4-trimethylbenzene | |

## Table 9A

## TO-15 Target Analytes and Reporting Limits
## Standard List

| COMPOUND | CAS # | Standard Reporting Limit, ppbV | Standard Reporting Limit, ug/m$^3$ |
|---|---|---|---|
| 1,1,1-trichloroethane | 71-55-6 | 0.2 | 1.09 |
| 1,1,2,2-tetrachloroethane | 79-34-5 | 0.2 | 1.37 |
| 1,1,2-trichloroethane | 79-00-5 | 0.2 | 1.09 |
| 1,1-dichloroethane | 75-34-3 | 0.2 | 0.81 |
| 1,1-dichloroethene | 75-35-5 | 0.2 | 0.79 |
| 1,2,4-trichlorobenzene | 120-82-1 | 0.2 | 1.48 |
| 1,2,4-trimethylbenzene | 95-63-6 | 0.2 | 0.98 |
| 1,2-dibromoethane | 106-93-4 | 0.2 | 1.54 |
| 1,2-dichlorobenzene | 95-50-1 | 0.2 | 1.2 |
| 1,2-dichloroethane | 107-06-2 | 0.2 | 0.81 |
| 1,2-dichloropropane | 78-87-5 | 0.2 | 0.92 |
| 1,3,5-trimethylbenzene | 108-67-8 | 0.2 | 0.98 |
| 1,3-butadiene | 106-99-0 | 0.2 | 0.44 |
| 1,3-dichlorobenzene | 541-73-1 | 0.2 | 1.2 |
| 1,4-dichlorobenzene | 106-46-7 | 0.2 | 1.2 |
| 1,4-dioxane | 123-91-1 | 0.2 | 0.72 |
| 2,2,4-trimethylpentane | 540-84-1 | 0.2 | 0.93 |
| 2-butanone | 78-93-3 | 0.5 | 1.48 |
| 2-hexanone | 591-78-6 | 0.2 | 0.82 |
| 3-chloropropene | 107-05-1 | 0.2 | 0.63 |
| 4-Ethyltoluene | 622-96-8 | 0.2 | 0.98 |
| Acetone | 67-64-1 | 1.0 | 2.38 |
| benzene | 71-43-2 | 0.2 | 0.64 |
| Benzyl Chloride | 100-44-7 | 0.2 | 1.03 |
| bromodichloromethane | 75-27-4 | 0.2 | 1.34 |
| bromoform | 75-25-2 | 0.2 | 2.07 |
| bromomethane | 74-83-9 | 0.2 | 0.78 |
| carbon disulfide | 75-15-0 | 0.2 | 0.62 |
| carbon tetrachloride | 56-23-5 | 0.2 | 1.26 |
| chlorobenzene | 108-90-7 | 0.2 | 0.92 |
| chloroethane | 75-00-3 | 0.2 | 0.53 |
| chloroform | 67-66-3 | 0.2 | 0.98 |

| COMPOUND | CAS # | Standard Reporting Limit, ppbV | Standard Reporting Limit, ug/m$^3$ |
|---|---|---|---|
| chloromethane | 74-87-3 | 0.2 | 0.41 |
| cis-1,2-dichloroethene | 156-59-2 | 0.2 | 0.79 |
| cis-1,3-dichloropropene | 10061-01-5 | 0.2 | 0.91 |
| cyclohexane | 110-82-7 | 0.2 | 0.69 |
| dibromochloromethane | 124-48-1 | 0.2 | 1.7 |
| dichlorodifluoromethane | 75-71-8 | 0.2 | 0.99 |
| ethanol | 64-17-5 | 2.5 | 4.71 |
| ethyl acetate | 141-78-6 | 0.5 | 1.8 |
| ethylbenzene | 100-41-4 | 0.2 | 0.87 |
| Freon-113 | 76-13-1 | 0.2 | 1.53 |
| Freon-114 | 76-14-2 | 0.2 | 1.4 |
| hexachlorobutadiene | 87-68-3 | 0.2 | 2.13 |
| hexane | 110-54-3 | 0.2 | 0.7 |
| isopropyl alcohol | 67-63-0 | 0.5 | 1.23 |
| methylene chloride | 75-09-2 | 0.5 | 1.74 |
| MIBK | 108-10-1 | 0.5 | 2.1 |
| MTBE | 1634-04-4 | 0.2 | 0.72 |
| m+p-xylene | 108-38-3<br>106-42-3 | 0.4 | 1.74 |
| n-heptane | 142-82-5 | 0.2 | 0.82 |
| o-xylene | 95-47-6 | 0.2 | 0.87 |
| propylene | 115-7-1 | 0.5 | 0.85 |
| styrene | 100-42-5 | 0.2 | 0.85 |
| tetrachloroethene | 127-18-4 | 0.2 | 1.36 |
| tetrahydrofuran | 109-99-9 | 0.5 | 1.48 |
| toluene | 108-88-3 | 0.2 | 0.75 |
| trans-1,2-dichloroethene | 156-60-5 | 0.2 | 0.79 |
| trans-1,3-dichloropropene | 10061-02-6 | 0.2 | 0.91 |
| trichloroethene | 79-01-6 | 0.2 | 1.07 |
| trichlorofluoromethane | 75-69-4 | 0.2 | 1.12 |
| vinyl acetate | 108-05-4 | 0.5 | 1.75 |
| vinyl bromide | 593-60-2 | 0.2 | 0.87 |
| vinyl chloride | 75-01-4 | 0.2 | 0.51 |

## Table 9B

## TO-15 Target Analytes and Reporting Limits
## Additional Analytes

| COMPOUND | CAS # | Standard Reporting Limit, ppbV | Standard Reporting Limit, ug/m$^3$ |
|---|---|---|---|
| **AP-42 Analytes** | | | |
| acrolein | 107-02-8 | 0.50 | 1.15 |
| acrylonitrile | 107-13-1 | 0.50 | 1.08 |
| butane | 106-97-8 | 0.20 | 0.48 |
| Chlorodifluoromethane | 75-45-6 | 0.20 | 0.71 |
| Dichlorofluoromethane | 75-71-8 | 0.20 | 0.84 |
| n-Pentane | 109-66-0 | 0.20 | 0.59 |
| Propane | 74-98-6 | 0.50 | 0.90 |
| **MADEP MCP 8260 Analytes** | | | |
| 1,1,1,2-tetrachloroethene | 630-20-6 | 0.20 | 1.37 |
| 1,1-dichloropropene | 563-58-6 | 0.20 | 0.91 |
| 1,2,3-trichlorobenzene | 87-61-6 | 0.20 | 1.48 |
| 1,2,3-Trichloropropane | 96-18-4 | 0.20 | 1.20 |
| 1,3-dichloropropane | 142-28-9 | 0.20 | 0.92 |
| 2,2-dichloropropane | 594-20-7 | 0.20 | 0.92 |
| 2-chlorotoluene | 95-49-8 | 0.20 | 1.03 |
| 4-chlorotoluene | 106-43-4 | 0.20 | 1.03 |
| bromobenzene | 108-86-1 | 0.20 | 1.28 |
| 1,2-dibromo-3-chloropropane | 96-12-8 | 0.20 | 1.93 |
| dibromomethane | 74-95-3 | 0.20 | 1.42 |
| diisopropyl ether | 108-20-3 | 0.20 | 0.84 |
| isopropylbenzene | 98-82-8 | 0.20 | 0.98 |
| isopropyltoluene | 99-87-6 | 0.20 | 1.10 |
| naphthalene | 91-20-3 | 0.20 | 1.05 |
| n-butylbenzene | 104-51-8 | 0.20 | 1.10 |
| n-propylbenzene | 103-65-1 | 0.20 | 0.98 |
| sec-butylbenzene | 135-98-8 | 0.20 | 1.10 |
| tert-amyl methyl ether | 994-05-8 | 0.20 | 0.84 |
| tert-Butyl ethyl ether | 637-92-3 | 0.20 | 0.84 |
| tert-butylbenzene | 98-06-6 | 0.20 | 1.10 |

| COMPOUND | CAS # | Standard Reporting Limit, ppbV | Standard Reporting Limit, ug/m$^3$ |
|---|---|---|---|
| **NYDEC Petroleum Indicator Compounds** | | | |
| nonane | 111-84-2 | 0.20 | 1.05 |
| octane | 111-65-9 | 0.20 | 0.93 |
| undecane | 1120-21-4 | 0.20 | 1.28 |
| decane | 124-18-5 | 0.20 | 1.16 |
| dodecane | 112-40-3 | 0.20 | 1.39 |
| indene | 95-13-6 | 0.20 | 0.95 |
| Indan | 496-11-7 | 0.20 | 0.97 |
| thiophene | 110-02-1 | 0.20 | 0.69 |
| 2-methylthiophene | 554-13-3 | 0.20 | 0.80 |
| 3-methylthiophene | 616-44-4 | 0.20 | 0.80 |
| 2-ethyl thiophene | 872-55-9 | 0.20 | 0.92 |
| benzothiophene | 934-80-5 | 0.50 | 2.74 |
| 1,2,3-trimethylbenzene | 526-73-8 | 0.20 | 0.98 |
| 1,2,4,5-tetramethylbenzene | 95-93-2 | 0.20 | 1.10 |
| 2-methylnaphthalene | 91-57-6 | 1.0 | 5.8 |
| 1-methylnaphthalene | 90-12-0 | 1.0 | 5.8 |
| **Project Specific Analytes** | | | |
| acetaldehyde | 75-07-0 | 2.5 | 4.51 |
| Acetonitrile | 75-05-8 | 0.20 | 0.34 |
| butyl acetate | 123-86-4 | 0.50 | 2.38 |
| ethyl ether | 60-29-7 | 0.20 | 0.61 |
| methanol | 67-56-1 | 5.0 | 6.55 |
| tert-butyl alcohol | 75-65-0 | 0.50 | 1.51 |
| Methyl methacrylate | 80-62-6 | 0.50 | 2.05 |
| | | | |
| | | | |
| **All reporting limits are subject to change.** | | | |
| | | | |
| | | | |

# Appendix A

## Cold Trap Dehydration technique (CTD) for Analysis of Sulfides and Mercaptans by EPA TO-15

Target analytes:

| Compound | CAS# | Reporting Limit, ppbV |
|---|---|---|
| Hydrogen sulfide | 7783-06-4 | 2.0 |
| Carbonyl sulfide | 463-58-1 | 2.0 |
| Methyl mercaptan | 74-93-1 | 2.0 |
| Ethyl mercaptan | 75-08-1 | 0.5 |
| Dimethyl sulfide | 75-18-3 | 0.5 |
| carbon disulfide | 75-15-0 | 0.5 |
| Isopropyl Mercaptan | 75-33-2 | 0.5 |
| tert-Butyl Mercaptan | 75-66-1 | 0.5 |
| n-Propyl Mercaptan | 107-03-9 | 0.5 |
| Ethyl Methyl Sulfide | 624-89-5 | 0.5 |
| | | |

| Compound | CAS# | Reporting Limit, ppbV |
|---|---|---|
| Thiophene | 110-02-1 | 0.5 |
| Isobutyl Mercaptan | 513-44-0 | 0.5 |
| diethyl sulfide | 352-93-2 | 0.5 |
| Butyl Mercaptan | 109-75-5 | 2.0 |
| dimethyl disulfide | 624-92-0 | 0.5 |
| 3-Methylthiophene | 616-44-4 | 0.5 |
| Tetrahydrothiophene | 110-01-0 | 0.5 |
| 2-Ethylthiophene | 872-55-9 | 0.5 |
| 2,5-Dimethylthiophene | 638-02-8 | 0.5 |
| Diethyl Disulfide | 110-81-6 | 0.5 |
| **All reporting limits are subject to change.** | | |

The cold trap dehydration method requires a blank trap (i.e. no trapping material) be installed in module 1 of the Entech 7200 or 7200A concentrator. This trap is cooled and a 250 mL aliquot of sample is allowed to pass through this trap and then directly onto the Tenax trap in module 2, which is also cooled (see Table A-5 for setpoints). The sample is then transferred to module 3 (cryofocusser) via ballistic heating. All requirements stated in this SOP must be applied to any TO15-Sulfide/Mercaptan analysis conducted in the laboratory.

SOP modifications:

Standard preparation and calibration procedures for these analytes are listed in Table A-1 through A-5. Quantitation parameters are listed in Table A-8. Table A-7 lists modified GC conditions.

Section 9.2.5 and 9.5.3.1: Use the Entech method alpha_H2S&SULF.CTD in place of the alpha_TO15.MPT method.

A second source laboratory check standard (LCS) is not readily available for these analytes. An LCS standard is prepared at a different concentration than the initial calibration standards using the same stock standard.

# Table A-1

## Sulfide/Mercaptan Primary Standard Mix #1

| COMPOUND | CAS# | Molecular Weight | Concentration, ppmV |
|---|---|---|---|
| hydrogen sulfide | 7783-06-4 | 34.08 | 5.0 |
| Carbonyl sulfide | 463-58-1 | 60.08 | 5.0 |
| Methyl mercaptan | 74-93-1 | 48.11 | 5.0 |
| ethyl mercaptan | 75-08-1 | 62.14 | 1.0 |
| dimethyl sulfide | 75-18-3 | 62.14 | 1.0 |
| carbon disulfide | 75-15-0 | 76.1 | 1.0 |
| Isopropyl Mercaptan | 75-33-2 | 76.2 | 1.0 |
| tert-Butyl Mercaptan | 75-66-1 | 90.19 | 1.0 |
| n-Propyl Mercaptan | 107-03-9 | 76.16 | 1.0 |
| Ethyl Methyl Sulfide | 624-89-5 | 76.16 | 1.0 |
| Thiophene | 110-02-1 | 84.1 | 1.0 |
| Isobutyl Mercaptan | 513-44-0 | 90.19 | 1.0 |
| diethyl sulfide | 352-93-2 | 90.2 | 1.0 |
| Butyl Mercaptan | 109-79-5 | 90.19 | 4.0 |
| 3-Methylthiophene | 616-44-4 | 98.17 | 1.0 |
| Tetrahydrothiophene | 110-01-0 | 88.17 | 1.0 |
| 2-Ethylthiophene | 872-55-9 | 112.19 | 1.0 |
| 2,5-Dimethylthiophene | 638-02-8 | 112.19 | 1.0 |

## Table A-2

## Sulfide/Mercaptan Primary Standard Mix #2

| COMPOUND | CAS# | Molecular Weight | Concentration, ppmV |
|---|---|---|---|
| dimethyl disulfide | 624-92-0 | 94.2 | 1.0 |

## Table A-3

## Sulfide/Mercaptan Primary Standard Mix #3

| COMPOUND | CAS# | Molecular Weight | Concentration, ppmV |
|---|---|---|---|
| Diethyl Disulfide | 110-81-6 | 122.25 | 1.0 |

## Table A-4

## Summary of Secondary Standards Preparation for Sulfides and Mercaptan Analysis

| Primary Standard | Primary Standard Conc. ppmV | Volume of Primary Standard Injected into Tedlar® Bag | Primary Standard Transfer Data | Final Volume Tedlar® Bag (L) | Final Concentration ppbV |
|---|---|---|---|---|---|
| **Secondary standards prepared using gas-tight syringes** | | | | | |
| Tedlar® bag primary standard-Low | 1- 5ppmV | 20 mL | Syringe Injection | 4.0 | 5.0 / 20.0 / 20 |
| Tedlar® bag primary standard-High | 1- 5ppmV | 400 mL | Syringe Injection | 4.0 | 100 / 400 / 500 |
| Tedlar® bag primary standard-LCS | 1- 5ppmV | 40 mL | Syringe Injection | 4.0 | 10 / 40 / 50 |

## Table A-5

## Calibration Standard Levels

| Calibration Level | Amount (ppbV) | Volume / Secondary Standard |
|---|---|---|
| 1 | 0.50 | 25 mL of 5.0 ppbV sec. standard |
| 2 | 1.0 | 50 mL of 5.0 ppbV sec. standard |
| 3 | 5.0 | 250 mL of 5.0 ppbV sec. standard |
| 4 | 10 | 25 mL of 100 ppbV sec. standard |
| 5 | 20 | 50 mL of 100 ppbV sec. standard |
| 6 | 50 | 125 mL of 100 ppbV sec. standard |
| 7 | 80 | 200 mL of 100 ppbV sec. standard |
| 8 | 100 | 250 mL of 100 ppbV sec. standard |
| | | |

## Table A-6

## ENTECH & 7016D/7200 or 7200A Operating Parameters for CTD Method

| Module 1 (Blank Trap) | |
|---|---|
| **Parameter** | **Setting** |
| Trapping Temperature | -20° C |
| Internal standard volume | 100 mL |
| Internal standard flow rate | 60 mL / min |
| Sample volume (may vary depending on sample concentrations) | 250 mL |
| Sample flow rate | 100 mL / min |
| Preheat Temperature | 10° C |
| Desorb Temperature | 10° C |
| Bake Temperature | 220° C |
| Bake Time | 10 min. |
| **Module 2 (Tenax trap)** | |
| **Parameter** | **Setting** |
| Trapping Temperature | -70° C |
| Desorb Temperature | 180° C |
| Bake Temperature | 220° C |
| Module 2 to Module 3 desorb time | 3.5 min |
| **Module 3 (Cryofocusser)** | |
| **Parameter** | **Setting** |
| Cryofocusing Temperature | -130° C |
| Desorb Temperature | Approx. 70° C |
| Module 3 to GC desorb time | 2 min |
| Bake temperature / event | Approx. 90° C / event 3 |
| Delay time | 10 min |

## Table A-7

## Internal Standard (IS) Assignments and Quantitation Ions for Sulfides & Mercaptans

| Compound | Quant Ion | Sec. Ion(s) |
|---|---|---|
| Bromochloromethane (IS) | 49 | 130 |
| Hydrogen sulfide | 34 | 33, 36 |
| Carbonyl sulfide | 60 | 62, 32 |
| Methyl mercaptan | 47 | 48, 45 |
| ethyl mercaptan | 62 | 47, 29 |
| dimethyl sulfide | 62 | 45, 47 |
| carbon disulfide | 76 | 44, 78 |
| Isopropyl Mercaptan | 43 | 41, 76 |
| tert-Butyl Mercaptan | 41 | 57, 90 |
| n-Propyl Mercaptan | 76 | 47, 43 |
| Ethyl Methyl Sulfide | 61 | 76, 48 |

| Compound | Quant Ion | Sec. Ion(s) |
|---|---|---|
| 1,4-difluorobenzene (IS) | 114 | 63 |
| Thiophene | 84 | 58, 45 |
| Isobutyl Mercaptan | 41 | 56, 90 |
| diethyl sulfide | 75 | 90, 47 |
| Butyl Mercaptan | 56 | 41, 90 |
| dimethyl disulfide | 94 | 79, 45 |
| | | |
| | | |
| | | |
| | | |
| | | |

| Compound | Quant Ion | Sec. Ion(s) |
|---|---|---|
| Chlorobenzene-D5 (IS) | 54 | 82, 117 |
| 3-Methylthiophene | 97 | 98, 45 |
| Tetrahydrothiophene | 60 | 88, 45 |
| 2-Ethylthiophene | 97 | 112, 45 |
| 2,5-Dimethylthiophene | 111 | 112,97 |
| Diethyl Disulfide | 122 | 66, 94 |
| | | |
| | | |
| | | |
| | | |
| | | |

# Appendix B

## Data Acquistion Parameters and Analysis Modifications for Conducting SIM Analysis

SIM analysis is conducted when full scan sensitivity does not meet the data quality objectives (DQO) of the project and/or regulatory criteria. The acquisition method used to acquire full scan data simultaneously acquires SIM data using the SIM ions and windows specified in Table B-1. The following modifications to the full scan SOP must be done to generate data using the SIM signal:

- SIM level calibration standards must be analyzed w/ the full scan curve (0.02,0.05, and 0.1 ppbV)
- A calibration curve is generated using the SIM signal utilizing the lower level calibration standards and must meet the same criteria as the full scan calibration criteria.
- The continuing calibration /LCS should be analyzed at a lower concentration (5.0 ppbV). If a separate continuing calibration run is needed, the continuing calibration level of 10 ppbV may be utilized. Laboratory Method Blanks must be evaluated for the SIM reporting limit as listed in Table B-2.

The SIM signal only acquires data for a limited target analyte list. These target analytes and reporting limits are listed in Table B-2. Additional ions have been added to allow for more analytes to be added, if requested by client. All requirements stated in this SOP must be applied to any TO15-SIM analysis conducted in the laboratory.

## Table B-1

## Calibration Standard Levels for SIM Analysis

| Calibration Level | Amount (ppbV) | Volume / Secondary Standard |
|---|---|---|
| 1 | 0.02 | 50 mL of 0.10 ppbV sec. standard |
| 2 | 0.05 | 125 mL of 0.1 ppbV sec. standard |
| 3 | 0.10 | 250 mL of 0.1 ppbV sec. standard |
| 4 | 0.20 | 50 mL of 1.0 ppbV sec. standard |
| 5 | 0.50 | 125 mL of 1.0 ppbV sec. standard |
| 6 | 1.0 | 250 mL of 1.0 ppbV sec. standard |
| 7 | 5.0 | 125 mL of 10 ppbV sec. standard |
| 8 | 10 | 250 mL of 10 ppbV sec. standard |
| 9 | 20 | 50 mL of 100 ppbV sec. standard |
| 10 | 50 | 125 mL of 100 ppbV sec. standard |

## Table B-2

## Seletive Ion Monitoring (SIM) Groupings

| Group # | 1 | 2 | 3 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Group Start Time, min | 0 | 5.9 | 8.75 | 10.1 | 10.9 | 11.6 | 13.8 | 14.6 | 16.6 | 18.8 | 20.5 |
| Dwell time, sec | 25 | 10 | 25 | 25 | 25 | 15 | 25 | 15 | 3 | 5 | 25 |
| Cycles/sec (calc) | 2 | 2.5 | 3.3 | 2.1 | 2.3 | 1.8 | 5.7 | 2.2 | 7 | 7 | 2.3 |
| Analyte Range, Compund # | C2-C7 | C8-C20 | C21-C26 | C27-C29, BCM (IS) | C30-C34 | C35-C44, 1,4-DFB (IS) | C45-C46 | C47-C55 | C56-C67, CHB-D5 (IS) | C68-C76 | C77-C80 |
| Ions | 29 | 29 63 | 43 | 41 | 42 | 39 85 | 39 | 39 92 | 39 106 | 39 | 43 |
| | 31 | 31 64 | 57 | 43 | 49 | 41 87 | 43 | 41 94 | 43 110 | 43 | 57 |
| | 32 | 39 66 | 61 | 45 | 51 | 43 88 | 58 | 43 97 | 45 112 | 57 | 63 |
| | 39 | 40 67 | 63 | 47 | 57 | 45 93 | 75 | 45 98 | 54 114 | 71 | 89 |
| | 41 | 41 69 | 65 | 49 | 59 | 52 95 | 77 | 49 100 | 57 117 | 75 | 102 |
| | 42 | 43 72 | 72 | 57 | 62 | 56 97 | 100 | 56 107 | 75 120 | 89 | 109 |
| | 43 | 44 76 | 73 | 59 | 63 | 57 99 | | 57 109 | 77 126 | 91 | 115 |
| | 44 | 45 84 | 86 | 61 | 64 | 58 100 | | 58 114 | 78 128 | 105 | 118 |
| | 50 | 47 85 | 96 | 70 | 65 | 62 110 | | 61 127 | 82 131 | 111 | 128 |
| | 51 | 49 94 | 98 | 77 | 67 | 63 114 | | 73 129 | 83 133 | 115 | 134 |
| | 52 | 51 96 | 117 | 83 | 71 | 69 117 | | 75 131 | 85 156 | 116 | 141 |
| | 54 | 52 101 | 198 | 85 | 72 | 73 119 | | 76 166 | 91 171 | 117 | 142 |
| | 58 | 53 103 | | 86 | 87 | 75 129 | | 77 | 95 173 | 118 | 145 |
| | 62 | 55 106 | | 87 | 97 | 78 130 | | 83 | 97 174 | 119 | 180 |
| | 64 | 56 108 | | 96 | 102 | 82 132 | | 85 | 103 175 | 120 | 225 |
| | 67 | 57 151 | | 97 | 119 | 83 174 | | 91 | 104 | 126 | 260 |
| | 85 | 58 | | 98 | | 84 | | | 105 | 134 | |
| | 87 | 59 | | 130 | | | | | | 142 | |
| | 135 | 61 | | | | | | | | 146 | |
| | | | | | | | | | | 156 | |
| | | | | | | | | | | 157 | |

## Table B-3

## TO15-SIM Target Analytes and Reporting Limits

| COMPOUND | CAS # | SIM Reporting Limit, ppbV | SIM Reporting Limit, ug/m$^3$ |
|---|---|---|---|
| 1,1,1-trichloroethane | 71-55-6 | 0.02 | 0.109 |
| 1,1,2,2-tetrachloroethane | 79-34-5 | 0.02 | 0.137 |
| 1,1,2-trichloroethane | 79-00-5 | 0.02 | 0.109 |
| 1,1-dichloroethane | 75-34-3 | 0.02 | 0.081 |
| 1,1-dichloroethene | 75-35-5 | 0.02 | 0.079 |
| 1,2,4-trimethylbenzene | 95-63-6 | 0.02 | 0.098 |
| 1,2-dibromoethane | 106-93-4 | 0.02 | 0.154 |
| 1,2-dichlorobenzene | 95-50-1 | 0.02 | 0.12 |
| 1,2-dichloroethane | 107-06-2 | 0.02 | 0.081 |
| 1,2-dichloropropane | 78-87-5 | 0.02 | 0.092 |
| 1,2-dibromo-3-chloropropane | 96-12-8 | 0.02 | 0.193 |
| 1,3,5-trimethylbenzene | 108-67-8 | 0.02 | 0.098 |
| 1,3-butadiene | 106-99-0 | 0.02 | 0.044 |
| 1,3-dichlorobenzene | 541-73-1 | 0.02 | 0.12 |
| 1,4-dichlorobenzene | 106-46-7 | 0.02 | 0.12 |
| 1,4-dioxane | 123-91-1 | 0.10 | 0.36 |
| 2-hexanone | 591-78-6 | 0.20 | 0.82 |
| 2,2,4-trimethylpentane | 540-84-1 | 0.20 | 0.93 |
| 3-chloropropene | 107-05-1 | 0.20 | 0.63 |
| 4-Ethyltoluene | 622-96-8 | 0.02 | 0.098 |
| benzene | 71-43-2 | 0.10 | 0.223 |
| benzyl chloride | 100-44-7 | 0.20 | 1.03 |
| bromodichloromethane | 75-27-4 | 0.02 | 0.134 |
| bromoform | 75-25-2 | 0.02 | 0.207 |
| bromomethane | 74-83-9 | 0.02 | 0.078 |
| carbon disulfide | 75-15-0 | 0.20 | 0.62 |
| hexachlorobutadiene | 87-68-3 | 0.05 | 533 |
| hexane | 110-54-3 | 0.20 | 0.7 |
| heptane | 142-82-5 | 0.20 | 0.82 |
| isopropyl alcohol | 67-63-0 | 0.50 | 1.23 |
| methylene chloride | 75-09-2 | 0.50 | 1.74 |
| MTBE | 1634-04-4 | 0.02 | 0.072 |
| m+p-xylene | 108-38-3<br>106-42-3 | 0.04 | 0.174 |
| o-xylene | 95-47-6 | 0.02 | 0.087 |
| propylene | 115-7-1 | 0.50 | 0.85 |
| styrene | 100-42-5 | 0.02 | 0.085 |
| tetrachloroethene | 127-18-4 | 0.02 | 0.136 |
| tetrahydrofuran | 109-99-9 | 0.5 | 1.48 |
| toluene | 108-88-3 | 0.05 | 0.188 |
| trans-1,2-dichloroethene | 156-60-5 | 0.02 | 0.079 |
| trans-1,3-dichloropropene | 10061-02-6 | 0.02 | 0.091 |
| trichlorofluoromethane | 75-69-4 | 0.05 | 0.280 |
| trichloroethene | 79-01-6 | 0.02 | 0.107 |
| 1,2,4-trichlorobenzene | 120-82-1 | 0.05 | 0.371 |
| vinyl acetate | 108-05-4 | 1.00 | 3.50 |
| vinyl bromide | 593-60-2 | 0.20 | 0.87 |
| vinyl chloride | 75-01-4 | 0.02 | 0.051 |
| **CT RSR Additional Analytes** | | | |
| 1,1,1,2-tetrachloroethane | 630-20-6 | 0.02 | 0.137 |
| acrylonitrile | 107-13-1 | 0.50 | 1.08 |
| isopropyltoluene | 99-87-6 | 0.20 | 1.1 |
| n-butylbenzene | 104-51-8 | 0.20 | 1.1 |

| | | | |
|---|---|---|---|
| carbon tetrachloride | 56-23-5 | 0.02 | 0.126 |
| chlorobenzene | 108-90-7 | 0.10 | 0.460 |
| chloroethane | 75-00-3 | 0.10 | 0.265 |
| chloroform | 67-66-3 | 0.02 | 0.098 |
| chloromethane | 74-87-3 | 0.20 | 0.41 |
| cis-1,2-dichloroethene | 156-59-2 | 0.02 | 0.079 |
| cis-1,3-dichloropropene | 10061-01-5 | 0.02 | 0.091 |
| cyclohexane | 110-82-7 | 0.2 | 0.69 |
| dibromochloromethane | 124-48-1 | 0.02 | 0.17 |
| dichlorodifluoromethane | 75-71-8 | 0.20 | 0.99 |
| ethanol | 64-17-5 | 5.0 | 9.42 |
| ethyl acetate | 141-78-6 | 0.50 | 1.8 |
| ethylbenzene | 100-41-4 | 0.02 | 0.087 |
| Freon-113 | 76-13-1 | 0.05 | 0.383 |
| Freon-114 | 76-14-2 | 0.05 | 0.349 |
| sec-butylbenzene | 135-98-8 | 0.20 | 1.1 |
| tert-butylbenzene | 98-06-6 | 0.20 | 1.1 |
| isopropylbenzene | 98-82-8 | 0.20 | 0.98 |
| 2-butanone (MEK) | 78-93-3 | 0.50 | 1.48 |
| Acetone | 67-64-1 | 1.00 | 2.35 |
| 4-methyl-2-pentanone (MIBK) | 108-10-1 | 0.50 | 2.05 |
| **Project & State Specific Analytes** | | | |
| 1,2,3-trichlorobenzene | 87-61-6 | 0.05 | 0.371 |
| 1,2,3-Trichloropropane | 96-18-4 | 0.02 | 0.12 |
| acrolein | 107-02-8 | 0.05 | 0.115 |
| bromobenzene | 108-86-1 | 0.20 | 1.28 |
| Dibromomethane | 74-95-3 | 0.20 | 1.42 |
| Naphthalene | 91-20-3 | 0.05 | 0.262 |
| tertiary butyl alcohol | 75-65-0 | 0.50 | 1.51 |
| **All reporting limits are subject to change.** | | | |

# Appendix C

## Modifications to Data Review and Case Narrative to Comply with MADEP MCP TO-15 Method

This addendum addresses modifications to this SOP required to be in compliance with the MADEP MCP TO-15 method, specifically "Quality Control Requirements and Performance Standards for the ***Analysis of Volatile Organic Compounds in Air Samples (TO-15) by Gas Chromatography/Mass Spectrometry (GC/MS)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)" Revision No. 0, Section IX B.

1) For duplicate analyses, the 25% RPD criteria stated in Sect. 9.6 of this SOP does not need to be applied to concentrations less than 5X the reporting limit.

2) Samples cannot be analyzed if any of the target analytes if the LCS recovery criteria stated in sect. 9.2 of this SOP is below 70% recovery. For compounds listed as difficult analytes (hexachlorobutadiene, 1,2,4-trichlorobenzene, naphthalene, acetone, and 1,4-dioxane), LCS recovery cannot be less than 50%.

3) Any analyte exceeding %RSD criteria of 30% during initial calibration that is a not listed in the MCP TO-15 method, but may still be reported to the client, must be noted in the case narrative.

# Appendix E

## Modifications to Data Review and Case Narrative to Comply with 2014 NJDEP Technical Guidance for EPA Method TO-15

This addendum addresses modifications to this SOP required to be in compliance with the NJDEP 2014 Technical Guidance, **"NJDEP Site Remediation Program, Data of Known Quality Protocol, Version 1, April 2014**". This guidance is to be used when analyzing samples via the EPA Method TO-15.

1) Per the Data of Know Quality Protocols Technical Guidance, surrogates are not required to be reported via EPA Method TO-15.

2) A target analyte list and reporting limits are specified in Table E-1, per the Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance.

3) For the analytes ethanol and isopropyl alcohol, results are allowed to be reported outside the calibration range of the instrument, per the Analytical Laboratory Data Generation, Assessment and Usability Technical Guidance. Results reported that exceed the calibration range will be designated with an "E" qualifier.

4) **Laboratory Control Sample (LCS) criteria -** Must contain all target analytes, recovery range is 70-130%. Exceptions for difficult analytes (hexachlorobutadiene, 1,2,4-trichlorobenzene, naphthalene, acetone, dichlorodifluoromethane, and 1,4-dioxane) must exhibit percent recoveries between 40-160%. In addition, the CCAL analysis cannot be reported as the LCS even if it meets LCS requirements. The LCS must be a separate analysis, analyzed after a passing CCAL standard.

5) **Duplicate analyses** - The 25% RPD criteria stated in Sect. 9.6 of this SOP does not need to be applied to concentrations less than 5X the reporting limit.

6) **Tentatively Identified Compounds (TICs)** – up to 15 TICs must be reported, if present. If a reduced target analyte list is requested by client, TICs are not reported.

## Table E-1

## NJDEP 2014 Target Analytes and Reporting Limits via EPA Method TO-15

| Required Compound Name | CAS Number | Molecular Weight | Reporting Limit ppbV | Reporting Limit ug/m³ |
|---|---|---|---|---|
| Acetone | 67-64-1 | 58.08 | 5.0 | 12 |
| Allyl chloride | 107-05-1 | 76.53 | 0.2 | 0.6 |
| Benzene | 71-43-2 | 78.11 | 0.2 | 0.6 |
| Bromodichloromethane | 75-27-4 | 163.8 | 0.2 | 1 |
| Bromoform | 75-25-2 | 252.8 | 0.2 | 2 |
| Bromomethane | 74-83-9 | 94.94 | 0.2 | 0.8 |
| 1,3-Butadiene | 106-99-0 | 54.09 | 0.2 | 0.4 |
| Chlorobenzene | 108-90-7 | 112.6 | 0.2 | 0.9 |
| Chloroethane | 75-00-3 | 64.52 | 0.5 | 1 |
| Chloroform | 67-66-3 | 119.4 | 0.2 | 1 |
| Chloromethane | 74-87-3 | 50.49 | 0.5 | 1 |
| Carbon disulfide | 75-15-0 | 76.14 | 0.5 | 2 |
| Carbon tetrachloride | 56-23-5 | 153.8 | 0.2 | 1 |
| 2-Chlorotoluene | 95-49-8 | 126.6 | 0.2 | 1 |
| Cyclohexane | 110-82-7 | 84.16 | 0.2 | 0.7 |
| Dibromochloromethane | 124-48-1 | 208.3 | 0.2 | 2 |
| 1,2-Dibromoethane | 106-93-4 | 187.9 | 0.2 | 2 |
| 1,2-Dichlorobenzene | 95-50-1 | 147.0 | 0.2 | 1 |
| 1,3-Dichlorobenzene | 541-73-1 | 147.0 | 0.2 | 1 |
| 1,4-Dichlorobenzene | 106-46-7 | 147.0 | 0.2 | 1 |
| Dichlorodifluoromethane | 75-71-8 | 120.9 | 0.5 | 2 |
| 1,1-Dichloroethane | 75-34-3 | 98.96 | 0.2 | 0.8 |
| 1,2-Dichloroethane | 107-06-2 | 98.96 | 0.2 | 0.8 |
| 1,1-Dichloroethene | 75-35-4 | 96.94 | 0.2 | 0.8 |
| 1,2-Dichloroethene (cis) | 156-59-2 | 96.94 | 0.2 | 0.8 |
| 1,2-Dichloroethene (trans) | 156-60-5 | 96.94 | 0.2 | 0.8 |
| 1,2-Dichloropropane | 78-87-5 | 113.0 | 0.2 | 0.9 |
| 1,3-Dichloropropene (cis) | 10061-01-5 | 111.0 | 0.2 | 0.9 |
| 1,3-Dichloropropene (trans) | 10061-02-6 | 111.0 | 0.2 | 0.9 |
| 1,2-Dichlorotetrafluoroethane | 76-14-2 | 170.9 | 0.2 | 1 |
| 1,4-Dioxane | 123-91-1 | 88.12 | 5 | 18 |
| Ethanol | 64-17-5 | 46.07 | 5 | 9 |
| Ethylbenzene | 100-41-4 | 106.2 | 0.2 | 0.9 |
| 4-Ethyltoluene | 622-96-8 | 120.2 | 0.2 | 1 |
| n-Heptane | 142-82-5 | 100.2 | 0.2 | 0.8 |
| 1,3-Hexachlorobutadiene | 87-68-3 | 260.8 | 0.2 | 2 |
| n-Hexane | 110-54-3 | 86.17 | 0.2 | 0.7 |
| Isopropanol | 67-63-0 | 60.10 | 5 | 12 |

## Table E-1 continued

| Required Compound Name | CAS Number | Molecular Weight | Reporting Limit ppbV | Reporting Limit ug/m³ |
|---|---|---|---|---|
| Methylene chloride | 75-09-2 | 84.94 | 0.5 | 2 |
| Methyl ethyl ketone | 78-93-3 | 72.11 | 0.5 | 1 |
| Methyl isobutyl ketone | 108-10-1 | 100.2 | 0.5 | 2 |
| Methyl methacrylate | 80-62-6 | 100.1 | 0.5 | 2 |
| Methyl tert-butyl ether | 1634-04-4 | 88.15 | 0.2 | 0.7 |
| Styrene | 100-42-5 | 104.1 | 0.2 | 0.9 |
| Tert-butyl alcohol | 75-65-0 | 74.12 | 5 | 15 |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | 167.9 | 0.2 | 1 |
| Tetrachloroethene | 127-18-4 | 165.8 | 0.2 | 1 |
| Tetrahydrofuran | 109-99-9 | 72.11 | 5 | 15 |
| Toluene | 108-88-3 | 92.14 | 0.2 | 0.8 |
| 1,2,4-Trichlorobenzene | 120-82-1 | 181.5 | 0.5 | 4 |
| 1,1,1-Trichloroethane | 71-55-6 | 133.4 | 0.2 | 1 |
| 1,1,2-Trichloroethane | 79-00-5 | 133.4 | 0.2 | 1 |
| Trichloroethene | 79-01-6 | 131.4 | 0.2 | 1 |
| Trichlorofluoromethane | 75-69-4 | 137.4 | 0.2 | 1 |
| 1,1,2-Trichloro-1,2,2-trifluoroethane | 76-13-1 | 187.4 | 0.2 | 2 |
| 1,2,4-Trimethylbenzene | 95-63-6 | 120.2 | 0.2 | 1 |
| 1,3,5-Trimethylbenzene | 108-67-8 | 120.2 | 0.2 | 1 |
| 2,2,4-Trimethylpentane | 540-84-1 | 114.2 | 0.2 | 0.9 |
| Vinyl bromide | 593-60-2 | 106.9 | 0.2 | 0.9 |
| Vinyl chloride | 75-01-4 | 62.50 | 0.2 | 0.5 |
| Xylenes (m&p) | 179601-23-1 | 106.2 | 0.5 | 2 |
| Xylenes (o) | 95-47-6 | 106.2 | 0.2 | 0.9 |
| Naphthalene (reported on request) | 91-20-3 | 128.2 | 0.2 | 1 |

**All reporting limits are subject to change.**

# Appendix F

## Modifications to EPA TO-15 SOP to comply with Ohio EPA Voluntary Action Program (VAP) Requirements

This Appendix defines requirements that are necessary to perform TO-15 analysis for any samples submitted via the VAP Program. No deviations from the SOP are allowed under the VAP program.

1) Sec. 3 caveat – reporting limits defined in this SOP are subject to change.

2) Sec. 7 caveat – Equipment and supplies are subject to change.

3) Initial calibration acceptance – %RSD criteria must be as defined in the EPA TO-15 method; only 2 analytes allowed greater than 30% RSD, and must be less than 40%.

4) The Laboratory check standard (LCS) may not be used as the continuing calibration verification (CCV). A CCV and LCS must be analyzed prior to samples.

5) Tentatively Identified Compounds (TICs) may not be reported as certified values.

6) CCV-10% rule not allowed. All analytes reported must be +/- 30% D. (Criteria in sections 10.4.2.6 and 2.1 do not apply to VAP samples.) All analytes in the CCV must be less than 30% RSD for VAP samples (criteria in Section 2.1, 10.4.2.6 and 10.4.2.7 do not apply.)

7) 10% Duplicate criteria not allowed. All duplicate %RPD results must be below 25%.

8) For Ohio projects, only the analytes listed in Table F-1 or as explained in the footnote to Table F-1 will be reported as certified data.

9) Affidavits are required with each report or for a series of reports generated for a particular project.

10) Surrogates are not required and will not be added to Ohio VAP samples.

11) The analysis of a Laboratory Control Sample Duplicate (LCSD) is not required and will not be analyzed for Ohio VAP samples.

12) The LCS is evaluated at +/- 30. Criteria in section 2.1 does not apply to VAP samples.

13) Corrective actions for the method blank are required if it contains analyte concentrations at or above the RL for reportable analytes.

## Table F-1

| Ohio EPA VAP Program Target Analyte List * | | |
|---|---|---|
| chloromethane | 1,2-dichloropropane | 1,1-Dichloropropene |
| vinyl chloride | 1,4-dioxane | 1,2,3-Trichloropropane |
| 1,3-butadiene | trichloroethene | 1,2,4-Trimethylbenzene |
| acetaldehyde | 2,2,4-trimethylpentane | 1,2-Dichlorobenzene |
| bromomethane | cis-1,3-dichloropropene | 1,3,5-Trimethylbenzene |
| chloroethane | 4-methyl-2-pentanone | 1,3-Dichlorobenzene |
| vinyl bromide | 1,1,2-trichloroethane | 1,3-Dichloropropane |
| acrolein | toluene | Acetone |
| acetonitrile | 1,2-dibromoethane | Bromodichloromethane |
| acrylonitrile | tetrachloroethene | Butyl acetate |
| 1,1-dichloroethene | chlorobenzene | cis-1,2-Dichloroethene |
| methylene chloride | ethylbenzene | Cyclohexane |
| 3-chloropropene | m+p-xylene | Dibromochloromethane |
| carbon disulfide | bromoform | Dibromomethane |
| 1,1-dichloroethane | styrene | Dichlorodifluoromethane |
| MTBE | 1,1,2,2-tetrachloroethane | Ethyl acetate |
| vinyl acetate | o-xylene | Ethyl ether |
| 2-butanone | isopropylbenzene | Methyl alcohol (methanol) |
| chloroform | Benzyl Chloride | Methyl Methacrylate |
| 1,2-dichloroethane | 1,4-dichlorobenzene | Naphthalene |
| hexane | 1,2-dibromo-3-chloropropane | Tetrahydrofuran |
| 1,1,1-trichloroethane | 1,2,4-trichlorobenzene | trans-1,2-Dichloroethene |
| benzene | hexachlorobutadiene | trans-1,3-Dichloropropene |
| carbon tetrachloride | 1,1,1,2-Tetrachloroethane | Trichlorofluoromethane |

***** The analytes appearing in this table are subject to change, however, any changes will not include identification of compounds that are not included in the TO-15 method referenced at the beginning of this document. However, it is acceptable to analyze and report, as certified data, compound(s) not included in the TO-15 method referenced at the beginning of this document if the compound(s) appear on the current and valid certificate issued by VAP on the date of analysis and if the laboratory has followed all applicable VAP approved documents.

# ATTACHMENT 3

Roux's Standard Operating Procedures

Date: May 5, 2000

## 1.0 PURPOSE

The purpose for this standard operating procedure (SOP) is to establish the guidelines for decontamination of all field equipment potentially exposed to contamination during drilling, and soil and water sampling. The objective of decontamination is to ensure that all drilling, and soil-sampling and water-sampling equipment is decontaminated (free of potential contaminants): 1) prior to being brought onsite to avoid the introduction of potential contaminants to the site; 2) between drilling and sampling events/activities onsite to eliminate the potential for cross-contamination between boreholes and/or wells; and 3) prior to the removal of equipment from the site to prevent the transportation of potentially contaminated equipment offsite.

In considering decontamination procedures, state and federal regulatory agency requirements must be considered because of potential variability between state and federal requirements and because of variability in the requirements of individual states. Decontamination procedures must be in compliance with state and/or federal protocols in order that regulatory agency(ies) scrutiny of the procedures and data collected do not result in non acceptance (invalidation) of the work undertaken and data collected.

## 2.0 PROCEDURE FOR DRILLING EQUIPMENT

The following is a minimum decontamination procedure for drilling equipment. Drilling equipment decontamination procedures, especially any variation from the method itemized below, will be documented on an appropriate field form or in the field notebook.

2.1 The rig and all associated equipment should be properly decontaminated by the contractor before arriving at the test site.

2.2 The augers, drilling casings, rods, samplers, tools, rig, and any piece of equipment that can come in contact (directly or indirectly) with the soil, will be steam cleaned onsite prior to set up for drilling to ensure proper decontamination.

2.3 The same steam cleaning procedures will be followed between boreholes (at a fixed on-site location[s], if appropriate) and before leaving the site at the end of the study.

2.4 All on-site steam cleaning (decontamination) activities will be monitored and documented by a member(s) of the staff of Roux Associates, Inc.

2.5 If drilling activities are conducted in the presence of thick, sticky oils (e.g., PCBs) which coat drilling equipment, then special decontamination procedures may have to be utilized before steam cleaning (e.g., hexane scrub and wash).

2.6 Containment of decontamination fluids may be necessary (e.g., rinseate from steam cleaning) or will be required (e.g., hexane), and disposal must be in accordance with state and/or federal procedures.

## 3.0 PROCEDURE FOR SOIL-SAMPLING EQUIPMENT

The following is a minimum decontamination procedure for soil-sampling equipment (e.g., split spoons, stainless-steel spatulas). Soil-sampling equipment decontamination procedures, especially any variation from the method itemized below, will be documented on an appropriate field form or in the field notebook.

3.1 Wear disposable gloves while cleaning equipment to avoid cross-contamination and change gloves as needed.

3.2 Steam clean the sampler or rinse with potable water. If soil-sampling activities are conducted in the presence of thick, sticky oils (e.g., PCBs) which coat sampling equipment, then special decontamination procedures may have to be utilized before steam cleaning and washing in detergent solution (e.g., hexane scrub and wash).

3.3 Prepare a non-phosphate, laboratory-grade detergent solution and distilled or potable water in a clean bucket.

3.4 Disassemble the sampler, as necessary and immerse all parts and other sampling equipment in the solution.

3.5 Scrub all equipment in the bucket with a brush to remove any adhering particles.

3.6 Rinse all equipment with copious amounts of potable water followed by distilled or deionized water.

3.7 Place clean equipment on a clean plastic sheet (e.g., polyethylene)

3.8 Reassemble the cleaned sampler, as necessary.

3.9 Transfer the sampler to the driller (or helper) making sure that this individual is also wearing clean gloves, or wrap the equipment with a suitable material (e.g., plastic bag, aluminum foil.

As part of the decontamination procedure for soil-sampling equipment, state and/or federal protocols must be considered. These may require procedures above those specified as minimum for Roux Associates, Inc., such as the use of nitric acid, acetone, etc. Furthermore, the containment and proper disposal of decontamination fluids must be considered with respect to regulatory agency(ies) requirements.

## 4.0 PROCEDURE FOR WATER-SAMPLING EQUIPMENT

The following is a decontamination procedure for water-sampling equipment (e.g., bailers, pumps). Water-sampling equipment decontamination procedures, especially any variation from the method itemized below, will be documented on an appropriate field form or in the field notebook.

4.1 Decontamination procedures for bailers follow:

a. Wear disposable gloves while cleaning bailer to avoid cross-contamination and change gloves as needed.

b. Prepare a non-phosphate, laboratory-grade detergent solution and potable water in a bucket.

c. Disassemble bailer (if applicable) and discard cord in an appropriate manner, and scrub each part of the bailer with a brush and solution.

d. Rinse with potable water and reassemble bailer.

e. Rinse with copious amounts of distilled or deionized water.

f. Air dry.

g. Wrap equipment with a suitable material (e.g., clean plastic bag, aluminum foil).

h. Rinse bailer at least three times with distilled or deionized water before use.

4.2 Decontamination procedures for pumps follow:

a. Wear disposable gloves while cleaning pump to avoid cross-contamination and change gloves as needed.

b. Prepare a non-phosphate, laboratory-grade detergent solution and potable water in a clean bucket, clean garbage can, or clean 55-gallon drum.

c. Flush the pump and discharge hose (if not disposable) with the detergent solution, and discard disposable tubing and/or cord in an appropriate manner.

d. Flush the pump and discharge hose (if not disposable) with potable water.

e. Place the pump on clear plastic sheeting.

f. Wipe any pump-related equipment (e.g., electrical lines, cables, discharge hose) that entered the well with a clean cloth and detergent solution, and rinse or wipe with a clean cloth and potable water.

g. Air dry.

h. Wrap equipment with a suitable material (e.g., clean plastic bag).

As part of the decontamination procedure for water-sampling equipment, state and/or federal protocols must be considered. These may require procedures above those specified as minimum for Roux Associates, Inc., such as the use of nitric acid, acetone, etc. Furthermore, the containment and proper disposal of decontamination fluids must be considered with respect to regulatory agency(ies) requirements.

Date: May 5, 2000

## 1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish guidelines for the collection of soil samples for laboratory analysis. This SOP is applicable to soil samples collected from split-spoon samplers during drilling, hand auger samples, grab samples from stockpiled soils, surface samples, test pit samples, etc.

## 2.0 CONSIDERATIONS

Soil samples may be collected in either a random or biased manner. Random samples can be based on a grid system or statistical methodology. Biased samples can be collected in areas of visible impact or suspected source areas. Soil samples can be collected at the surface, shallow subsurface, or at depth. When samples are collected at depth the water content should be noted, since generally "soil sampling" is restricted to the unsaturated zone. Equipment selection will be determined by the depth of the sample to be collected. A thorough description of the sampling locations and proposed methods of sample collection should be included in the work plan.

Commonly, surface sampling refers to the collection of samples at a 0 to 6 inch depth interval. Certain regulatory agencies may define the depth interval of a surface sample differently, and this must be defined in the work plan. Collection of surface soil samples is most efficiently accomplished with the use of a stainless steel trowel or scoop. For samples at greater depths a decontaminated bucket auger or power auger may be needed to advance the hole to the point of sample collection. Another clean bucket auger should then be used to collect the sample. To collect samples at depths of greater than approximately six feet the use of a drill rig and split spoon samples will usually be necessary. In some situations, sample locations are accessed with the use of a backhoe.

## 3.0 MATERIALS/EQUIPMENT

a. A work plan which outlines soil sampling requirements.

b. Field notebook, field form(s), maps, chain-of-custody forms, and custody seals.

c. Decontamination supplies (including: non-phosphate, laboratory grade detergent, buckets, brushes, potable water, distilled water, regulatory-required reagents, aluminum foil, plastic sheeting, etc.).

d. Sampling device (split-spoon sampler, stainless steel hand auger, stainless steel trowel, etc.).

e. Stainless steel spoons or spatulas.

f. Disposable sampling gloves.

g. Laboratory-supplied sample containers with labels.

h. Cooler with blue or wet ice.

i. Plastic sheeting.

j. Black pen and indelible marker.

k. Zip-lock bags and packing material.

l. Tape measure.

m. Paper towels or clean rags.

n. Masking and packing tape.

o. Overnight (express) mail forms.

## 4.0 DECONTAMINATION

All reusable sampling equipment will be thoroughly cleaned according to the decontamination SOP. Where possible, thoroughly pre-cleaned and wrapped sampling equipment should be used and dedicated to individual sampling locations. Disposable items such as sampling gloves, aluminum foil, and plastic sheeting will be changed after each use and discarded in an appropriate manner.

## 5.0 PROCEDURE

5.1 Prior to collecting soil samples, ensure that all sampling equipment has been thoroughly cleaned according to the decontamination SOP. If samples are to be collected at depth, then the boring must be advanced with thoroughly cleaned equipment to the desired sampling horizon and a different thoroughly cleaned sampler must be used to collect the sample.

5.2 Using disposable gloves and a pre-cleaned, stainless steel spatula or spoon, extract the soil sample from the sampler, measure the recovery, and separate the wash from the true sample. Where allowed by regulatory agency(ies), disposable plastic spoons may be used.

5.3 Place the sample in a laboratory-supplied, pre-cleaned sample container. This should be done as quickly as possible and this is especially important when sampling for volatile organic compounds (VOCs). Samples to be analyzed for VOCs must be collected prior to other constituents.

5.4 The sample container will be labeled with appropriate information such as, client name, site location, sample identification (location, depth, etc.), date and time of collection, and sampler's initials.

5.5 Using the remaining portion of soil from the sampler, log the sample in detail and record sediment characteristics (color, odor, moisture, texture, density, consistency, organic content, layering, grain size, etc.).

5.6 If soil samples are to be composited in the field, then equal portions from selected locations will be placed on a clean plastic sheet and homogenized. Alternately, several samples may be submitted to the laboratory for compositing by weight. The method used is dependent upon regulatory requirements. Specific compositing procedures shall be approved by the appropriate regulatory agency and described in the work plan. Samples to be analyzed for VOCs will not be composited unless required by a regulatory agency.

5.7 After the sample has been collected, labeled, and logged in detail, it is placed in a zip-lock bag and stored in a cooler at 4°C.

5.8 A chain-of-custody form is completed for all samples collected. One copy is retained and two are sent with the samples in a zip-lock bag to the laboratory. A custody seal is placed on the cooler prior to shipment.

5.9 Samples collected from Monday to Friday are to be delivered to the laboratory within 24 hours of collection. If Saturday delivery is unavailable, samples collected on Friday must be delivered by Monday morning. Check the work plan to determine if any analytes require a shorter delivery time.

5.10 The field notebook and appropriate forms should include, but not be limited to the following: client name, site location, sample location, sample depth, sample identification, date and time collected, sampler's name, method of sample collection, number and type of containers, geologic description of material, description of decontamination procedures, etc. A site map should be prepared with exact measurements to each sample location in case follow-up sampling is necessary.

5.11 All reusable sampling equipment must be thoroughly cleaned in accordance with the decontamination SOP. Following the final decontamination (after all samples are collected) the sampling equipment is wrapped in aluminum foil. Discard any gloves, foil, plastic, etc. in an appropriate manner that is consistent with site conditions.

END OF PROCEDURE

Date: May 5, 2000

## 1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to establish guidelines for sample handling which will allow consistent and accurate results. Valid chemistry data are integral to investigations that characterize media-quality conditions. Thus, this SOP is designed to ensure that once samples are collected, they are preserved, packed and delivered in a manner which will maintain sample integrity to as great an extent as possible. The procedures outlined are applicable to most sampling events and any required modifications must be clearly described in the work plan.

## 2.0 CONSIDERATIONS

Sample containers, sampling equipment decontamination, quality assurance/quality control (QA/QC), sample preservation, and sample handling are all components of this SOP.

### 2.1 Sample Containers

Prior to collection of a sample, considerations must be given to the type of container that will be used to store and transport the sample. The type and number of containers selected is usually based on factors such as sample matrix, potential contaminants to be encountered, analytical methods requested, and the laboratory's internal quality assurance requirements. In most cases, the overriding considerations will be the analytical methodology, or the state or federal regulatory requirements because these regulations generally encompass the other factors. The sample container selected is usually based on some combination of the following criteria:

a. Reactivity of Container Material with Sample

Choosing the proper composition of sample containers will help to ensure that the chemical and physical integrity of the sample is maintained. For sampling potentially hazardous material, glass is the recommended container type because it is chemically inert to most substances. Plastic containers are not recommended for most hazardous wastes because the potential exists for contaminants to adsorb to the surface of the plastic or for the plasticizer to leach into the sample.

In some instances, however, the sample characteristics or analytes of interest may dictate that plastic containers be used instead of glass. Because some metals species will adhere to the sides of the glass containers in an aqueous matrix, plastic bottles (e.g., nalgene) must be used for samples collected for metals analysis. A separate, plastic container should accompany glass containers if metals analysis is to be

performed along with other analyses. Likewise, other sample characteristics may dictate that glass cannot be used. For example, in the case of a strong alkali waste or hydrofluoric solution, plastic containers may be more suitable because glass containers may be etched by these compounds and create adsorptive sites on the container's surface.

b. Volume of the Container

The volume of sample to be collected will be dictated by the analysis being performed and the sample matrix. The laboratory must supply bottles of sufficient volume to perform the required analysis. In most cases, the methodology dictates the volume of sample material required to complete the analysis. However, individual laboratories may provide larger volume containers for various analytes to ensure sufficient quantities for duplicates or other QC checks.

To facilitate transfer of the sample from the sampler into the container and to minimize spillage and sample disturbance, wide-mouth containers are recommended. Aqueous volatile organic samples must be placed into 40-milliliter (ml) glass vials with polytetrafluoroethylene (PTFE) (e.g., TeflonTM) septums. Non-aqueous volatile organic samples should be collected in the same type of vials or in 4-ounce (oz) wide-mouth jars provided by the laboratory. These jars should have PTFE-lined screw caps.

c. Color of Container

Whenever possible, amber glass containers should be used to prevent photodegradation of the sample, except when samples are being collected for metals analysis. If amber containers are not available, then containers holding samples should be protected from light (i.e., place in cooler with ice immediately after filling).

d. Container Closures

Container closures must screw on and off the containers and form a leak-proof seal. Container caps must not be removed until the container is ready to be filled with the sample, and the container cap must be replaced (securely) immediately after filling it. Closures should be constructed of a material which is inert with respect to the sampled material, such as PTFE (e.g., TeflonTM). Alternately, the closure may be separated from the sample by a closure liner that is inert to the sample material such as PTFE sheeting. If soil or sediment samples are being collected, the threads of the container must be wiped clean with a dedicated paper towel or cloth so the cap can be threaded properly.

e. Decontamination of Sample Containers

Sample containers must be laboratory cleaned by the laboratory performing the analysis. The cleaning procedure is dictated by the specific analysis to be performed on the sample. Sample containers must be carefully examined to ensure that all containers appear clean. Do not mistake the preservative as unwanted residue. The bottles should not be field cleaned. If there is any question regarding the integrity of the bottle, then the laboratory must be contacted immediately and the bottle(s) replaced.

f. Sample Bottle Storage and Transport

No matter where the sample bottles are, whether at the laboratory waiting to be packed for shipment or in the field waiting to be filled with sample, care must be taken to avoid contamination. Sample shuttles or coolers, and sample bottles must be stored and transported in clean environments. Sample bottles and clean sampling equipment must never be stored near solvents, gasoline, or other equipment that is a potential source of cross-contamination. When under chain of custody, sample bottles must be secured in locked vehicles, and custody sealed in shuttles or in the presence of authorized personnel. Information which documents that proper storage and transport procedures have been followed must be included in the field notebook and on appropriate field forms.

2.2 Decontamination of Sampling Equipment

Proper decontamination of all re-usable sampling equipment is critical for all sampling episodes. The SOP for Decontamination of Field Equipment and SOPs for method-specific or instrument-specific tasks must also be referred to for guidance for decontamination of various types of equipment.

2.3 Quality Assurance/Quality Control Samples

QA/QC samples are intended to provide control over the proper collection and tracking of environmental measurements, and subsequent review, interpretation and validation of generated analytical data. The SOPs for Collection of Quality Control Samples, for Evaluation and Validation of Data, and for Field Record Keeping and Quality Assurance/Quality Control must be referred to for detailed guidance regarding these respective procedures. SOPs for method-specific or instrument-specific tasks must also be referred to for guidance for QA/QC procedures.

2.4 Sample Preservation Requirements

Certain analytical methodologies for specific analytes require chemical additives in order to stabilize and maintain sample integrity. Generally, this is accomplished under the following two scenarios:

a. Sample bottles are preserved at the laboratory prior to shipment into the field.

b. Preservatives are added in the field immediately after the samples are collected.

Many laboratories provide pre-preserved bottles as a matter of convenience and to help ensure that samples will be preserved immediately upon collection. A problem associated with this method arises if not enough sample could be collected, resulting in too much preservative in the sample. More commonly encountered problems with this method include the possibility of insufficient preservative provided to achieve the desired pH level or the need for additional preservation due to chemical reactions caused by the addition of sample liquids to pre-preserved bottles. The use of pre-preserved bottles is acceptable; however, field sampling teams must always be prepared to add additional preservatives to samples if the aforementioned situations occur. Furthermore, care must be exercised not to overfill sample bottles containing preservatives to prevent the sample and preservative from spilling and therefore diluting the preservative (i.e., not having enough preservative for the volume of sample).

When samples are preserved after collection, special care must be taken. The transportation and handling of concentrated acids in the field requires additional preparation and adherence to appropriate preservation procedures. All preservation acids used in the field should be trace-metal or higher-grade.

2.5 Sample Handling

After the proper sample bottles have been received under chain-of-custody, properly decontaminated equipment has been used to collect the sample, and appropriate preservatives have been added to maintain sample integrity, the final step for the field personnel is checking the sample bottles prior to proper packing and delivery of the samples to the laboratory.

All samples should be organized and the labels checked for accuracy. The caps should be checked for tightness and any 40-ml volatile organic compound (VOC) bottles must be checked for bubbles. Each sample bottle must be placed in an individual "zip-lock" bag to protect the label, and placed on ice. The bottles must be carefully packed to prevent breakage during transport. When several bottles have been collected for an individual sample, they should not be placed adjacent to each other in the cooler to prevent possible breakage of all bottles for a given sample. If there are any samples which are known or suspected to be highly contaminated, these should be placed in an individual cooler under separate chain-of-custody to prevent possible cross contamination. Sufficient ice (wet or blue packs) should be placed in the cooler to maintain the temperature at 4 degrees Celsius (°C) until delivery at the laboratory. Consult the work plan to determine if a particular ice is specified as the preservation for transportation (e.g., the United States Environmental Protection Agency does not like the use of

blue packs because they claim that the samples will not hold at 4°C). Blue ice packs will not be used to transport samples being analyzed for Per- and Polyfluoroalkyl Substances (PFAS). If additional coolers are required, then they should be purchased. The chain-of-custody form should be properly completed, placed in a "zip-lock" bag, and placed in the cooler. One copy must be maintained for the project files. The cooler should be sealed with packing tape and a custody seal. The custody seal number should be noted in the field book. Samples collected from Monday through Friday will be delivered to the laboratory within 24 hours of collection. If Saturday delivery is not available, samples collected on Friday must be delivered by Monday morning. Check the work plan to determine if certain analytes require a shorter delivery time. If overnight mail is utilized, then the shipping bill must be maintained for the files and the laboratory must be called the following day to confirm receipt.

## 3.0 EQUIPMENT AND MATERIALS

3.1 General equipment and materials may include, but not necessarily be limited to, the following:

a. Sample bottles of proper size and type with labels.

b. Cooler with ice (wet or blue pack; no blue packs for PFAS samples).

c. Field notebook, appropriate field form(s), chain-of-custody form(s), custody seals.

d. Black pen and indelible marker.

e. Packing tape, "bubble wrap," and "zip-lock" bags.

f. Overnight (express) mail forms and laboratory address.

g. Health and safety plan (HASP).

h. Work plan/scope of work.

i. Pertinent SOPs for specified tasks and their respective equipment and materials.

3.2 Preservatives for specific samples/analytes as specified by the laboratory. Preservatives must be stored in secure, spillproof glass containers with their content, concentration, and date of preparation and expiration clearly labeled.

3.3 Miscellaneous equipment and materials including, but not necessarily limited to, the following:

a. Graduated pipettes.

b. Pipette bulbs.

c. Litmus paper.

d. Glass stirring rods.

e. Protective goggles.

f. Disposable gloves.

g. Lab apron.

h. First aid kit.

i. Portable eye wash station.

j. Water supply for immediate flushing of spillage, if appropriate.

k. Shovel and container for immediate containerization of spillage-impacted soils, if appropriate.

## 4.0 PROCEDURE

4.1 Examine all bottles and verify that they are clean and of the proper type, number, and volume for the sampling to be conducted.

4.2 Label bottles carefully and clearly with project name and number, site location, sample identification, date, time, and the sampler's initials using an indelible marker.

4.3 Collect samples in the proper manner (refer to specific sampling SOPs).

4.4 Conduct preservation activities as required after each sample has been collected. Field preservation must be done immediately and must not be done later than 30 minutes after sample collection.

4.5 Conduct QC sampling, as required.

4.6 Seal each container carefully and place in an individual "zip lock" bag.

4.7 Organize and carefully pack all samples in the cooler immediately after collection (e.g., bubble wrap). Insulate samples so that breakage will not occur.

4.8 Complete and place the chain-of-custody form in the cooler after all samples have been collected. Maintain one copy for the project file. If the cooler is to be transferred several times prior to shipment or delivery to the laboratory, it may be easier to tape the chain-of-custody to the exterior of the sealed cooler. When exceptionally hazardous samples are known or suspected to be present, this should be identified on the chain-of-custody as a courtesy to the laboratory personnel.

4.9 Add additional ice as necessary to ensure that it will last until receipt by the laboratory.

4.10 Seal the cooler with packing tape and a custody seal. Record the number of the custody seal in the field notebook and on the field form. If there are any exceptionally hazardous samples, then shipping regulations should be examined to ensure that the sample containers and coolers are in compliance and properly labeled.

4.11 Samples collected from Monday through Friday will be delivered to the laboratory within 24 hours of collection. If Saturday delivery is not available, samples collected on Friday must be delivered by Monday morning. Check the work plan to determine if certain analytes require a shorter delivery time.

4.12 Maintain the shipping bill for the project files if overnight mail is utilized and call the laboratory the following day to confirm receipt.

END OF PROCEDURE

Date: May 5, 2000

## 1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to establish guidelines for the sampling of ground-water monitoring wells for dissolved constituents. As part of the SOP for the sampling of ground-water monitoring wells, sample collection equipment and devices must be considered, and equipment decontamination and pre-sampling procedures (e.g., measuring water levels, sounding wells, and purging wells) must be implemented. Sampling objectives must be firmly established in the work plan before considering the above.

Valid water-chemistry data are integral to a hydrogeologic investigation that characterizes ground-water quality conditions. Water-quality data are used to evaluate both current and historic aquifer chemistry conditions, as well as to estimate future conditions (e.g., trends, migration pathways). Water-quality data can be used to construct ground-water quality maps to illustrate chemical conditions within the flow system, to generate water-quality plots to depict conditions with time and trends, and to perform statistical analyses to quantify data variability, trends, and cleanup levels.

## 2.0 EQUIPMENT AND MATERIALS

2.1 In order to sample ground water from monitoring wells, specific equipment and materials are required. The equipment and materials list may include, but not necessarily be limited to, the following:

a. Bailers (Teflon™ or stainless steel).

b. Pumps (centrifugal, peristaltic, bladder, electric submersible, bilge, hand-operated diaphragm, etc.).

c. Gas-displacement device(s).

d. Air-lift device(s).

e. Teflon™ tape, electrical tape.

f. Appropriate discharge hose.

g. Appropriate discharge tubing (e.g., polypropylene, teflon, etc.) if using a peristaltic pump.

h. Appropriate compressed gas if using bladder-type or gas-displacement device.

i. Portable generator and gasoline or alternate power supply if using an electric submersible pump.

j. Non-absorbent cord (e.g., polypropylene, etc.).

k. Plastic sheeting.

l. Tape measure (stainless steel, steel, fiberglass) with 0.01-foot measurement increments and chalk (blue carpenter's).

m. Electronic water-level indicators (e.g., m-scope, etc.) or electric water-level/product level indicators.

n. Non-phosphate, laboratory-grade detergent.

o. Distilled/Deionized water.

p. Potable water.

q. Paper towels, clean rags.

r. Roux Associates' field forms (e.g., daily log, well inspection checklist, sampling, etc.) and field notebook.

s. Well location and site map.

t. Well keys.

u. Stop watch, digital watch with second increments, or watch with a second hand.

v. Water Well Handbook.

w. Calculator.

x. Black pen and water-proof marker.

y. Tools (e.g., pipe wrenches, screwdrivers, hammer, pliers, flashlight, pen knife, etc.).

z. Appropriate health and safety equipment, as specified in the site health and safety plan (HASP).

aa. pH meter(s) and buffers.

bb. Conductivity meter(s) and standards.

cc. Thermometer(s).

dd. Extra batteries (meters, thermometers, flashlight).

ee. Filtration apparatus, filters, pre-filters.

ff. Plasticware (e.g., premeasured buckets, beakers, flasks, funnels).

gg. Disposable gloves.

hh. Water jugs.

ii. Laboratory-supplied sample containers with labels.

jj. Cooler(s).

kk. Ice (wet, blue packs).

ll. Masking, duct, and packing tape.

mm. Chain-of-custody form(s) and custody seal(s).

nn. Site sampling and analysis plan (SAP).

oo. Site health and safety plan (HASP).

pp. Packing material (e.g., bubble wrap)

qq. "Zip-lock" plastic bags.

rr. Overnight (express) mail forms.

## 3.0 DECONTAMINATION

3.1 Make sure all equipment is decontaminated and cleaned before use (refer to the SOP for Decontamination of Field Equipment for detailed decontamination methods, summaries for bailers and pumps are provided below). Use new, clean materials when decontamination is not appropriate (e.g., non-absorbent cord, disposable gloves). Document, and initial and date the decontamination procedures on the appropriate field form and in the field notebook.

a. Decontaminate a bailer by: 1) wearing disposable gloves, 2) disassembling (if appropriate) and scrubbing in a non-phosphate, laboratory-grade detergent and distilled/deionized water solution, and 3) rinsing first with potable water and then distilled/deionized water.

b. Decontaminate a pump by: 1) wearing disposable gloves, 2) flushing the pump and discharge hose (if not disposable) first with a non-phosphate, laboratory-grade detergent and potable water solution in an appropriate container (clean bucket, garbage can, or 55-gallon drum) and then with

distilled/deionized water or potable water, and 3) wiping pump-related equipment (e.g., electrical lines, cables, discharge hose) first with a clean cloth and detergent solution and then rinsing or wiping with a clean cloth and distilled/deionized water or potable water.

3.2 Note that the decontamination procedures for bailers and pumps are the minimum that must be performed. Check the work plan to determine if chemicals specified by individual state regulatory agencies must also be used for decontamination procedures (e.g., hexane, nitric acid, acetone, isopropanol, etc.).

## 4.0 CALIBRATION OF FIELD ANALYSIS EQUIPMENT

Calibrate field analysis equipment before use (e.g., thermometers, pH and conductivity meters, etc.). Refer to the specific SOP for field analysis for each respective piece of equipment. Document, and initial and date the calibration procedures on the appropriate field form, in the field notebook, and in the calibration log book.

## 5.0 PROCEDURE

5.1 Document, and initial and date well identification, pre-sampling information, and problems encountered on the appropriate field form and in the field notebook as needed.

5.2 Inspect the protective casing of the well and the well casing, and note any items of concern such as a missing lock, or bent or damaged casing(s).

5.3 Place plastic sheeting around the well to protect sampling equipment from potential cross contamination.

5.4 Remove the well cap or plug and, if necessary, clean the top of the well off with a clean rag. Place the cap or plug on the plastic sheeting. If the well is not vented, allow several minutes for the water level in the well to equilibrate. If fumes or gases are present, then diagnose these with the proper safety equipment. Never inhale the vapors.

5.5 Measure the depth to water (DTW) from the measuring point (MP) on the well using a steel tape and chalk or an electronic sounding device (m-scope). Refer to the specific SOPs for details regarding the use of a steel tape or a m-scope for measuring water levels. Calculate the water-level elevation. Document, and initial and date the information on the appropriate field form and in the field notebook.

5.6 Measuring the total depth of the well from the MP with a weighted steel tape. Calculate and record the volume of standing water in the well casing on the appropriate field form and in the field notebook.

5.7 Decontaminate the equipment used to measure the water level and sound the well with a non-phosphate, laboratory-grade detergent solution followed by a distilled/deionized water rinse.

5.8 Purge the well prior to sampling (refer to the SOP for Purging a Well). The well should be pumped or bailed to remove the volume of water specified in the work plan. Usually three to five casing volumes are removed if the recharge rate is adequate to accomplish this within a reasonable amount of time.

If the formation cannot produce enough water to sustain purging, then one of two options must be followed. These include: 1) pumping or bailing the well dry, or 2) pumping or bailing the well to "near-dry" conditions (i.e., leaving some water in the well). The option employed must be specified in the work plan and be in accordance with regulatory requirements.

If the well is purged dry, then all the standing water has been removed and upon recovery the well is ready for sampling. However, depending on the rate of recovery and the time needed to complete the sampling round, one of the following procedures may have to be implemented: 1) the well may have to be sampled over a period of more than one day; 2) the well may not yield enough water to collect a complete suite of samples and only select (most important) samples will be collected; or 3) the well may not recover which will preclude sampling. Regardless of the option that must be followed, the sampling procedure must be fully documented. When preparing to conduct a sampling round, review drilling, development and previous sampling information (if available) to identify low-yielding wells in order to purge them first, and potentially allow time for the well to recover for sampling.

5.9 Record the physical appearance of the water (i.e., color, turbidity, odor, etc.) on the appropriate field form and in the field notebook, as it is purged. Note any changes that occur during purging.

5.10 If a bailer is used to collect the sample, then:

a. Flush the decontaminated bailer three times with distilled/deionized water.

b. Tie the non-absorbent cord (polypropylene) to the bailer with a secure knot and then tie the free end of the bailer cord to the protective casing or, if possible, some nearby structure to prevent losing the bailer and cord down the well.

c. Lower the bailer slowly down the well and into the water column to minimize disturbance of the water surface. If a bottom-filling bailer is used, then do not submerge the top of the bailer; however, if a top-filling bailer is used, then submerge the bailer several feet below the water surface.

d. Remove and properly discard one bailer volume from the well to rinse the bailer with well water before sampling. Again, lower the bailer slowly down the well to the appropriate depth depending on the bailer type (as discussed above in 5.11 c). When removing the bailer from the well, do not allow the bailer cord to rest on the ground but coil it on the protective plastic sheeting placed around the well. Certain regulatory agencies require that the first bailer volume collected be utilized for the samples.

5.11 If a pump is used to collect the sample, then use the same pump used to purge the well and, if need be, reduce the discharge rate to facilitate filling sample containers and to avoid problems that can occur while filling sample containers (as listed in Number 5.14, below). Alternately, the purge pump may be removed and a thoroughly decontaminated bailer can be used to collect the sample.

5.12 Remove each appropriate container's cap only when ready to fill each with the water sample, and then replace and secure the cap immediately.

5.13 Fill each appropriate, pre-labeled sample container carefully and cautiously to prevent: 1) agitating or creating turbulence; 2) breaking the container; 3) entry of, or contact with, any other medium; and 4) spilling/splashing the sample and exposing the sampling team to contaminated water. Immediately place the filled sample container in a ice-filled (wet ice or blue pack) cooler for storage. If wet ice is used it is recommended that it be repackaged in zip-lock bags to help keep the cooler dry and the sample labels secure. Check the work plan as to whether wet ice or blue packs are specified for cooling the samples because certain regulatory agencies may specify the use of one and not the other.

5.14 "Top-off" containers for volatile organic compounds (VOCs) and tightly seal with Teflon™-lined septums held in place by open-top screw caps to prevent volatilization. Ensure that there are no bubbles by turning the container upside down and tapping it gently.

5.15 Filter water samples (Procedure 4.6) collected for dissolved metals analysis prior to preservation to remove the suspended sediment from the sample. If water samples are to be collected for total metals analysis, then collect a second set of samples without field filtering.

In the event that the regulatory agency(ies) want unfiltered samples for metals analysis, a second set of filtered samples should also be collected. Because unfiltered samples are indications of total metals (dissolved and suspended) they are not representative of aquifer conditions because ground water does not transport sediment (except in some rare cases). Thus, the results for dissolved metals in ground water should be based on filtered samples even if both filtered and unfiltered sets are presented in a report.

5.16 Add any necessary preservative(s) to the appropriate container(s) prior to, or after (preferred), the collection of the sample, unless the appropriate preservative(s) have already been added by the laboratory before shipment.

5.17 Collect quality control (QC) samples as required in the work plan to monitor sampling and laboratory performance. Refer to the SOP for Collection of Quality Control Samples.

5.18 Conduct field analyses after sample collection is complete by measuring and recording the temperature, conductivity, pH, etc. (as called for in the work plan). Note and record the "final" physical appearance of the water (after purging and sampling) on an appropriate field form and in the field notebook.

5.19 Wipe the well cap with a clean rag, replace the well cap and protective cover (if present). Lock the protective cover.

5.20 Verify that each sample is placed in an individual "zip-lock" bag, wrapped with "bubble wrap," placed in the cooler, and that the cooler has sufficient ice (wet ice or blue packs) to preserve the samples for transportation to the analytical laboratory.

5.21 Decontaminate bailers, hoses, and pumps as discussed in the decontamination SOP. Wrap decontaminated equipment with a suitable material (e.g., clean plastic bag or aluminum foil). Discard cords, rags, gloves, etc. in a manner consistent with site conditions.

5.22 Complete all necessary field forms, field notebook entries, and the chain-of-custody forms. Retain one copy of each chain-of-custody form. Secure the cooler with sufficient packing tape and a custody seal.

5.23 Samples collected from Monday through Friday will be delivered within 24 hours of collection. If Saturday delivery is not available, samples collected on Friday must be delivered by Monday morning. Consult the work plan to determine if any of the analytes require a shorter delivery time.

END OF PROCUDURE

# ATTACHMENT 4

NYSDEC PFAS Sampling Guidance

NEW YORK STATE OF OPPORTUNITY.

Department of Environmental Conservation

# SAMPLING, ANALYSIS, AND ASSESSMENT OF PER- AND POLYFLUOROALKYL SUBSTANCES (PFAS)

## Under NYSDEC's Part 375 Remedial Programs

April 2023



www.dec.ny.gov

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ERRATA SHEET for

*SAMPLING, ANALYSIS, AND ASSESSMENT OF PER- AND POLYFLUOROALKYL SUBSTANCES (PFAS) Under NYSDEC's Part 375 Remedial Programs* Issued January 17, 2020

| Citation and Page Number | Current Text | Corrected Text | Date |
|---|---|---|---|
| Title of Appendix I, page 32 | Appendix H | Appendix I | 2/25/2020 |
| Document Cover, page 1 | Guidelines for Sampling and Analysis of PFAS | Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) Under NYSDEC's Part 375 Remedial Programs | 9/15/2020 |
| Data Assessment and Application to Site Cleanup Page 3 | Until such time as Ambient Water Quality Standards (AWQS) and Soil Cleanup Objectives (SCOs) for PFOA and PFOS are published | Until such time as Soil Cleanup Objectives (SCOs) for PFOA and PFOS are published | 3/28/2023 |
| Water Sample Results Page 3 | PFOA and PFOS should be further assessed and considered as potential contaminants of concern in groundwater or surface water if PFOA or PFOS is detected in any water sample at or above 10 ng/L (ppt) and is determined to be attributable to the site, either by a comparison of upgradient and downgradient levels, or the presence of soil source areas, as defined below. | NYSDEC has adopted ambient water quality guidance values for PFOA and PFOS. Groundwater samples should be compared to the human health criteria of 6.7 ng/l (ppt) for PFOA and 2.7 ng/l (ppt) for PFOS. These guidance values also include criteria for surface water for PFOS applicable for aquatic life, which may be applicable at some sites. Drinking water sample results should be compared to the NYS maximum contaminant level (MCL) of 10 ng/l (ppt).Analysis to determine if PFOA and PFOS concentrations are attributable to the site should include a comparison between upgradient and downgradient levels, and the presence of soil source areas, as defined below. | 3/28/2023 |
| Soil Sample Results Page 3 | Soil cleanup objectives for PFOA and PFOS have been proposed in an upcoming revision to 6 NYCRR Part 375-6. Until SCOs are in effect, the following are to be used as guidance values: | NYSDEC will delay adding soil cleanup objectives for PFOA and PFOS to 6 NYCRR Part 375-6 until the PFAS rural soil background study has been completed. Until SCOs are in effect, the following are to be used as guidance values: | 3/28/2023 |
| Protection of Groundwater Page 3 | PFOA (ppb) 1.1<br>PFOS (ppb) 3.7 | PFOA (ppb) 0.8<br>PFOS (ppb) 1.0 | 3/28/2023 |

| Citation and Page Number | Current Text | Corrected Text | Date |
|---|---|---|---|
| Footnote 2 Page 3 | The movement of PFAS in the environment is being aggressively researched at this time; that research will eventually result in more accurate models for the behaviors of these chemicals. In the meantime, DEC has calculated the guidance value for the protection of groundwater using the same procedure used for all other chemicals, as described in Section 7.7 of the Technical Support Document (http://www.dec.ny.gov/docs/remediation_hudson_pdf/techsuppdoc.pdf). | The Protection of Groundwater values are based on the above referenced ambient groundwater guidance values. Details on that calculation are available in the following document, prepared for the February 2022 proposed changes to Part 375 (https://www.dec.ny.gov/docs/remediation_hudson_pdf/part375techsupport.pdf). The movement of PFAS in the environment is being aggressively researched at this time; that research will eventually result in more accurate models for the behaviors of these chemicals. In the meantime, DEC has calculated the guidance value for the protection of groundwater using the same procedure used for all other chemicals, as described in Section 7.7 of the Technical Support Document (http://www.dec.ny.gov/docs/remediation_hudson_pdf/techsuppdoc.pdf). | 3/28/2023 |
| Testing for Imported Soil Page 4 | If the concentrations of PFOA and PFOS in leachate are at or above 10 ppt (the Maximum Contaminant Levels established for drinking water by the New York State Department of Health), then the soil is not acceptable. | If the concentrations of PFOA and PFOS in leachate are at or above the ambient water quality guidance values for groundwater, then the soil is not acceptable. | 3/28/2023 |
| Routine Analysis, page 9 | "However, laboratories analyzing environmental samples…PFOA and PFOS in drinking water by EPA Method 537, 537.1 or ISO 25101." | "However, laboratories analyzing environmental samples…PFOA and PFOS in drinking water by EPA Method 537, 537.1, ISO 25101, or Method 533." | 9/15/2020 |
| Additional Analysis, page 9, new paragraph regarding soil parameters | None | "In cases where site-specific cleanup objectives for PFOA and PFOS are to be assessed, soil parameters, such as Total Organic Carbon (EPA Method 9060), soil pH (EPA Method 9045), clay content (percent), and cation exchange capacity (EPA Method 9081), should be included in the analysis to help evaluate factors affecting the leachability of PFAS in site soils." | 9/15/2020 |

| Citation and Page Number | Current Text | Corrected Text | Date |
|---|---|---|---|
| Data Assessment and Application to Site Cleanup Page 10 | Until such time as Ambient Water Quality Standards (AWQS) and Soil Cleanup Objectives (SCOs) for PFAS are published, the extent of contaminated media potentially subject to remediation should be determined on a case-by-case basis using the procedures discussed below and the criteria in DER-10. Target levels for cleanup of PFAS in other media, including biota and sediment, have not yet been established by the DEC. | Until such time as Ambient Water Quality Standards (AWQS) and Soil Cleanup Objectives (SCOs) for PFOA and PFOS are published, the extent of contaminated media potentially subject to remediation should be determined on a case-by-case basis using the procedures discussed below and the criteria in DER-10. Preliminary target levels for cleanup of PFOA and PFOS in other media, including biota and sediment, have not yet been established by the DEC. | 9/15/2020 |
| Water Sample Results Page 10 | PFAS should be further assessed and considered as a potential contaminant of concern in groundwater or surface water (…)<br>If PFAS are identified as a contaminant of concern for a site, they should be assessed as part of the remedy selection process in accordance with Part 375 and DER-10. | PFOA and PFOS should be further assessed and considered as potential contaminants of concern in groundwater or surface water (…)<br>If PFOA and/or PFOS are identified as contaminants of concern for a site, they should be assessed as part of the remedy selection process in accordance with Part 375 and DER-10. | 9/15/2020 |

| Citation and Page Number | Current Text | Corrected Text | Date |
|---|---|---|---|
| Soil Sample Results, page 10 | "The extent of soil contamination for purposes of delineation and remedy selection should be determined by having certain soil samples tested by Synthetic Precipitation Leaching Procedure (SPLP) and the leachate analyzed for PFAS. Soil exhibiting SPLP results above 70 ppt for either PFOA or PFOS (individually or combined) are to be evaluated during the cleanup phase." | "Soil cleanup objectives for PFOA and PFOS will be proposed in an upcoming revision to 6 NYCRR Part 375-6. Until SCOs are in effect, the following are to be used as guidance values. "<br><br>[Interim SCO Table]<br>"PFOA and PFOS results for soil are to be compared against the guidance values listed above. These guidance values are to be used in determining whether PFOA and PFOS are contaminants of concern for the site and for determining remedial action objectives and cleanup requirements. Site-specific remedial objectives for protection of groundwater can also be presented for evaluation by DEC. Development of site-specific remedial objectives for protection of groundwater will require analysis of additional soil parameters relating to leachability. These additional analyses can include any or all the parameters listed above (soil pH, cation exchange capacity, etc.) and/or use of SPLP.<br><br>As the understanding of PFAS transport improves, DEC welcomes proposals for site-specific remedial objectives for protection of groundwater. DEC will expect that those may be dependent on additional factors including soil pH, aqueous pH, % organic carbon, % Sand/Silt/Clay, soil cations: K, Ca, Mg, Na, Fe, Al, cation exchange capacity, and anion exchange capacity. Site-specific remedial objectives should also consider the dilution attenuation factor (DAF). The NJDEP publication on DAF can be used as a reference: https://www.nj.gov/dep/srp/guidance/rs/daf.pdf. " | 9/15/2020 |

| Citation and Page Number | Current Text | Corrected Text | Date |
|---|---|---|---|
| Testing for Imported Soil Page 11 | Soil imported to a site for use in a soil cap, soil cover, or as backfill is to be tested for PFAS in general conformance with DER-10, Section 5.4(e) for the PFAS Analyte List (Appendix F) using the analytical procedures discussed below and the criteria in DER-10 associated with SVOCs. If PFOA or PFOS is detected in any sample at or above 1 µg/kg, then soil should be tested by SPLP and the leachate analyzed for PFAS. If the SPLP results exceed 10 ppt for either PFOA or PFOS (individually) then the source of backfill should be rejected, unless a site-specific exemption is provided by DER. SPLP leachate criteria is based on the Maximum Contaminant Levels proposed for drinking water by New York State's Department of Health, this value may be updated based on future Federal or State promulgated regulatory standards. Remedial parties have the option of analyzing samples concurrently for both PFAS in soil and in the SPLP leachate to minimize project delays. Category B deliverables should be submitted for backfill samples, though a DUSR is not required. | Testing for PFAS should be included any time a full TAL/TCL analyte list is required. Results for PFOA and PFOS should be compared to the applicable guidance values. If PFOA or PFOS is detected in any sample at or above the guidance values then the source of backfill should be rejected, unless a site-specific exemption is provided by DER based on SPLP testing, for example. If the concentrations of PFOA and PFOS in leachate are at or above 10 ppt (the Maximum Contaminant Levels established for drinking water by the New York State Department of Health), then the soil is not acceptable.<br><br>PFOA, PFOS and 1,4-dioxane are all considered semi-volatile compounds, so composite samples are appropriate for these compounds when sampling in accordance with DER-10, Table 5.4(e)10. Category B deliverables should be submitted for backfill samples, though a DUSR is not required. | 9/15/2020 |

| Citation and Page Number | Current Text | Corrected Text | Date |
|---|---|---|---|
| Footnotes | None | [1] TOP Assay analysis of highly contaminated samples, such as those from an AFFF (aqueous film-forming foam) site, can result in incomplete oxidation of the samples and an underestimation of the total perfluoroalkyl substances.<br>[2] The movement of PFAS in the environment is being aggressively researched at this time; that research will eventually result in more accurate models for the behaviors of these chemicals. In the meantime, DEC has calculated the soil cleanup objective for the protection of groundwater using the same procedure used for all other chemicals, as described in Section 7.7 of the Technical Support Document (http://www.dec.ny.gov/docs/remediation_hudson_pdf/techsuppdoc.pdf). | 9/15/2020 |
| Additional Analysis, page 9 | In cases… soil parameters, such as Total Organic Carbon (EPA Method 9060), soil… | In cases… soil parameters, such as Total Organic Carbon (Lloyd Kahn), soil… | 1/8/2021 |
| Appendix A, General Guidelines, fourth bullet | List the ELAP-approved lab(s) to be used for analysis of samples | List the ELAP- certified lab(s) to be used for analysis of samples | 1/8/2021 |
| Appendix E, Laboratory Analysis and Containers | Drinking water samples collected using this protocol are intended to be analyzed for PFAS by ISO Method 25101. | Drinking water samples collected using this protocol are intended to be analyzed for PFAS by EPA Method 537, 537.1, 533, or ISO Method 25101 | 1/8/2021 |
| Water Sample Results Page 9 | "In addition, further assessment of water may be warranted if either of the following screening levels are met:<br>a. any other individual PFAS (not PFOA or PFOS) is detected in water at or above 100 ng/L; or<br>b. total concentration of PFAS (including PFOA and PFOS) is detected in water at or above 500 ng/L" | Deleted | 6/15/2021 |

| Citation and Page Number | Current Text | Corrected Text | Date |
|---|---|---|---|
| Routine Analysis, Page XX | Currently, New York State Department of Health's Environmental Laboratory Approval Program (ELAP)… criteria set forth in the DER's laboratory guidelines for PFAS in non-potable water and solids (Appendix H - Laboratory Guidelines for Analysis of PFAS in Non-Potable Water and Solids). | Deleted | 5/31/2022 |
| Analysis and Reporting, Page XX | As of October 2020, the United States Environmental Protection Agency (EPA) does not have a validated method for analysis of PFAS for media commonly analyzed under DER remedial programs (non-potable waters, solids). DER has developed the following guidelines to ensure consistency in analysis and reporting of PFAS. | Deleted | 5/31/2022 |
| Routine Analysis, Page XX | LC-MS/MS analysis for PFAS using methodologies based on EPA Method 537.1 is the procedure to use for environmental samples. Isotope dilution techniques should be utilized for the analysis of PFAS in all media. | EPA Method 1633 is the procedure to use for environmental samples. | |
| Soil Sample Results, Page XX | Soil cleanup objectives for PFOA and PFOS will be proposed in an upcoming revision to 6 NYCRR Part 375-6 | Soil cleanup objectives for PFOA and PFOS have been proposed in an upcoming revision to 6 NYCRR Part 375-6 | |
| Appendix A | "Include in the text… LC-MS/MS for PFAS using methodologies based on EPA Method 537.1" | "Include in the text ….EPA Method 1633" | |
| Appendix A | "Laboratory should have ELAP certification for PFOA and PFOS in drinking water by EPA Method 537, 537.1, EPA Method 533, or ISO 25101" | Deleted | |
| Appendix B | "Samples collected using this protocol are intended to be analyzed for PFAS using methodologies based on EPA Method 537.1" | "Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633" | |

| Citation and Page Number | Current Text | Corrected Text | Date |
|---|---|---|---|
| Appendix C | "Samples collected using this protocol are intended to be analyzed for PFAS using methodologies based on EPA Method 537.1" | "Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633" | |
| Appendix D | "Samples collected using this protocol are intended to be analyzed for PFAS using methodologies based on EPA Method 537.1" | "Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633" | |
| Appendix G | | Updated to include all forty PFAS analytes in EPA Method 533 | |
| Appendix H | | Deleted | |
| Appendix I | Appendix I | Appendix H | |
| Appendix H | "These guidelines are intended to be used for the validation of PFAS analytical results for projects within the Division of Environmental Remediation (DER) as well as aid in the preparation of a data usability summary report." | "These guidelines are intended to be used for the validation of PFAS using EPA Method 1633 for projects within the Division of Environmental Remediation (DER)." | |
| Appendix H | "The holding time is 14 days…" | "The holding time is 28 days…" | |
| Appendix H, Initial Calibration | "The initial calibration should contain a minimum of five standards for linear fit…" | "The initial calibration should contain a minimum of six standards for linear fit…" | |
| Appendix H, Initial Calibration | Linear fit calibration curves should have an $R^2$ value greater than 0.990. | Deleted | |
| Appendix H, Initial Calibration Verification | Initial Calibration Verification Section | Deleted | |
| Appendix H | secondary Ion Monitoring Section | Deleted | |
| Appendix H | Branched and Linear Isomers Section | Deleted | |

# Sampling, Analysis, and Assessment of Per- and Polyfluoroalkyl Substances (PFAS) Under NYSDEC's Part 375 Remedial Programs

## Objective

New York State Department of Environmental Conservation's Division of Environmental Remediation (DER) performs or oversees sampling of environmental media and subsequent analysis of PFAS as part of remedial programs implemented under 6 NYCRR Part 375. To ensure consistency in sampling, analysis, reporting, and assessment of PFAS, DER has developed this document which summarizes currently accepted procedures and updates previous DER technical guidance pertaining to PFAS.

## Applicability

All work plans submitted to DEC pursuant to one of the remedial programs under Part 375 shall include PFAS sampling and analysis procedures that conform to the guidelines provided herein.

As part of a site investigation or remedial action compliance program, whenever samples of potentially affected media are collected and analyzed for the standard Target Analyte List/Target Compound List (TAL/TCL), PFAS analysis should also be performed. Potentially affected media can include soil, groundwater, surface water, and sediment. Based upon the potential for biota to be affected, biota sampling and analysis for PFAS may also be warranted as determined pursuant to a Fish and Wildlife Impact Analysis. Soil vapor sampling for PFAS is not required.

## Field Sampling Procedures

DER-10 specifies technical guidance applicable to DER's remedial programs. Given the prevalence and use of PFAS, DER has developed "best management practices" specific to sampling for PFAS. As specified in DER-10 Chapter 2, quality assurance procedures are to be submitted with investigation work plans. Typically, these procedures are incorporated into a work plan, or submitted as a stand-alone document (e.g., a Quality Assurance Project Plan). Quality assurance guidelines for PFAS are listed in Appendix A - Quality Assurance Project Plan (QAPP) Guidelines for PFAS.

Field sampling for PFAS performed under DER remedial programs should follow the appropriate procedures outlined for soils, sediments, or other solids (Appendix B), non-potable groundwater (Appendix C), surface water (Appendix D), public or private water supply wells (Appendix E), and fish tissue (Appendix F).

QA/QC samples (e.g. duplicates, MS/MSD) should be collected as specified in DER-10, Section 2.3(c). For sampling equipment coming in contact with aqueous samples only, rinsate or equipment blanks should be collected. Equipment blanks should be collected at a minimum frequency of one per day per site or one per twenty samples, whichever is more frequent.

## Analysis and Reporting

The investigation work plan should describe analysis and reporting procedures, including laboratory analytical procedures for the methods discussed below. As specified in DER-10 Section 2.2, laboratories should provide a full Category B deliverable. In addition, a Data Usability Summary Report (DUSR) should be prepared by an independent, third-party data validator. Electronic data submissions should meet the requirements provided at: https://www.dec.ny.gov/chemical/62440.html.

DER has developed a *PFAS Analyte List* (Appendix G) for remedial programs to understand the nature of contamination at sites. It is expected that reported results for PFAS will include, at a minimum, all the compounds listed. If lab and/or matrix specific issues are encountered for any analytes, the DER project manager, in consultation with the DER chemist, will make case-by-case decisions as to whether certain analytes may be temporarily or permanently discontinued from analysis at each site. As with other contaminants that are analyzed for at a site, the *PFAS Analyte List* may be refined for future sampling events based on investigative findings.

### Routine Analysis

EPA Method 1633 is the procedure to use for environmental samples. Reporting limits for PFOA and PFOS in aqueous samples should not exceed 2 ng/L. Reporting limits for PFOA and PFOS in solid samples should not exceed 0.5 µg/kg. Reporting limits for all other PFAS in aqueous and solid media should be as close to these limits as possible. If laboratories indicate that they are not able to achieve these reporting limits for the entire *PFAS Analyte List*, site-specific decisions regarding acceptance of elevated reporting limits for specific PFAS can be made by the DER project manager in consultation with the DER chemist. Data review guidelines were developed by DER to ensure data comparability and usability (Appendix H - Data Review Guidelines for Analysis of PFAS in Non-Potable Water and Solids).

### Additional Analysis

Additional laboratory methods for analysis of PFAS may be warranted at a site, such as the Synthetic Precipitation Leaching Procedure (SPLP) and Total Oxidizable Precursor Assay (TOP Assay).

In cases where site-specific cleanup objectives for PFOA and PFOS are to be assessed, soil parameters, such as Total Organic Carbon (Lloyd Kahn), soil pH (EPA Method 9045), clay content (percent), and cation exchange capacity (EPA Method 9081), should be included in the analysis to help evaluate factors affecting the leachability of PFAS in site soils.

SPLP is a technique used to determine the mobility of chemicals in liquids, soils and wastes, and may be useful in determining the need for addressing PFAS-containing material as part of the remedy. SPLP by EPA Method 1312 should be used unless otherwise specified by the DER project manager in consultation with the DER chemist.

Impacted materials can be made up of PFAS that are not analyzable by routine analytical methodology. A TOP Assay can be utilized to conceptualize the amount and type of oxidizable PFAS which could be liberated in the environment, which approximates the maximum concentration of perfluoroalkyl substances that could be generated if all polyfluoroalkyl substances were oxidized. For example, some polyfluoroalkyl substances may degrade or transform to form perfluoroalkyl substances (such as PFOA or PFOS), resulting in an increase in perfluoroalkyl substance concentrations as contaminated groundwater moves away from a source. The TOP Assay converts, through oxidation, polyfluoroalkyl substances (precursors) into perfluoroalkyl substances that can be detected by routine analytical methodology.[1]

[1] TOP Assay analysis of highly contaminated samples, such as those from an AFFF (aqueous film-forming foam) site, can result in incomplete oxidation of the samples and an underestimation of the total perfluoroalkyl substances.

Commercial laboratories have adopted methods which allow for the quantification of targeted PFAS in air and biota. The EPA's Office of Research and Development (ORD) is currently developing methods which allow for air emissions characterization of PFAS, including both targeted and non-targeted analysis of PFAS. Consult with the DER project manager and the DER chemist for assistance on analyzing biota/tissue and air samples.

## Data Assessment and Application to Site Cleanup

Until such time as Soil Cleanup Objectives (SCOs) for PFOA and PFOS are published, the extent of contaminated media potentially subject to remediation should be determined on a case-by-case basis using the procedures discussed below and the criteria in DER-10. Preliminary target levels for cleanup of PFOA and PFOS in other media, including biota and sediment, have not yet been established by the DEC.

### Water Sample Results

NYSDEC has adopted ambient water quality guidance values for PFOA and PFOS. Groundwater samples should be compared to the human health criteria of 6.7 ng/l (ppt) for PFOA and 2.7 ng/l (ppt) for PFOS. These human health criteria should also be applied to surface water that is used as a water supply. This guidance also includes criteria for surface water for PFOS applicable for aquatic life, which may be applicable at some sites. Drinking water sample results should be compared to the NYS maximum contaminant level (MCL) of 10 ng/l (ppt). Analysis to determine if PFOA and PFOS concentrations are attributable to the site should include a comparison between upgradient and downgradient levels, and the presence of soil source areas, as defined below.

If PFOA and/or PFOS are identified as contaminants of concern for a site, they should be assessed as part of the remedy selection process in accordance with Part 375 and DER-10.

### Soil Sample Results

NYSDEC will delay adding soil cleanup objectives for PFOA and PFOS to 6 NYCRR Part 375-6 until the PFAS rural soil background study has been completed. Until SCOs are in effect, the following are to be used as guidance values:

| Guidance Values for Anticipated Site Use | PFOA (ppb) | PFOS (ppb) |
|---|---|---|
| Unrestricted | 0.66 | 0.88 |
| Residential | 6.6 | 8.8 |
| Restricted Residential | 33 | 44 |
| Commercial | 500 | 440 |
| Industrial | 600 | 440 |
| Protection of Groundwater[2] | 0.8 | 1.0 |

PFOA and PFOS results for soil are to be compared against the guidance values listed above. These guidance values are to be used in determining whether PFOA and PFOS are contaminants of concern for the site and for determining remedial action objectives and cleanup requirements. Site-specific remedial objectives for protection of groundwater can also be presented for evaluation by DEC. Development of site-specific remedial objectives for protection of groundwater will require analysis of additional soil parameters relating to leachability. These

[2] The Protection of Groundwater values are based on the above referenced ambient groundwater guidance values. Details on that calculation are available in the following document, prepared for the February 2022 proposed changes to Part 375 (https://www.dec.ny.gov/docs/remediation_hudson_pdf/part375techsupport.pdf). The movement of PFAS in the environment is being aggressively researched at this time; that research will eventually result in more accurate models for the behaviors of these chemicals. In the meantime, DEC has calculated the guidance value for the protection of groundwater using the same procedure used for all other chemicals, as described in Section 7.7 of the Technical Support Document (http://www.dec.ny.gov/docs/remediation_hudson_pdf/techsuppdoc.pdf).

additional analyses can include any or all the parameters listed above (soil pH, cation exchange capacity, etc.) and/or use of SPLP.

As the understanding of PFAS transport improves, DEC welcomes proposals for site-specific remedial objectives for protection of groundwater. DEC will expect that those may be dependent on additional factors including soil pH, aqueous pH, % organic carbon, % Sand/Silt/Clay, soil cations: K, Ca, Mg, Na, Fe, Al, cation exchange capacity, and anion exchange capacity. Site-specific remedial objectives should also consider the dilution attenuation factor (DAF). The NJDEP publication on DAF can be used as a reference: https://www.nj.gov/dep/srp/guidance/rs/daf.pdf.

## Testing for Imported Soil

Testing for PFAS should be included any time a full TAL/TCL analyte list is required. Results for PFOA and PFOS should be compared to the applicable guidance values. If PFOA or PFOS is detected in any sample at or above the guidance values then the source of backfill should be rejected, unless a site-specific exemption is provided by DER based on SPLP testing, for example. If the concentrations of PFOA and PFOS in leachate are at or above the ambient water quality guidance values for groundwater, then the soil is not acceptable.

PFOA, PFOS and 1,4-dioxane are all considered semi-volatile compounds, so composite samples are appropriate for these compounds when sampling in accordance with DER-10, Table 5.4(e)10. Category B deliverables should be submitted for backfill samples, though a DUSR is not required.

# Appendix A - Quality Assurance Project Plan (QAPP) Guidelines for PFAS

The following guidelines (general and PFAS-specific) can be used to assist with the development of a QAPP for projects within DER involving sampling and analysis of PFAS.

## General Guidelines in Accordance with DER-10

- Document/work plan section title – Quality Assurance Project Plan
- Summarize project scope, goals, and objectives
- Provide project organization including names and resumes of the project manager, Quality Assurance Officer (QAO), field staff, and Data Validator
    - The QAO should not have another position on the project, such as project or task manager, that involves project productivity or profitability as a job performance criterion
- List the ELAP certified lab(s) to be used for analysis of samples
- Include a site map showing sample locations
- Provide detailed sampling procedures for each matrix
- Include Data Quality Usability Objectives
- List equipment decontamination procedures
- Include an "Analytical Methods/Quality Assurance Summary Table" specifying:
    - Matrix type
    - Number or frequency of samples to be collected per matrix
    - Number of field and trip blanks per matrix
    - Analytical parameters to be measured per matrix
    - Analytical methods to be used per matrix with minimum reporting limits
    - Number and type of matrix spike and matrix spike duplicate samples to be collected
    - Number and type of duplicate samples to be collected
    - Sample preservation to be used per analytical method and sample matrix
    - Sample container volume and type to be used per analytical method and sample matrix
    - Sample holding time to be used per analytical method and sample matrix
- Specify Category B laboratory data deliverables and preparation of a DUSR

## Specific Guidelines for PFAS

- Include in the text that sampling for PFAS will take place
- Include in the text that PFAS will be analyzed by EPA Method 1633
- Include the list of PFAS compounds to be analyzed (*PFAS Analyte List*)
- Include the laboratory SOP for PFAS analysis
- List the minimum method-achievable Reporting Limits for PFAS
    - Reporting Limits should be less than or equal to:
        - Aqueous – 2 ng/L (ppt)
        - Solids – 0.5 µg/kg (ppb)
- Include the laboratory Method Detection Limits for the PFAS compounds to be analyzed
- 
- Include detailed sampling procedures
    - Precautions to be taken
    - Pump and equipment types
    - Decontamination procedures
    - Approved materials only to be used
- Specify that regular ice only will be used for sample shipment
- Specify that equipment blanks should be collected at a minimum frequency of 1 per day per site for each matrix

# Appendix B - Sampling Protocols for PFAS in Soils, Sediments and Solids

## General

The objective of this protocol is to give general guidelines for the collection of soil, sediment and other solid samples for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

## Laboratory Analysis and Containers

Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633.

The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

## Equipment

Acceptable materials for sampling include stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation's Division of Environmental Remediation.

No sampling equipment components or sample containers should come in to contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials including sample bottle cap liners with a PTFE layer.

A list of acceptable equipment is provided below, but other equipment may be considered appropriate based on sampling conditions.

- stainless steel spoon
- stainless steel bowl
- steel hand auger or shovel without any coatings

## Equipment Decontamination

Standard two step decontamination using detergent (Alconox is acceptable) and clean, PFAS-free water will be performed for sampling equipment. All sources of water used for equipment decontamination should be verified in advance to be PFAS-free through laboratory analysis or certification.

## Sampling Techniques

Sampling is often conducted in areas where a vegetative turf has been established. In these cases, a pre-cleaned trowel or shovel should be used to carefully remove the turf so that it may be replaced at the conclusion of sampling. Surface soil samples (e.g. 0 to 6 inches below surface) should then be collected using a pre-cleaned, stainless steel spoon. Shallow subsurface soil samples (e.g. 6 to ~36 inches below surface) may be collected by digging a hole using a pre-cleaned hand auger or shovel. When the desired subsurface depth is reached, a pre-cleaned hand auger or spoon shall be used to obtain the sample.

When the sample is obtained, it should be deposited into a stainless steel bowl for mixing prior to filling the sample containers. The soil should be placed directly into the bowl and mixed thoroughly by rolling the material into the middle until the material is homogenized. At this point the material within the bowl can be placed into the laboratory provided container.

## Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

## Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at 4 ± 2º Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- Request appropriate data deliverable (Category B) and an electronic data deliverable

## Documentation

A soil log or sample log shall document the location of the sample/borehole, depth of the sample, sampling equipment, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate. Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

## Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appropriate rain gear (PVC, polyurethane, or rubber rain gear are acceptable), bug spray, and sunscreen should be used that does not contain PFAS. Well washed cotton coveralls may be used as an alternative to bug spray and/or sunscreen.

PPE that contains PFAS is acceptable when site conditions warrant additional protection for the samplers and no other materials can be used to be protective. Documentation of such use should be provided in the field notes.

# Appendix C - Sampling Protocols for PFAS in Monitoring Wells

## General

The objective of this protocol is to give general guidelines for the collection of groundwater samples for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

## Laboratory Analysis and Container

Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633.

The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

## Equipment

Acceptable materials for sampling include: stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation's Division of Environmental Remediation.

No sampling equipment components or sample containers should come in contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials including plumbers tape and sample bottle cap liners with a PTFE layer.

A list of acceptable equipment is provided below, but other equipment may be considered appropriate based on sampling conditions.

- stainless steel inertia pump with HDPE tubing
- peristaltic pump equipped with HDPE tubing and silicone tubing
- stainless steel bailer with stainless steel ball
- bladder pump (identified as PFAS-free) with HDPE tubing

## Equipment Decontamination

Standard two step decontamination using detergent (Alconox is acceptable) and clean, PFAS-free water will be performed for sampling equipment. All sources of water used for equipment decontamination should be verified in advance to be PFAS-free through laboratory analysis or certification.

## Sampling Techniques

Monitoring wells should be purged in accordance with the sampling procedure (standard/volume purge or low flow purge) identified in the site work plan, which will determine the appropriate time to collect the sample. If sampling using standard purge techniques, additional purging may be needed to reduce turbidity levels, so samples contain a limited amount of sediment within the sample containers. Sample containers that contain sediment may cause issues at the laboratory, which may result in elevated reporting limits and other issues during the sample preparation that can compromise data usability. Sampling personnel should don new nitrile gloves prior to sample collection due to the potential to contact PFAS containing items (not related to the sampling equipment) during the purging activities.

## Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

## Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at 4 ± 2º Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- Collect one equipment blank per day per site and minimum 1 equipment blank per 20 samples. The equipment blank shall test the new and decontaminated sampling equipment utilized to obtain a sample for residual PFAS contamination. This sample is obtained by using laboratory provided PFAS-free water and passing the water over or through the sampling device and into laboratory provided sample containers
- Additional equipment blank samples may be collected to assess other equipment that is utilized at the monitoring well
- Request appropriate data deliverable (Category B) and an electronic data deliverable

## Documentation

A purge log shall document the location of the sample, sampling equipment, groundwater parameters, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate. Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

## Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appropriate rain gear (PVC, polyurethane, or rubber rain gear are acceptable), bug spray, and sunscreen should be used that does not contain PFAS. Well washed cotton coveralls may be used as an alternative to bug spray and/or sunscreen.

PPE that contains PFAS is acceptable when site conditions warrant additional protection for the samplers and no other materials can be used to be protective. Documentation of such use should be provided in the field notes.

# Appendix D - Sampling Protocols for PFAS in Surface Water

## General

The objective of this protocol is to give general guidelines for the collection of surface water samples for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

## Laboratory Analysis and Container

Samples collected using this protocol are intended to be analyzed for PFAS using EPA Method 1633.

The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

## Equipment

Acceptable materials for sampling include: stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation's Division of Environmental Remediation.

No sampling equipment components or sample containers should come in contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials including sample bottle cap liners with a PTFE layer.

A list of acceptable equipment is provided below, but other equipment may be considered appropriate based on sampling conditions.

- stainless steel cup

## Equipment Decontamination

Standard two step decontamination using detergent (Alconox is acceptable) and clean, PFAS-free water will be performed for sampling equipment. All sources of water used for equipment decontamination should be verified in advance to be PFAS-free through laboratory analysis or certification.

## Sampling Techniques

Where conditions permit, (e.g. creek or pond) sampling devices (e.g. stainless steel cup) should be rinsed with site medium to be sampled prior to collection of the sample. At this point the sample can be collected and poured into the sample container.

If site conditions permit, samples can be collected directly into the laboratory container.

## Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

## Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at 4 ± 2º Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- Collect one equipment blank per day per site and minimum 1 equipment blank per 20 samples. The equipment blank shall test the new and decontaminated sampling equipment utilized to obtain a sample for residual PFAS contamination. This sample is obtained by using laboratory provided PFAS-free water and passing the water over or through the sampling device and into laboratory provided sample containers
- Request appropriate data deliverable (Category B) and an electronic data deliverable

## Documentation

A sample log shall document the location of the sample, sampling equipment, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate. Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

## Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

Appropriate rain gear (PVC, polyurethane, or rubber rain gear are acceptable), bug spray, and sunscreen should be used that does not contain PFAS. Well washed cotton coveralls may be used as an alternative to bug spray and/or sunscreen.

PPE that contains PFAS is acceptable when site conditions warrant additional protection for the samplers and no other materials can be used to be protective. Documentation of such use should be provided in the field notes.

# Appendix E - Sampling Protocols for PFAS in Private Water Supply Wells

## General

The objective of this protocol is to give general guidelines for the collection of water samples from private water supply wells (with a functioning pump) for PFAS analysis. The sampling procedure used should be consistent with Sampling Guidelines and Protocols – Technological Background and Quality Control/Quality Assurance for NYS DEC Spill Response Program – March 1991 (http://www.dec.ny.gov/docs/remediation_hudson_pdf/sgpsect5.pdf), with the following limitations.

## Laboratory Analysis and Container

Drinking water samples collected using this protocol are intended to be analyzed for PFAS by EPA Method 537, 537.1, 533, or ISO Method 25101. The preferred material for containers is high density polyethylene (HDPE). Pre-cleaned sample containers, coolers, sample labels, and a chain of custody form will be provided by the laboratory.

## Equipment

Acceptable materials for sampling include stainless steel, HDPE, PVC, silicone, acetate, and polypropylene. Additional materials may be acceptable if pre-approved by New York State Department of Environmental Conservation's Division of Environmental Remediation.

No sampling equipment components or sample containers should come in contact with aluminum foil, low density polyethylene, glass, or polytetrafluoroethylene (PTFE, Teflon™) materials (e.g. plumbers tape), including sample bottle cap liners with a PTFE layer.

## Equipment Decontamination

Standard two step decontamination using detergent (Alconox is acceptable) and clean, PFAS-free water will be performed for sampling equipment. All sources of water used for equipment decontamination should be verified in advance to be PFAS-free through laboratory analysis or certification.

## Sampling Techniques

Locate and assess the pressure tank and determine if any filter units are present within the building. Establish the sample location as close to the well pump as possible, which is typically the spigot at the pressure tank. Ensure sampling equipment is kept clean during sampling as access to the pressure tank spigot, which is likely located close to the ground, may be obstructed and may hinder sample collection.

Prior to sampling, a faucet downstream of the pressure tank (e.g., washroom sink) should be run until the well pump comes on and a decrease in water temperature is noted which indicates that the water is coming from the well. If the homeowner is amenable, staff should run the water longer to purge the well (15+ minutes) to provide a sample representative of the water in the formation rather than standing water in the well and piping system including the pressure tank. At this point a new pair of nitrile gloves should be donned and the sample can be collected from the sample point at the pressure tank.

## Sample Identification and Logging

A label shall be attached to each sample container with a unique identification. Each sample shall be included on the chain of custody (COC).

## Quality Assurance/Quality Control

- Immediately place samples in a cooler maintained at 4 ± 2º Celsius using ice
- Collect one field duplicate for every sample batch, minimum 1 duplicate per 20 samples. The duplicate shall consist of an additional sample at a given location
- Collect one matrix spike / matrix spike duplicate (MS/MSD) for every sample batch, minimum 1 MS/MSD per 20 samples. The MS/MSD shall consist of an additional two samples at a given location and identified on the COC
- If equipment was used, collect one equipment blank per day per site and a minimum 1 equipment blank per 20 samples. The equipment blank shall test the new and decontaminated sampling equipment utilized to obtain a sample for residual PFAS contamination. This sample is obtained by using laboratory provided PFAS-free water and passing the water over or through the sampling device and into laboratory provided sample containers.
- A field reagent blank (FRB) should be collected at a rate of one per 20 samples. The lab will provide a FRB bottle containing PFAS free water and one empty FRB bottle. In the field, pour the water from the one bottle into the empty FRB bottle and label appropriately.
- Request appropriate data deliverable (Category B) and an electronic data deliverable
- For sampling events where multiple private wells (homes or sites) are to be sampled per day, it is acceptable to collect QC samples at a rate of one per 20 across multiple sites or days.

## Documentation

A sample log shall document the location of the private well, sample point location, owner contact information, sampling equipment, purge duration, duplicate sample, visual description of the material, and any other observations or notes determined to be appropriate and available (e.g. well construction, pump type and location, yield, installation date). Additionally, care should be performed to limit contact with PFAS containing materials (e.g. waterproof field books, food packaging) during the sampling process.

## Personal Protection Equipment (PPE)

For most sampling Level D PPE is anticipated to be appropriate. The sampler should wear nitrile gloves while conducting field work and handling sample containers.

Field staff shall consider the clothing to be worn during sampling activities. Clothing that contains PTFE material (including GORE-TEX®) or that have been waterproofed with PFAS materials should be avoided. All clothing worn by sampling personnel should have been laundered multiple times.

# Appendix F - Sampling Protocols for PFAS in Fish

This appendix contains a copy of the current SOP developed by the Division of Fish and Wildlife (DFW) entitled "General Fish Handling Procedures for Contaminant Analysis" (Ver. 8). This SOP should be followed when collecting fish for contaminant analysis. Note, however, that the Bureau of Ecosystem Health will not be supplying bags or tags. All supplies are the responsibility of the collector

**Procedure Name:** General Fish Handling Procedures for Contaminant Analysis

**Number:** FW-005

**Purpose:** This procedure describes data collection, fish processing and delivery of fish collected for contaminant monitoring. It contains the chain of custody and collection record forms that should be used for the collections.

**Organization:** Environmental Monitoring Section
Bureau of Ecosystem Health
Division of Fish and Wildlife (DFW)
New York State Department of Environmental Conservation (NYSDEC)
625 Broadway
Albany, New York 12233-4756

**Version:** 8

**Previous Version Date:** 21 March 2018

**Summary of Changes to this Version:** Updated bureau name to Bureau of Ecosystem Health. Added direction to list the names of all field crew on the collection record. Minor formatting changes on chain of custody and collection records.

**Originator or Revised by:** Wayne Richter, Jesse Becker

**Date:** 26 April 2019

**Quality Assurance Officer and Approval Date:** Jesse Becker, 26 April 2019

**NEW YORK STATE**
**DEPARTMENT OF ENVIRONMENTAL CONSERVATION**

# GENERAL FISH HANDLING PROCEDURES FOR CONTAMINANT ANALYSES

A. Original copies of all continuity of evidence (i.e., Chain of Custody) and collection record forms must accompany delivery of fish to the lab. A copy shall be directed to the Project Leader or as appropriate, Wayne Richter. All necessary forms will be supplied by the Bureau of Ecosystem Health. Because some samples may be used in legal cases, it is critical that each section is filled out completely. Each Chain of Custody form has three main sections:

   1. The top box is to be filled out **and signed** by the person responsible for the fish collection (e.g., crew leader, field biologist, researcher). This person is responsible for delivery of the samples to DEC facilities or personnel (e.g., regional office or biologist).

   2. The second section is to be filled out **and signed** by the person responsible for the collections while being stored at DEC, before delivery to the analytical lab. This may be the same person as in (1), but it is still required that they complete the section. Also important is the **range of identification numbers** (i.e., tag numbers) included in the sample batch.

   3. Finally, the bottom box is to record any transfers between DEC personnel and facilities. Each subsequent transfer should be **identified, signed, and dated**, until laboratory personnel take possession of the fish.

B. The following data are required on each **Fish Collection Record** form:

   1. Project and Site Name.

   2. DEC Region.

   3. All personnel (and affiliation) involved in the collection.

   4. Method of collection (gill net, hook and line, etc.)

   5. Preservation Method.

C. The following data are to be taken on each fish collected and recorded on the **Fish Collection Record** form:

   1. Tag number - Each specimen is to be individually jaw tagged at time of collection with a unique number. Make sure the tag is turned out so that the number can be read without opening the bag. Use tags in sequential order. For small fish or composite samples place the tag inside the bag with the samples. The Bureau of Ecosystem Health can supply the tags.

   2. Species identification (please be explicit enough to enable assigning genus and species). Group fish by species when processing.

   3. Date collected.

   4. Sample location (waterway and nearest prominent identifiable landmark).

   5. Total length (nearest mm or smallest sub-unit on measuring instrument) and weight (nearest g or

smallest sub-unit of weight on weighing instrument). Take all measures as soon as possible with calibrated, protected instruments (e.g. from wind and upsets) and prior to freezing.

6. Sex - fish may be cut enough to allow sexing or other internal investigation, but do not eviscerate. Make any incision on the right side of the belly flap or exactly down the midline so that a left-side fillet can be removed.

D. General data collection recommendations:

1. It is helpful to use an ID or tag number that will be unique. It is best to use metal striped bass or other uniquely numbered metal tags. If uniquely numbered tags are unavailable, values based on the region, water body and year are likely to be unique: for example, R7CAY11001 for Region 7, Cayuga Lake, 2011, fish 1. If the fish are just numbered 1 through 20, we have to give them new numbers for our database, making it more difficult to trace your fish to their analytical results and creating an additional possibility for errors.

2. Process and record fish of the same species sequentially. Recording mistakes are less likely when all fish from a species are processed together. Starting with the bigger fish species helps avoid missing an individual.

3. If using Bureau of Ecosystem Health supplied tags or other numbered tags, use tags in sequence so that fish are recorded with sequential Tag Numbers. This makes data entry and login at the lab and use of the data in the future easier and reduces keypunch errors.

4. Record length and weight as soon as possible after collection and before freezing. Other data are recorded in the field upon collection. An age determination of each fish is optional, but if done, it is recorded in the appropriate “Age” column.

5. For composite samples of small fish, record the number of fish in the composite in the Remarks column. Record the length and weight of each individual in a composite. All fish in a composite sample should be of the same species and members of a composite should be visually matched for size.

6. Please submit photocopies of topographic maps or good quality navigation charts indicating sampling locations. GPS coordinates can be entered in the Location column of the collection record form in addition to or instead for providing a map. These records are of immense help to us (and hopefully you) in providing documented location records which are not dependent on memory and/or the same collection crew. In addition, they may be helpful for contaminant source trackdown and remediation/control efforts of the Department.

7. When recording data on fish measurements, it will help to ensure correct data recording for the data recorder to call back the numbers to the person making the measurements.

E. Each fish is to be placed in its own individual plastic bag. For small fish to be analyzed as a composite, put all of the fish for one composite in the same bag but use a separate bag for each composite. It is important to individually bag the fish to avoid difficulties or cross contamination when processing the fish for chemical analysis. Be sure to include the fish’s tag number inside the bag, preferably attached to the fish with the tag number turned out so it can be read. Tie or otherwise secure the bag closed. **The Bureau of Ecosystem Health will supply the bags.** If necessary, food grade bags may be procured from a suitable vendor (e.g., grocery store). It is preferable to redundantly label each bag with a manila tag tied between the knot and the body of the bag. This tag should be labeled with the project name, collection location, tag number, collection date, and fish species. If scales are collected, the scale envelope should be labeled with

the same information.

F. Groups of fish, by species, are to be placed in one large plastic bag per sampling location. **The Bureau of Ecosystem Health will supply the larger bags.** Tie or otherwise secure the bag closed. Label the site bag with a manila tag tied between the knot and the body of the bag. The tag should contain: project, collection location, collection date, species and **tag number ranges**. Having this information on the manila tag enables lab staff to know what is in the bag without opening it.

G. Do not eviscerate, fillet or otherwise dissect the fish unless specifically asked to. If evisceration or dissection is specified, the fish must be cut along the exact midline or on the right side so that the left side fillet can be removed intact at the laboratory. If filleting is specified, the procedure for taking a standard fillet (SOP PREPLAB 4) must be followed, including removing scales.

H. Special procedures for PFAS: Unlike legacy contaminants such as PCBs, which are rarely found in day to day life, PFAS are widely used and frequently encountered. Practices that avoid sample contamination are therefore necessary. While no standard practices have been established for fish, procedures for water quality sampling can provide guidance. The following practices should be used for collections when fish are to be analyzed for PFAS:

No materials containing Teflon.
No Post-it notes.
No ice packs; only water ice or dry ice.
Any gloves worn must be powder free nitrile.
No Gore-Tex or similar materials (Gore-Tex is a PFC with PFOA used in its manufacture).
No stain repellent or waterproof treated clothing; these are likely to contain PFCs.
Avoid plastic materials, other than HDPE, including clipboards and waterproof notebooks.
Wash hands after handling any food containers or packages as these may contain PFCs.
Keep pre-wrapped food containers and wrappers isolated from fish handling.
Wear clothing washed at least six times since purchase.
Wear clothing washed without fabric softener.
Staff should avoid cosmetics, moisturizers, hand creams and similar products on the day of sampling as many of these products contain PFCs (Fujii et al. 2013). Sunscreen or insect repellent should not contain ingredients with “fluor” in their name. Apply any sunscreen or insect repellent well downwind from all materials. Hands must be washed after touching any of these products.

I. All fish must be kept at a temperature <45° F (<8° C) immediately following data processing. As soon as possible, freeze at -20° C ± 5° C. Due to occasional freezer failures, daily freezer temperature logs are required. The freezer should be locked or otherwise secured to maintain chain of custody.

J. In most cases, samples should be delivered to the Analytical Services Unit at the Hale Creek field station. Coordinate delivery with field station staff and send copies of the collection records, continuity of evidence forms and freezer temperature logs to the field station. For samples to be analyzed elsewhere, non-routine collections or other questions, contact Wayne Richter, Bureau of Ecosystem Health, NYSDEC, 625 Broadway, Albany, New York 12233-4756, 518-402-8974, or the project leader about sample transfer. Samples will then be directed to the analytical facility and personnel noted on specific project descriptions.

K. A recommended equipment list is at the end of this document.

**NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION**
**DIVISION OF FISH AND WILDLIFE**
**FISH COLLECTION RECORD**



Project and Site Name _____ DEC Region _____

Collections made by (include all crew) _____

Sampling Method: □Electrofishing □Gill netting □Trap netting □Trawling □Seining □Angling □Other _____

Preservation Method: □Freezing □Other _____ Notes (SWFDB survey number): _____

| FOR LAB USE ONLY- LAB ENTRY NO. | COLLECTION OR TAG NO. | SPECIES | DATE TAKEN | LOCATION | AGE | SEX &/OR REPROD. CONDIT | LENGTH ( ) | WEIGHT ( ) | REMARKS |
|---|---|---|---|---|---|---|---|---|---|
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richter: revised 2011, 5/7/15, 10/4/16, 3/20/17; becker: 3/23/17, 4/26/19

# NEW YORK STATE DEPARTMENT OF ENVIRONMENTAL CONSERVATION
# CHAIN OF CUSTODY

I, _____, of _____ collected the
(Print Name) (Print Business Address)

following on _____, 20____ from _____
(Date) (Water Body)

in the vicinity of _____
(Landmark, Village, Road, etc.)

Town of _____, in _____ County.

Item(s) _____

_____

Said sample(s) were in my possession and handled according to standard procedures provided to me prior to collection. The sample(s) were placed in the custody of a representative of the New York State Department of Environmental Conservation on _____, 20_____.

_____ _____
Signature Date

I, _____, received the above mentioned sample(s) on the date specified and assigned identification number(s) _____ to the sample(s). I have recorded pertinent data for the sample(s) on the attached collection records. The sample(s) remained in my custody until subsequently transferred, prepared or shipped at times and on dates as attested to below.

_____ _____
Signature Date

| | | |
|---|---|---|
| SECOND RECIPIENT (Print Name) | TIME & DATE | PURPOSE OF TRANSFER |
| SIGNATURE | UNIT | |
| THIRD RECIPIENT (Print Name) | TIME & DATE | PURPOSE OF TRANSFER |
| SIGNATURE | UNIT | |
| FOURTH RECIPIENT (Print Name) | TIME & DATE | PURPOSE OF TRANSFER |
| SIGNATURE | UNIT | |
| RECEIVED IN LABORATORY BY (Print Name) | TIME & DATE | REMARKS |
| SIGNATURE | UNIT | |
| LOGGED IN BY (Print Name) | TIME & DATE | ACCESSION NUMBERS |
| SIGNATURE | UNIT | |

richter: revised 21 April 2014; becker: 23 March 2017, 26 April, 2019

## NOTICE OF WARRANTY

By signature to the chain of custody (reverse), the signatory warrants that the information provided is truthful and accurate to the best of his/her ability. The signatory affirms that he/she is willing to testify to those facts provided and the circumstances surrounding the same. Nothing in this warranty or chain of custody negates responsibility nor liability of the signatories for the truthfulness and accuracy of the statements provided.

## HANDLING INSTRUCTIONS

On day of collection, collector(s) name(s), address(es), date, geographic location of capture (attach a copy of topographic map or navigation chart), species, number kept of each species, and description of capture vicinity (proper noun, if possible) along with name of Town and County must be indicated on reverse.

Retain organisms in manila tagged plastic bags to avoid mixing capture locations. Note appropriate information on each bag tag.

Keep samples as cool as possible. Put on ice if fish cannot be frozen within 12 hours. If fish are held more than 24 hours without freezing, they will not be retained or analyzed.

Initial recipient (either DEC or designated agent) of samples from collector(s) is responsible for obtaining and recording information on the collection record forms which will accompany the chain of custody. This person will seal the container using packing tape and writing his signature, the time and the date across the tape onto the container with indelible marker. Any time a seal is broken, for whatever purpose, the incident must be recorded on the Chain of Custody (reason, time, and date) in the purpose of transfer block. Container then is resealed using new tape and rewriting signature, with time and date.

EQUIPMENT LIST

Scale or balance of appropriate capacity for the fish to be collected.

Fish measuring board.

Plastic bags of an appropriate size for the fish to be collected and for site bags.

Individually numbered metal tags for fish.

Manila tags to label bags.

Small envelops, approximately 2” x 3.5”, if fish scales are to be collected.

Knife for removing scales.

Chain of custody and fish collection forms.

Clipboard.

Pens or markers.

Paper towels.

Dish soap and brush.

Bucket.

Cooler.

Ice.

Duct tape.

# Appendix G – PFAS Analyte List

| Group | Chemical Name | Abbreviation | CAS Number |
|---|---|---|---|
| Perfluoroalkyl sulfonic acids | 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 |
| Perfluoroalkyl carboxylic acids | 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 |
| Per- and Polyfluoroether carboxylic acids | Hexafluoropropylene oxide dimer 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 |
| Fluorotelomer sulfonic acids | 4:2 Fluorotelomer sulfonic acid | 4:2-FTS | 757124-72-4 |
| | 6:2 Fluorotelomer sulfonic acid | 6:2-FTS | 27619-97-2 |
| | 8:2 Fluorotelomer sulfonic acid | 8:2-FTS | 39108-34-4 |
| Fluorotelomer carboxylic acids | 3:3 Fluorotelomer carboxylic acid | 3:3 FTCA | 356-02-5 |
| | 5:3 Fluorotelomer carboxylic acid | 5:3 FTCA | 914637-49-3 |
| | 7:3 Fluorotelomer carboxylic acid | 7:3 FTCA | 812-70-4 |
| Perfluorooctane sulfonamides | Perfluorooctane sulfonamide | PFOSA | 754-91-6 |
| | N-methylperfluorooctane sulfonamide | NMeFOSA | 31506-32-8 |
| | N-ethylperfluorooctane sulfonamide | NEtFOSA | 4151-50-2 |
| Perfluorooctane sulfonamidoacetic acids | N-methylperfluorooctane sulfonamidoacetic acid | N-MeFOSAA | 2355-31-9 |
| | N-ethylperfluorooctane sulfonamidoacetic acid | N-EtFOSAA | 2991-50-6 |
| Perfluorooctane sulfonamide ethanols | N-methylperfluorooctane sulfonamidoethanol | MeFOSE | 24448-09-7 |
| | N-ethylperfluorooctane sulfonamidoethanol | EtFOSE | 1691-99-2 |

| Group | Chemical Name | Abbreviation | CAS Number |
|---|---|---|---|
| Ether sulfonic acids | 9-Chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (F-53B Major) | 9Cl-PF3ONS | 756426-58-1 |
| | 11-Chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (F-53B Minor) | 11Cl-PF3OUdS | 763051-92-9 |
| | Perfluoro(2-ethoxyethane) sulfonic acid | PFEESA | 113507-82-7 |

# Appendix H - Data Review Guidelines for Analysis of PFAS in Non-Potable Water and Solids

## General

These guidelines are intended to be used for the validation of PFAS using EPA Method 1633 for projects within the Division of Environmental Remediation (DER). Data reviewers should understand the methodology and techniques utilized in the analysis. Consultation with the end user of the data may be necessary to assist in determining data usability based on the data quality objectives in the Quality Assurance Project Plan. A familiarity with the laboratory's Standard Operating Procedure may also be needed to fully evaluate the data. If you have any questions, please contact DER's Quality Assurance Officer, Dana Barbarossa, at dana.barbarossa@dec.ny.gov.

## Preservation and Holding Time

Samples should be preserved with ice to a temperature of less than 6°C upon arrival at the lab. The holding time is 28 days to extraction for aqueous and solid samples. The time from extraction to analysis for aqueous samples is 28 days and 40 days for solids.

| | |
|---|---|
| Temperature greatly exceeds 6ºC upon arrival at the lab* | Use professional judgement to qualify detects and non-detects as estimated or rejected |
| Holding time exceeding 28 days to extraction | Use professional judgement to qualify detects and non-detects as estimated or rejected if holding time is grossly exceeded |

*Samples that are delivered to the lab immediately after sampling may not meet the thermal preservation guidelines. Samples are considered acceptable if they arrive on ice or an attempt to chill the samples is observed.

## Initial Calibration

The initial calibration should contain a minimum of six standards for linear fit and six standards for a quadratic fit. The relative standard deviation (RSD) for a quadratic fit calibration should be less than 20%.

The low-level calibration standard should be within 50% - 150% of the true value, and the mid-level calibration standard within 70% - 130% of the true value.

| | |
|---|---|
| %RSD >20% | J flag detects and UJ non detects |

## Continuing Calibration Verification

Continuing calibration verification (CCV) checks should be analyzed at a frequency of one per ten field samples. If CCV recovery is very low, where detection of the analyte could be in question, ensure a low level CCV was analyzed and use to determine data quality.

| | |
|---|---|
| CCV recovery <70 or >130% | J flag results |

## Blanks

There should be no detections in the method blanks above the reporting limits. Equipment blanks, field blanks, rinse blanks etc. should be evaluated in the same manner as method blanks. Use the most contaminated blank to evaluate the sample results.

| Blank Result | Sample Result | Qualification |
|---|---|---|
| Any detection | <Reporting limit | Qualify as ND at reporting limit |
| Any detection | >Reporting Limit and >10x the blank result | No qualification |
| >Reporting limit | >Reporting limit and <10x blank result | J+ biased high |

## Field Duplicates

A blind field duplicate should be collected at rate of one per twenty samples. The relative percent difference (RPD) should be less than 30% for analyte concentrations greater than two times the reporting limit. Use the higher result for final reporting.

| | |
|---|---|
| RPD >30% | Apply J qualifier to parent sample |

## Lab Control Spike

Lab control spikes should be analyzed with each extraction batch or one for every twenty samples. In the absence of lab derived criteria, use 70% - 130% recovery criteria to evaluate the data.

| | |
|---|---|
| Recovery <70% or >130% (lab derived criteria can also be used) | Apply J qualifier to detects and UJ qualifier to non detects |

## Matrix Spike/Matrix Spike Duplicate

One matrix spike and matrix spike duplicate should be collected at a rate of one per twenty samples. Use professional judgement to reject results based on out of control MS/MSD recoveries.

| | |
|---|---|
| Recovery <70% or >130% (lab derived criteria can also be used) | Apply J qualifier to detects and UJ qualifier to non detects of parent sample only |
| RPD >30% | Apply J qualifier to detects and UJ qualifier to non detects of parent sample only |

## Extracted Internal Standards (Isotope Dilution Analytes)

Problematic analytes (e.g. PFBA, PFPeA, fluorotelomer sulfonates) can have wider recoveries without qualification. Qualify corresponding native compounds with a J flag if outside of the range.

| | |
|---|---|
| Recovery <50% or >150% | Apply J qualifier |
| Recovery <25% or >150% for poor responding analytes | Apply J qualifier |
| Isotope Dilution Analyte (IDA) Recovery <10% | Reject results |

## Signal to Noise Ratio

The signal to noise ratio for the quantifier ion should be at least 3:1. If the ratio is less than 3:1, the peak is discernable from the baseline noise and symmetrical, the result can be reported. If the peak appears to be baseline noise and/or the shape is irregular, qualify the result as tentatively identified.

## Reporting Limits

If project-specific reporting limits were not met, please indicate that in the report along with the reason (e.g. over dilution, dilution for non-target analytes, high sediment in aqueous samples).

## Peak Integrations

Target analyte peaks should be integrated properly and consistently when compared to standards. Ensure branched isomer peaks are included for PFAS where standards are available. Inconsistencies should be brought to the attention of the laboratory or identified in the data review summary report.

# APPENDIX C

Site Specific Health and Safety Plan

# Site-Specific Health and Safety Plan (HASP)

2030 Fulton Street
Brooklyn, New York

September 30, 2026

Prepared for:

**Steps Family LLC**
**c/o Riseboro Community Partnership**
217 Wyckoff Avenue
Brooklyn, New York 11237

Prepared by:

**Roux Environmental Engineering and Geology, D.P.C.**
209 Shafter Street
Islandia, New York 11749

3805.0002Y115/CV

Environmental Consulting
& Management
+1.800.322.ROUX
rouxinc.com

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# Site-Specific Emergency Information

## Emergency Phone Numbers

Most emergency services can be obtained by calling 911. Where 911 service is not available, use the telephone numbers provided in the below table. The following is a master emergency phone list for use by the project management personnel. A more condensed version of the emergency numbers listed below will be posted throughout project work areas. Emergencies encountered on the site will be responded to by a combination of off-site emergency services and on-site personnel.

| **Emergency Contact Information** | | | |
|---|---|---|---|
| **Site Personnel** | | | |
| **Title** | **Contact** | | **Telephone** |
| Operations Manager (OM) | Jeffrey Wills | | 516-637-0213 |
| Project Principal (PP) | Jessica Taylor | | 631-766-0607 |
| Project Manager (PM) | Brooke Hildebrand | | 613-356-3719 |
| Site Supervisor (SS) | Brooke Hildebrand | | 631-356-3719 |
| Site Health and Site Safety Officer (SHSO) | TBD | | TBD |
| Office Health and Safety Manager (OHSM) | Nevin Pahlad, CSP, CHMM | | 347-885-6930 |
| Corporate Health and Safety Director (CHSD) | Brian Hobbs, CIH, CSP | | 631-807-0193 |
| Occupational Health Care Management Provider | WorkCare, Inc. | | 888-449-7787 |
| Client Emergency Contact | Stephan Petryczka | | 718-366-3800 |
| **Outside Assistance** | | | |
| **Agency** | **Contact** | **Telephone** | **Address/Location** |
| Ambulance/emergency medical services (EMS) | Kings County Hospital Center | 718-245-3131 | 451 Clarkson Ave Brooklyn, NY 11203 |
| Police | New York City Police Department - 73rd Precinct | 718-495-5411 | 1470 E New York Ave Brooklyn, NY 11212 |
| Fire | FDNY Engine 227 | 718-965-8227 | 423 Ralph Ave Brooklyn, NY 11233 |
| Site Address | 2030 Fulton Street, Brooklyn, NY | | |

See **Figure 3** for Routes to Urgent Care and Hospital. Directions are described below.

**CityMD Crown Heights Urgent Care- Brooklyn:**

256 Utica Ave, Brooklyn, NY 11213

- Head west on Fulton St toward Hull St
- Turn left onto Utica Ave
- Destination will be on the right

**Kings County Hospital Center:**

451 Clarkson Ave, Brooklyn, NY 11203

- Head west on Fulton St toward Hull St
- Turn left onto Utica Ave
- Head south on Utica Ave toward Lincoln Pl
- Turn right onto Eastern Pkwy
- Turn left onto Dr Wesley MacDonald Holder Ave/ Schenectady Ave
- Turn right onto Winthrop St
- Turn left onto Albany Ave
- Turn right onto Clarkson Ave
- Destination will be on the right

# 1. Introduction

This Site-specific Health and Safety Plan (HASP) has been prepared by Roux Environmental Engineering and Geology, D.P.C. (Roux) for use during the Remedial Investigation (RI) activities being performed by Roux at the Family Building project (Steps Family), located at 2030 Fulton Street in the Bedford Stuyvesant neighborhood of Brooklyn (Site; **Figure 1**). This HASP was prepared in general accordance with the requirements of the Occupational Safety and Health Administration (OSHA) standards promulgated at 29 CFR 1910.120 and 29 CFR 1926.65, both commonly referred to as the Hazardous Waste Operations and Emergency Response (HAZWOPER) Standard. In accordance with the HAZWOPER Standard, this Site-specific HASP was prepared to address the safety and health hazards associated with the RI activities being performed at the Site by Roux and to provide requirements and procedures for the protection of Roux employees, subcontractor personnel, government oversight personnel, Site personnel, and the general public. It also addresses client- and Site-specific requirements for health and safety. Additionally, subcontractors may be required to submit their own HASP as it relates to their specific work activities and will be kept onsite during such work.

Implementation of this HASP is the joint responsibility of the Project Manager (PM), the Site Health and Safety Officer (SHSO), and all field staff, with assistance from the Project Principal (PP), Office Health and Safety Manager (OHSM), and Corporate Health and Safety Director (CHSD). The PM for this project is Brooke Hildebrand. The Site Supervisor (SS) is Brooke Hildebrand and Site Health and Safety Officer (SHSO) is TBD.

This HASP will be introduced to, reviewed, and signed off on by all Roux personnel through a formal training session prior to commencing work. A copy of the HASP will be kept at the Site at all times. The Roux SHSO or PM will be responsible for posting any changes, amendments, memos, etc. to the HASP. Any revisions to this HASP will be signed by appropriate personnel, which can include Roux's PP, CHSD, and SS. Any changes will be announced to all workers at the next safety meeting.

## 1.1 Roles and Responsibilities

Overall Roles and Responsibilities (R&Rs) of Roux personnel are provided in Roux's Policies and Procedures Manual. Only those R&Rs specific to HASP requirements are listed below.

**Project Manager (PM)**

The PM has responsibility and authority to direct all work operations. The PM coordinates safety and health functions with the Site Health and Safety Officer (SHSO), has the authority to oversee and monitor the performance of the SHSO, and bears ultimate responsibility for the proper implementation of this HASP. The specific duties of the PM are:

- Preparing and coordinating the Site work plan;
- Providing Site supervisor(s) with work assignments and overseeing their performance; Coordinating safety and health efforts with the SSHO;
- Ensuring effective emergency response through coordination with the Emergency Response Coordinator (ERC); and
- Serving as primary Site liaison with public agencies and officials and Site contractors.

**Site Health and Safety Officer (SHSO)**

The SHSO has the full responsibility and authority to develop and implement this HASP and to verify compliance. The SHSO reports to the Project Manager. The SHSO is on Site or readily accessible to the Site during all work operations and has the authority to halt Site work if unsafe conditions are detected. The specific responsibilities of the SHSO include:

- Managing the safety and health functions on this Site;
- Serving as the Site's point of contact for safety and health matters;
- Ensuring Site monitoring, worker training, and effective selection and use of PPE;
- Assessing Site conditions for unsafe acts and conditions and providing corrective action;
- Assisting the preparation and review of this HASP;
- Maintaining effective safety and health records as described in this HASP; and
- Coordinating with the SS and others as necessary for safety and health efforts.

**Site Supervisor (SS)**

The Site Supervisor (SS) is responsible for field operations and reports to the Project Manager (PM). The SS ensures the implementation of the HASP requirements and procedures in the field. The specific responsibilities of the Site Supervisor include:

- Executing the work plan and schedule as detailed by the PM;
- Coordination with the SHSO on safety and health; and
- Ensuring Site work compliance with the requirements of this HASP.

**Employees**

All Roux employees are responsible for reading and following all provisions of the Corporate Health and Safety Manual, including this HASP. Employees report to the SS at the project Site. Each employee is also responsible for the following:

- Wearing all appropriate PPE as outlined within this HASP;
- Attending all safety meetings;
- Inspecting tools and equipment prior to use, and taking any defective tools or equipment out of service;
- Appropriately documenting field events as they occur within a logbook or equivalent;
- Properly operating machinery and/or equipment only if trained to do so;
- Stopping work operations if unsafe conditions exist;
- Identifying and mitigating hazards when observed;
- Reporting all incidents and near misses to the Roux SHSO and SS immediately; and
- Knowing where emergency equipment is located (e.g., first aid kit, fire extinguisher).

**Subcontractors and Visitors**

Subcontractors and visitors are responsible for complying with the same health and safety requirements. It is the responsibility of all to make sure subcontractors and visitors comply and uphold the HASP. Subcontractors and visitors have the following additional responsibilities:

- Designating a qualified safety representative for the project that can make the necessary changes in work practices, as necessary;
- Attending all safety meetings while participating in Roux Site work activities;
- Reporting all incidents and near misses to Roux SHSO and SS immediately;
- Conducting initial and periodic equipment inspections in accordance with manufacturer and regulatory guidelines; and
- Providing copies of all Safety Data Sheets (SDS) to Roux SHSO for materials brought to the Site.

# 2. Background

Relevant background information is provided below, including a general description of the Site; a brief review of the Site's history with respect to hazardous material use, handling, and/or storage; and a review of known and potential releases of hazardous substances at the Site.

## 2.1 Site Description

The Site is located in the Bedford Stuyvesant neighborhood of Brooklyn, New York. The Subject Property was comprised of twelve contiguous parcels, consisting of Block 1549 Lots 23, 24, 25, 26, 28, 29, 30, 31, 32, 33, 34, and 35 on the New York City (NYC) Tax Map but has since been merged into a single tax lot, Lot 28, which is referenced throughout this document. The Address associated with the subject property was identified as the following as per the NYC Department of Buildings: 2030 Fulton Street, Brooklyn, New York. The proposed development consists of an affordable residential housing building for families with 12 floors, a rooftop and a cellar. The Site represents a total area of approximately 0.587 acres.

## 2.2 Site History

Based on review of Roux's Phase I ESA, dated October 25, 2023, the Site was first developed prior to 1908 with mixed use buildings on all lots except for 224 Saratoga Avenue, which was developed with a dwelling. Listings for the property consisted of commercial and manufacturing operations including a machine shop, noodle manufacturing and packing facility, upholstery facility, blind manufacturer, tin smith, sheet metal and roofing company. According to a Sanborn Map dated 2001 the Site was depicted as completely vacant, which was confirmed in 2023 during reconnaissance.

## 2.3 Known and Potential Releases of Hazardous Substances at the Site

There is known historic fill at the site, which could result in metals, semivolatile organic compounds (SVOCs), and Polycyclic aromatic hydrocarbons (PAHs) contamination in soil. There are no known releases of hazardous substances at the Site.

# 3. Scope of Work

The objective of the investigation is to collect environmental sampling data to investigate potential contamination onsite. The scope of field investigation is as described below. A total of 28 boring locations will be advanced.

## 3.1 Soil Investigation

To further characterize the soil conditions at the Site, 10 borings (RXS-01 through RXS-10) will be advanced at the locations shown in Figure 5 of the Remedial Investigation Work Plan (RIWP). Four soil borings (RXS-01 through RXS-04) will be advanced ten feet below observed groundwater at the locations shown in Figure 5 of the RIWP for the installation of co-located groundwater monitoring wells as described below. It is anticipated groundwater will be encountered approximately 75 to 80 feet below land surface (ft bls). Six additional soil borings (RXS-05 through RXS-10) will be advanced to two feet below the observed fill layer at the locations shown in Figure 5 of the RIWP for the installation of co-located soil vapor points as described below. At each borehole location utility clearances will be performed prior to advancement of soil borings using hand tools and/or vacuum excavation to a minimum of five ft bls to confirm that no subsurface utilities are present. Should a utility or other feature be observed during pre-clearance activities, the sampling location will be relocated. Soil samples will be collected by utilizing a Geoprobe direct-push drill rig, or roto-sonic drill rig, if required. Soil samples will be collected continuously from grade to the terminal depth of each soil boring. During soil boring advancement, lithology will be recorded according to the Unified Soils Classification System (USCS), and soil will be inspected for evidence (visual or olfactory) of contamination and field screened continuously for VOCs using a PID with a 10.6 electron volt (eV) lamp.

The following table provides a listing of the soil sampling locations, sample depth intervals, analytical suites and rationale for sampling:

| Soil Boring Location | Sample Depth Interval (ft bls) | Analytical Suites | Rationale |
|---|---|---|---|
| RXS-01<br>RXS-02<br>RXS-03<br>RXS-04 | ▪ 0-0.5 ft bls<br>▪ Most impacted 2 ft interval, if applicable*<br>▪ Bottom 2 ft of Fill<br>▪ Clean 2 ft Interval Beneath Impacts or Fill, whichever is deeper<br>▪ 2 ft above Groundwater Table | Full Part 375** and ECs*** | Evaluate soil conditions on Site and determine extent of contamination both vertically to the groundwater interface and horizontally across the Site. |
| RXS-05<br>RXS-06<br>RXS-07<br>RXS-08<br>RXS-09<br>RXS-10<br>RXS-11 | ▪ 0-0.5 ft bls<br>▪ Most impacted 2 ft interval, if applicable*<br>▪ Bottom 2 ft of Fill<br>▪ Clean 2ft Interval Beneath Impacts or Fill<br>▪ Soil Boring Termination Depth | Full Part 375 and ECs | Evaluate soil conditions on Site and determine extent of contamination both vertically to the historical fill/native soil interface and horizontally across the Site. |

* Note: If applicable, a sample will be collected from the most impacted interval as determined by field observations (e.g. staining and odors). If no impacts are observed, then a sample will not be collected.

** Note: Part 375/TCL plus 30/ TAL (TCL + 30/TAL) includes:

- Part 375/TCL VOCs + 10 TICs;
- Part 375/TCL Base Neutral Acids (BNA)/ SVOCs + 20 TICs;
- Part 375/TCL Pesticides;
- Part 375/TCL Herbicides;
- Part 375/TCL PCBs;
- Part 375/TAL Metals (including hexavalent chromium); and
- Total Cyanide.

***Note: Emerging Contaminants (ECs) list includes 1,4-Dioxane and the Per- and Polyfluoroalkyl Substances (PFAS) compounds listed in the NYSDEC April 2023 "Sampling, Analysis, and Assessment of Per-And Polyfluoroalkyl Substances (PFAS)". PFAS in soil will be analyzed by USEPA Method 1633A and 1,4-Dioxane in soil will be analyzed by USEPA Method 8270D.

## 3.2 Soil Vapor Investigation

Soil vapor sampling points will be installed at six locations on the Site and will be installed approximately 15 ft bls.

In accordance with the New York State Department of Health (NYSDOH) Guidance for Evaluating Soil Vapor Intrusion, the integrity of each soil vapor sampling point seal will be checked via a tracer gas (Helium) test following installation to verify that the vapor sample will not be compromised by inadvertent introduction of ambient air into the sample. A batch-certified summa canister and flow controller will be used to collect the soil vapor samples over a two-hour duration. Three soil vapor samples will be analyzed at a NYSDOH ELAP-certified laboratory for organic vapors via USEPA Method TO-15. Quality Assurance/ Quality Control (QA/QC) samples (i.e., field duplicates and field blanks) are proposed as part of this investigation to meet Part 375 data quality objectives.

If there are any changes with the scope a revision of the HASP will be required to address any new hazards.

## 3.3 Groundwater Investigation

To evaluate groundwater quality conditions across the Site, four permanent monitoring wells (RXS-01 through RXS-04) will be installed in converted soil boring locations. Roux will collect a groundwater sample from each newly installed monitoring well location, as shown on Figure 5 of the RIWP. All monitoring wells will be surveyed for horizontal coordinates, land surface elevation and measuring point elevation.

Monitoring wells will be constructed of 2-inch inside diameter, Schedule 40 polyvinyl chloride (PVC) casing and, 0.020 inch slot, machined screen. Well construction will be as follows:

- Well screens will be 10 feet long, and will be installed with three feet above and seven feet below the water table.
- A sand pack will be placed around the well screen, extending two feet above the top of the screened zone.
- A minimum two foot thick bentonite pellet seal will be placed above the sand pack. Once the pellets have been allowed to hydrate, cement bentonite grout will be pumped into the remaining annular space from the bottom up.
- The wells will be completed using locking well plugs, and flush mounted, bolt down, watertight, manhole covers cemented into place.

Each monitoring well to be sampled will be developed to ensure proper hydraulic connection with the aquifer and to reduce/eliminate turbidity of the groundwater. The wells will be developed using a submersible pump, which will be surged periodically until well yield is consistent and has turbidity below 50 nephelometric turbidity units (NTUs), if possible. Detailed procedures regarding well development can be found in the QAPP/FSP (Appendix B of the RIWP).

Prior to sampling, the depth to groundwater in each monitoring well will be measured using an electronic water level meter and a groundwater contour map will be developed using the survey data. At least one week following redevelopment, all on-Site wells will then be sampled consistent with USEPA low-flow sampling requirements using a peristaltic pump and one round of groundwater samples will be collected in accordance with the QAPP/FSP. Field parameters (e.g., pH, dissolved oxygen, oxidation-reduction potential [ORP], etc.) will also be collected using a water quality meter during purging and prior to sampling.

# 4. Site Control

This Site control program is designed to reduce the spread of hazardous substances from contaminated areas to clean areas, to identify and isolate contaminated areas of the Site, to facilitate emergency evacuation and medical care, to prevent unauthorized entry to the Site, and to deter vandalism and theft.

## 4.1 Site Map

A map of this Site, showing Site boundaries, designated work zones, and points of entry and exit is provided in Figure 2.

## 4.2 Site Access

Access to the work areas at the Site is restricted to reduce the potential for exposure to its safety and health hazards. During hours of Site operation, Site entry and exit is authorized only at the points identified in **Figure 2**. Entry and exit at these points are controlled by the following: chain linked fences and locked gates. When the Site is not operating, access to the Site is controlled by the following: chain linked fences and locked gates.

## 4.3 Buddy System

This section is not applicable for all components of the SOW described in Section 3. Some Site inspections and investigation activities are completed by a single Roux employee. However, when completing these tasks, the single Roux employee is accompanied either by Roux subcontractors or the Site caretaker/other representatives from Steps Senior. Any time Roux is on-site, Steps Senior is made aware and communications with Steps Senior and the Roux PM is maintained via cellular phone.

While working in the Exclusion Zone, Site workers use the buddy system. The buddy system means that personnel work in pairs and stay in close visual contact to be able to observe one another and summon rapid assistance in case of an emergency. The responsibilities of workers using the buddy system include:

- Remaining in close visual contact with partner;
- Providing partner with assistance as needed or requested;
- Observing partner for signs of heat stress, chemical overexposure or other difficulties;
- Periodically checking the integrity of partner's PPE; and
- Notifying the SS or other Site personnel if emergency assistance is needed.

## 4.4 Site Communications

The following communication equipment is used to support on-site communication:

- Hand signals to communicate with heavy equipment operators; and
- Cell phones, as needed. As applicable, hand signals will be used according to the following:

**Hand Signals**

| SIGNAL | MEANING |
|---|---|
| Hand gripping throat | Out of air; I cannot breathe |
| Grip partner's wrist | Leave area immediately |
| Hands on top of head | Need assistance |
| Thumbs up | I am all right; okay |
| Thumbs down | No; negative |

A current list of emergency contact numbers is included in the Site-Specific Emergency Information at the beginning of this HASP.

## 4.5 Site Work Zones

This Site is divided into three (3) major zones, described below. These zones are characterized by the likely presence or absence of biological, chemical, or physical hazards and the activities performed within them. Zone boundaries are clearly marked at all times and the flow of personnel among the zones is controlled. The Site is monitored for changing conditions that may warrant adjustment of zone boundaries. Zone boundaries are adjusted as necessary to protect personnel and clean areas. Whenever boundaries are adjusted, zone markings are also changed and workers are immediately notified of the change.

**Exclusion Zone**

The area where contamination exists is the Exclusion Zone (EZ). All areas where excavation and handling of contaminated materials take place are considered part of the EZ. This zone will be clearly delineated by chain link fencing, caution tape, cones or other effective barriers, as necessary. Safety tape may be used as a secondary delineation within the EZ. The zone delineation markings may be opened in areas for varying lengths of time to accommodate equipment operation or specific construction activities. The SHSO may establish more than one EZ where different levels of protection may be employed or where different hazards exist. Personnel are not allowed in the EZ without:

- A buddy (co-worker);
- Required minimum level PPE;
- Medical authorization;
- Training certification; and
- Requirement to be in the zone.

**Contamination Reduction Zone**

A Contamination Reduction Zone (CRZ) is established between the exclusion zone and the support zone. The CRZ contains the Contamination Reduction Corridor (CRC) and provides an area for decontamination of personnel and equipment. The CRZ will be used for general Site entry and egress in addition to access for heavy equipment and emergency support services. Personnel are not allowed in the CRZ without:

- A buddy (co-worker);

- Appropriate PPE;
- Medical authorization;
- Training certification; and
- Requirement to be in the zone.

**Support Zone**

The Support Zone (SZ) is an uncontaminated area that will be the field support area for the Site operations. The SZ will contain the appropriate equipment and materials for the SOW. Appropriate sanitary facilities and safety equipment will be located in this zone. Potentially contaminated personnel or materials are not allowed in this zone. The only exception will be appropriately packaged/decontaminated and labeled samples.

# 5. Job Hazard Evaluation

Roux's work at the Site is expected to entail a variety of physical, chemical, and biological hazards, all of which must be sufficiently managed to allow the work to be performed safely. Some of the hazards are Site-specific (i.e., they are associated with the nature, physical characteristics, and/or routine operation of the Site itself), while others are activity-specific (i.e., they are associated with [or arise from] the particular activity being performed). The various hazards can be grouped into the following categories:

- **Caught/Crushed** – the potential to become caught in, under, between, or by an object or parts of an object, such as equipment with parts that open and close or move up and down ("pinch points") or equipment that rotates, and the accompanying potential to have body parts cut, mangled, or crushed thereby.
- **Contact** – the potential to be struck by or against moving or stationary objects that can cause physical injury, such as heavy machinery, overhead piping, moving vehicles, falling objects, and equipment (including tools and hand-held equipment) or infrastructure with the ability to cut or impale.
- **Energy Sources** – the potential for bodily harm associated with energy sources, most notably electricity, but also including latent energy sources such as compressed air and equipment under tension (which when released could cause injurious contact or a fall).
- **Ergonomics** – the potential for musculoskeletal injury associated with lifting/carrying, pushing/pulling, bending, reaching, and other physical activity attributable to poor body position/mechanics, repetitive motion, and/or vibration.
- **Exposure** – the potential for injury/illness due to physical, chemical, or biological exposures in the work environment, including, but not limited to, temperature extremes, solar radiation, and noise (physical), chemical splashes and hazardous atmospheres (chemical), and animal/insect bites and poisonous plants (biological).
- **Falls** – the potential to slip or trip and thus fall or drop a load, resulting in bodily injury to oneself or others.

The foregoing is intended to provide Roux employees with a general awareness of the hazards involved with Site work. A more detailed review of the potential hazards associated with each specific activity planned for the Site (or on-going activity, as the case may be) is provided in the activity-specific Job Safety Analysis (JSA) forms in **Appendix A**. As can be seen in the JSA forms, the hazards are identified by category per the above, and specific measures designed to mitigate/manage those hazards are also identified. In preparing the JSA forms, all categories of hazards were considered, and all anticipated potential hazards were identified to the extent possible based on the experience of the personnel preparing and reviewing the JSA forms. However, there is always the possibility for an unanticipated hazard to arise, potentially as condition change over the course of the workday. Roux personnel must maintain a continual awareness of potential hazards in the work zone, regardless of whether the hazard is identified in the JSA form. Particular attention should be paid to hazards associated with exposure to hazardous substances (see **Table 1** for a listing of the hazardous substances most likely to be encountered in environmental media at the Site) and to Site personnel being located "in the line of fire" with respect to moving equipment, pinch points, and latent energy (e.g., being located or having body parts located within the swing radius of an excavator, between two sections of pipe being connected, below a piece of suspended equipment, or adjacent to a compressed air line).

## 5.1 Hazard Communication and Overall Site Information Program

The information in the JSAs and SDSs is made available to all employees and subcontractors who could be affected by it prior to the time they begin their work activities.

Modifications to JSAs are communicated during routine pre-work briefings, and periodically updated as needed in the HASP. SDSs will be maintained by the SHSO/SS for new chemicals brought on-site as needed. Copies of SDSs can be found in **Appendix B**.

## 5.2 Noise

Noise is associated with the operation of heavy equipment, power tools, pumps, and generators. Noise is also a potential hazard when working near operating equipment such as excavators, drill rigs or pole drivers. High noise (i.e., < 85 dBA) operations may be evaluated by the SHSO utilizing a type 2 handheld sound level meter (SLM) operating on the "A"-weighted scale with slow response because this scale most closely resembles human response to noise and complies with OSHA 29 CFR 1910.95. Hearing protection is required in areas with noise exposure greater than 85 dBA. Double hearing protection (ear plugs and earmuffs) should be donned in areas where the noise exposure is more than 95 dBA. Noise exposure will be controlled by hearing protection as described above or by maintaining set-backs from high-noise equipment, as warranted. Personnel handling heavy equipment and using power tools that produce noise levels exceeding those described levels above are required by OSHA 29 CFR 1910.95 to wear the appropriate Noise Reduction Rating (NRR) level of hearing protection. Appropriate hearing protection will be evaluated by the SHSO as necessary in consultation with the OHSM and CHSD.

## 5.3 Slip, Trip, and Fall Hazards

Slip, trip, and fall hazards may include, but are not limited to, general slip and trip hazards associated with uneven ground, possible debris, wet grass, and equipment. Prior to work, walking paths will be assessed for solid footings, any ground penetrations that may cause a tripping hazard will be appropriately marked, and other areas will be noted and discussed with the field team.

Personnel shall be aware of their surroundings and footings at all time, and all accommodations should be made for proper housekeeping and organized equipment placement at the Site, where possible, to help prevent any slip, trip, and fall-related incidents. All tools and materials should be appropriately stored when not in use and placed in appropriate storage containers.

## 5.4 Biological Hazards

Biological hazards that may potentially be present at a Site, include poisonous plants, insects (ticks, spiders, bees), animals (snakes, dogs), etc. Information on biological hazards can be found within Roux's Biological Hazard Awareness Management Program located within Roux's Corporate Health and Safety Manual. There is also potential for transmission and/or exposure to SARS-CoV-2, the virus that causes COVID-19. Prior to beginning work, on-Site protocols shall be established by the project team, including subcontractors, in accordance with federal, state, county, city, and/or other guidance, as applicable.

# 6. Emergency Response Plan

This emergency response plan details actions to be taken in the event of Site emergencies. The PM and SHSO is responsible for the implementation of emergency response procedures onsite. The SHSO/PM provides specific direction for emergency action based upon information available regarding the incident and response capabilities and initiates emergency procedures and notification of appropriate authorities. In the event of an emergency, Site personnel are evacuated and do not participate in emergency response activities, response is facilitated through external emergency services.

## 6.1 Emergency Response

The SHSO, after investigating the incident and relevant information, shall determine the level of response required for containment, rescue and medical care. Limited on-site emergency response activities could occur therefore the SHSO is responsible for notifying external emergency response agencies. The SHSO provides relevant information to the responding organizations, including, but not limited to, the hazards associated with the emergency incident, potential containment problems, and missing Site personnel.

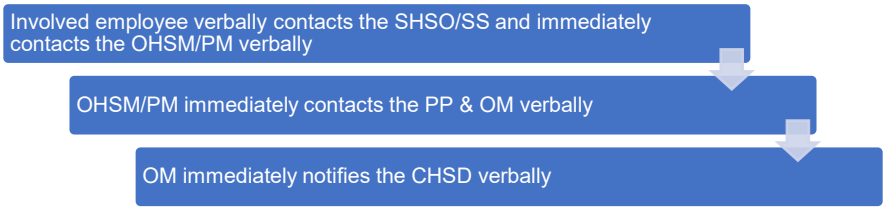
## 6.2 Emergency Alerting and Evacuation

If evacuation notice is given, Site workers leave the worksite, if possible, by way of the nearest exit. Appropriate primary and alternate evacuation routes and assembly areas have been identified and are shown on the Site Plan with Emergency Muster Area (**Figure 2**). The routes and assembly area will be determined by conditions at the time of the evacuation based on wind direction, the location of the hazard source, and other factors as determined by SHSO/PM.

Personnel exiting the Site gather at a designated assembly point. To determine that everyone has successfully exited the Site, personnel will be accounted for at the assembly point. If any worker cannot be accounted for, notification is given to the SHSO, PM, and any arriving response authorities so that appropriate action can be initiated. Subcontractors on this Site have coordinated their emergency response plans to ensure that these plans are compatible and potential emergencies are recognized, alarm systems are clearly understood, and evacuation routes are accessible to all personnel relying upon them.

## 6.3 Emergency Medical Treatment and First Aid

In the event of a work-related injury or illness, employees are required to follow the procedures outlined below. All work-place injury and illness situations require Roux's Project and Corporate Management Team to be notified when an injury/illness incident occurs, and communication with the contracted Occupational Health Care Management Provider, WorkCare, Inc., is initiated, as necessary. The Injury/Illness Notification Flowchart is provided below and within Roux's Incident Investigation and Reporting program included within Roux's Corporate Health and Safety Manual.



If on-Site personnel require any medical treatment, the following steps will be taken:

a. Notify Roux's Project and Corporate Management Team for any work-related injury and/or illness occurrence, and communicate with the contracted Occupational Health Care Management Provider, WorkCare, immediately following the notifications provided above.

b. Based on discussions with the Project Team, Corporate Management and the WorkCare evaluation, if medical attention beyond onsite first aid is warranted, transport the injured / ill person (IP) to the Urgent Care Center, or notify the Fire Department or Ambulance Emergency service and request an ambulance or transport the victim to the hospital, and continue communications with Corporate Management Team. An Urgent Care/Hospital Route map with location to CityMD Crown Heights Brooklyn Urgent Care and King County Hospital Center is included as **Figure 3**.

c. Decontaminate to the extent possible prior to administration of first aid or movement to medical or emergency facilities.

d. First aid medical support will be provided by onsite personnel trained and certified in First Aid, Cardio Pulmonary Resuscitation (CPR), Automatic External Defibrillation (AED), and Blood-Borne Pathogens (BBP) Awareness, until relieved by emergency medical services (EMS).

e. The SHSO and PM will perform a Loss Investigation (LI) and the Project Team will complete the final Loss Report. If a Roux employee is involved in a vehicular incident, the employee must also complete the Acord Automobile Loss Notice.

# 7. Environmental Conditions and Response

## 7.1 Adverse Weather Conditions

In the event of adverse weather conditions, the SHSO or project principal will determine if work can continue without jeopardizing the health and safety of field workers. Some of the items related to adverse weather conditions to be considered prior to determining if work should continue include:

- Potential for heat stress and heat-related injuries;
- Potential for cold stress and cold-related injuries;
- Treacherous weather-related conditions. If wind speed is greater than 15 mph averaged over a 15-minute period or wind gusts over 25 mph, earthmoving operations will be ceased;
- Limited visibility; and
- Electrical storm potential.

Site activities will be limited to daylight hours and acceptable weather conditions. Inclement working conditions may include heavy rain, fog, high winds, and lightning. The SHSO and/or PM shall observe daily weather reports and evacuate, if necessary, in case of inclement weather conditions.

## 7.2 Electrical Storm Guidelines

In the event that lightning and/or thunder are observed while working onsite, all onsite activities shall stop and personnel shall seek proper shelter (e.g., substantial building, enclosed vehicle, etc.). Work shall not resume until the threat of lighting has subsided and no lightning or thunder has been observed for 30 minutes. If the possibility of lightning is forecast for the day, advise the onsite personnel on the risks and proper procedure at the pre-work safety briefing. Continuously monitor for changing weather conditions and allow enough time to properly stop work if lightning is forecast.

## 7.3 Environmental Stressors, Heat Stress, Heat Exhaustion, and Heat Stroke

It is the employer's responsibility to monitor weather forecasts and ambient air temperatures, both prior to the work shift and during the shift. The National Oceanic and Atmospheric Administration records average minimum/maximum temperatures of 20-87 degrees Fahrenheit during the year in Brookyln, New York.

To prevent potential heat illness, the following strategies will be implemented:

- Adjusting personnel work/rest intervals;
- Monitoring for symptoms of heat illness;
- Providing shaded rest areas;
- Providing cool potable water so that each employee has access to at least one quart per hour for the entire shift, free of charge;
- Allowing for employees to acclimatize to the weather conditions and work demands;
- Observe workers during a heat wave (i.e., when the temperature is at least 80 degrees F, and 10 degrees hotter than the average temperature of the five preceding days); and
- Implementing high heat procedures when the temperature reaches 95oF.

Roux's Heat Illness Prevention Plan is implemented when the when ambient temperatures exceed 80oF. Roux's Heat Illness Prevention Program can be found within **Appendix C**. Additional information regarding heat illnesses is provided below. This can include, but is not limited to, access to shade that is sufficient in size to fit all workers who are on break; a car with air conditioning is acceptable, too. Preventative cool-down breaks shall be allowed at any time, and anyone taking such a break will be monitored for heat illness symptoms and not required to return to work until all symptoms (if present) of heat illness have disappeared. If necessary, first aid will be offered, but if symptoms are severe, emergency response procedures will be implemented per Section 6.3. Anyone who has shown symptoms of severe heat illness will not be sent home without being offered first aid or medical treatment.

High heat procedures must be implemented when temperatures reach 95oF. These procedures include:

- Ensuring that effective communication by voice, observation, or electronic means is maintained so that employees at the work site can contact a supervisor when necessary. An electronic device, such as a cell phone or text messaging device, may be used for this purpose only if the reception in the area is reliable.
- Observing employees for alertness and signs or symptoms of heat illness. The employer shall ensure effective employee observation/monitoring by implementing one or more of the following:
    - Supervisor or designee observation of 20 or fewer employees;
    - Mandatory buddy system; and
    - Regular communication with sole employee such as by radio or cellular phone, or other effective means of observation.
- Designating one or more employees on each worksite as authorized to call for emergency medical services and allowing other employees to call for emergency services when no designated employee is available.
- Encouraging employees throughout the work shift to drink plenty of water.
- A review of the high heat procedures during the daily tailgate meeting and remind employees of their right to take a cool-down rest when necessary.

### 7.3.1 Heat Stress

Heat stress is the body's response to excessive heat and can be a significant potential hazard. The risk of heat stress can be increased with heavy physical activity and/or the use of personal protective equipment in hot, humid weather environments. There are also personal risk factors that can contribute to the risk of suffering from heat stress, such as obesity, water intake, alcohol and caffeine consumption, pregnancy, age, medication, etc. Heat illness includes heat cramps, heat exhaustion, heat syncope, and heat stroke.

### 7.3.2 Heat Cramps

Heat cramps may be brought on by prolonged exposure to heat. As an individual sweats, water and salts are lost by the body resulting in painful muscle cramps, typically in the legs.

First aid treatment includes, but is not limited to, shade, rest, and fluid replacement. Typically, the individual should recover within one-half hour while being monitored constantly. If the individual has not improved substantially within 30 minutes and the body temperature has not decreased, the individual should be transported to a hospital for medical attention.

Per Roux's Heat Illness Prevention Plan, employees shall have access to potable drinking water that is fresh, pure, suitably cool, free of charge and in sufficient quantities. Access to shade shall be present when temperatures exceed 80 degrees Fahrenheit and shall be available when temperatures do not exceed 80 degrees Fahrenheit.

### 7.3.3 Heat Exhaustion

Heat exhaustion may occur in an individual who has been exposed to excessive heat while working or exercising. The circulatory system of the individual fails as blood collects near the skin to rid the body of excess heat through transference. The signs and symptoms of heat exhaustion are as follows:

- Rapid and shallow breathing;
- Weak pulse;
- Cold and clammy skin with heavy perspiration;
- Skin appears pale;
- Fatigue and weakness;
- Dizziness; and
- Elevated body temperature.

First aid treatment includes, but is not limited to, cooling the victim, elevating the feet, and replacing fluids.

If the individual is not substantially improved within 30 minutes and the body temperature has not decreased, the individual should be transported to the hospital for medical attention.

### 7.3.4 Heat Stroke

Heat stroke occurs when an individual is exposed to excessive heat and stops sweating. This condition is classified as a MEDICAL EMERGENCY requiring immediate cooling of the victim and transport to a medical facility. The signs and symptoms of heat stroke are as follows:

- Dry, hot, red skin;
- Body temperature approaching or above 105oF;
- Confusion, altered mental state, slurred speech;
- Seizures;
- Large (dilated) pupils; and
- Loss of consciousness – the individual may go into a coma.

First aid treatment requires immediate cooling and transportation to a medical facility. Heat stress is a significant hazard if any type of protective equipment (semi-permeable or impermeable) that prevents evaporative cooling when worn in hot weather environments.

## 7.4 Cold Stress

Cold stress is a danger at low temperatures and when the wind-chill factor is low. Prevention of cold-related illnesses is a function of whole-body protection. Adequate insulating clothing must be used when the air temperature is below 60°F. A work/rest regimen will be initiated when ambient temperatures and protective

clothing cause a stressful situation. In addition, reduced work periods followed by rest in a warm area may be necessary in extreme conditions. The signs and symptoms of cold stress include the following:

- Severe shivering;
- Abnormal behavior;
- Slowing;
- Weakness;
- Stumbling or repeated falling;
- Inability to walk;
- Collapse; and/or
- Unconsciousness.

First aid requires removing the victim from the cold environment and seeking medical attention immediately. Also, prevent further body heat loss by covering the victim lightly with blankets. Do not cover the victim's face. If the victim is still conscious, administer hot drinks and encourage activity such as walking, wrapped in a blanket.

# 8. Safety Procedures

This section of the HASP presents the specific safety procedures to be implemented during Roux's activities at the Site in order to protect the health and safety of various on-site personnel. Minimum OSHA-mandated procedures are presented first, followed by client- and Site-specific procedures. Lastly, activity-specific procedures are discussed. These Site and activity-specific procedures supplement the general safety procedures included in Roux's Corporate Health and Safety Manual, which also must be followed in their entirely.

## 8.1 Training

At a minimum, Site personnel who will perform work in areas where there exists the potential for toxic exposure will be health and safety-trained prior to performing work onsite per OSHA 29 CFR 1910.120(e) and 29 CFR 1926.65(e). More specifically, all Roux, subcontractor, and other personnel engaged in sampling and remedial activities at the Site and who are exposed or potentially exposed to hazardous substances, health hazards, or safety hazards must have received at a minimum the 40 hour initial HAZWOPER training consistent with the requirements of 29CFR 1910.120(e)(3)(i) training and a minimum of 3 days' actual field experience under the direct supervision of a trained experienced supervisor, plus 8 hours of refresher training on an annual basis. Depending on tasks performed, less training may be permitted. Evidence of such training must be maintained at the Site at all times. Furthermore, all on-Site management and supervisory personnel directly responsible for or who supervise the employees engaged in Site remedial operations, must have received an additional 8 hours of specialized training at the time of job assignment on topics including, but not limited to, the employer's safety and health program and the associated employee training program, personal protective equipment program, spill containment program, and health hazard monitoring procedure and techniques, plus 8 hours of refresher training on an annual basis. Additionally, all workers who will be required to don a respirator will be properly trained on their employer's Respiratory Protection Program, which also includes being medically cleared to wear a respirator and passed a fit test, at least initially prior to use, and then annually thereafter in accordance with 29 CFR 1910.134, Respiratory Protection.

Roux personnel training records are maintained in a corporate database with records available upon request from either the OHSM/SHSO/CHSD or Human Resources Department.

## 8.2 Site-Specific Safety Briefings for Visitors

A site-specific briefing is provided to all site visitors who enter this site beyond the site entry point. For visitors, the site-specific briefing provides information about site hazards, the site lay-out including work zones and places of refuge, the emergency alarm system and emergency evacuation procedures, and other pertinent safety and health requirements as appropriate.

## 8.3 HASP Information and Site-Specific Briefings for Workers

Site personnel review this HASP and are provided a Site-specific tailgate briefing prior to the commencement of work to ensure employees are familiar with this HASP and the information and requirements it contains, as well as the relevant JSAs included in **Appendix A**. Additional briefings are provided as necessary to notify employees of any changes to this HASP as a result of information gathered during on-going Site characterization and analysis of changing conditions. Conditions for which we

schedule additional briefings include, but are not limited to: changes in site conditions, changes in the work schedule/plan, newly discovered hazards, and incidents occurring during Site work.

## 8.4 Medical Surveillance

The medical surveillance section of the Health and Safety Plan describes how worker health status is monitored at this site. Medical surveillance is used when there is the potential for worker exposure to hazardous substance at levels above OSHA Permissible Exposure Limits (PEL) or other published limits. The purpose of a medical surveillance program is to medically monitor worker health to ensure that personnel are not adversely affected by site hazards. The provisions for medical surveillance at this site are based on the site characterization and job hazard analysis found in Section 4 of this HASP and are consistent with OSHA requirements in 29 CFR 1910.120(f) as applicable.

### 8.4.1 Site Medical Surveillance Program

Medical surveillance requirements are based on a worker's potential for exposure as determined by the site characterization and job hazard analysis documented in Section 4 and JSAs within **Appendix A** of this HASP and in compliance with the requirements of 29 CFR 1910.120(f)(2). Based on site information and use of direct reading instruments, limited use of respirators (less than 30 days per year), and the absence of an employee-staffed HAZMAT team, a limited medical surveillance program is required and implemented at this site. The medical surveillance program provides that:

1. Workers assigned to tasks requiring the use of respirators receive medical examinations in accordance with 29 CFR 1910.134(e) to ensure they are physically capable to perform the work and use the equipment, and
2. If a worker is injured, becomes ill, or develops signs or symptoms of possible over-exposure to hazardous substance or health hazards, medical examinations are provided to that worker as soon as possible after the occurrence and as required by the attending physician.
3. These medical examinations and procedures are performed by or under the supervision of a licensed physician and are provided to workers free of cost, without loss of pay, and at a reasonable time and place. In addition, the need to implement a more comprehensive medical surveillance program will be re-evaluated after any apparent over-exposure.

### 8.4.2 Medical Recordkeeping Procedures

Medical recordkeeping procedures are consistent with the requirements of 29 CFR 1910.1020 and are described in the company's overall safety and health program. A copy of that program is available at our Islandia, New York office.

The following items are maintained in worker medical records:

- Respirator fit test and selection;
- Physician's medical opinion of fitness for duty (pre-placement, periodic, termination);
- Physician's medical opinion of fitness for respirator protection (pre-placement, periodic); and
- Exposure monitoring results.

### 8.4.3 Program Review

The medical program is reviewed to ensure its effectiveness. The Corporate Health and Safety Manager, in coordination with the Human Resources Director, is responsible for this review. At minimum, this review consists of:

- Review of accident and injury records and medical records to determine whether the causes of accidents and illness were promptly investigated and whether corrective measures were taken wherever possible;
- Evaluation of the appropriateness of required medical tests based on site exposures; and
- Review of emergency treatment procedures and emergency contacts list to ensure they were site specific, effective, and current.

## 8.5 Personnel Protection

Site safety and health hazards are eliminated or reduced to the greatest extent possible through engineering controls and work practices. Where hazards are still present, a combination of engineering controls, work practices and PPE are used to protect employees. Appropriate personal protective equipment (PPE) shall be worn by Site personnel when there is a potential exposure to chemical, biological or physical hazards (e.g., falling objects, flying particles, sharp edges, electricity, and noise), as determined by the SHSO. The level of personal protection, type and kind of equipment selected will depend on the hazardous conditions and in some cases cost, availability, compatibility with other equipment, and performance. An accurate assessment of all these factors will be made before work can be safely executed.

Roux maintains a comprehensive written PPE program that addresses proper PPE selection, use, maintenance, storage, fit and inspection. Roux's PPE program can be found within **Appendix D**. PPE to be used at the Site will meet the appropriate American National Standards Institute (ANSI) standards and the following OSHA (General/Construction Industry) standards for minimum PPE requirements.

The minimum level of PPE for entry onto the Site is Level D. The following equipment shall be worn:

- Work uniform (long pants, sleeved shirt);
- Hard hat;
- Steel or composite toe work boots (must comply with American Society for Testing and Materials [ASTM]) F 2412-05, Standard Test Methods for Foot Protection and ASTM F 2413-05, Standard Specification for Performance Requirements for Foot Protection);
- Safety Glasses (must comply with one of the following ANSI/ISEA Z87.1-2010, ANSI Z87.1-2003, ANSI Z87.1-2003);
- Boot Covers (as needed);
- Hearing protection (as needed);
- High visibility clothing (shirt/vest); and
- Hand protection (e.g., minimum cut resistance meeting ANSI 105-2000 Level 2).

Note that jewelry shall be removed or appropriately secured to prevent it from becoming caught in rotating equipment or unexpectedly snagged on a fixed object (e.g., wrist watches, bracelets, rings, chains and necklaces, open earrings). Do not wear loose clothing and all shoulder-length hair should be tied back.

Site specific PPE ensembles and materials are identified within task specific JSAs located within **Appendix A**, and any upgrades or downgrades of the level of protection (i.e., not specified in the JSA) must be approved by the PP and immediately communicated to all Roux personnel and subcontractors as applicable. PPE is used in accordance with manufacturer's recommendations.

### 8.5.1 Hearing Conservation

Hearing protection is made available when noise exposures equal or exceed an 8-hour time-weighted average sound level of 85 dBA. Hearing protection is required when the 8-hour time weighted average sound level > 85 dBA, or when noise levels exceed 140 dBA at any point or exceeds 115 dBA for at least 15 minutes. Where noise exposure meets or exceeds this level, noise is listed as a physical hazard in the JSA for the tasks/operation, and hearing protection is included as one of the control measures (PPE).

## 8.6 Monitoring

An air monitoring program is important to the safety of on- and off-Site personnel, and the surrounding area. A preliminary survey, to establish background conditions in the immediate sampling area, may be made prior to the initiation of Site work including, but not limited to, monitoring wind direction (e.g., wind socks) and approximate temperature during all invasive Site activities. This survey will be conducted with the appropriate pre-calibrated air monitoring instrument(s), as warranted by the field activity. Once this survey has been complete, any changes in the type of PPE will be determined and relayed to those working on Site.

Work zone air monitoring will be performed to verify that the proper level of PPE is used, and to determine if increased protection or work stoppage is required. The following equipment shall be used to monitor conditions:

- A Photoionization Detector (PID) with a lamp energy of 10.6 eV will be used to provide direct readings of organic vapor concentrations during intrusive activities to determine that personnel protection is adequate. Concentrations shall be recorded during intrusive activities with the potential to encounter contaminant vapors.

Personal exposure monitoring utilizing activated charcoal tubes may be considered based on whether or not the area sample results are at or above half of the PEL. The decision to perform the monitoring will be made by, and under the control of, the CHSD.

Below are monitoring action levels for Site-specific chemicals of concern. In the event PID readings above the thresholds identified below are sustained for 5 minutes in the breathing zone, worker protection will require upgrading following notification to the OHSM and applicable parties.

### 8.6.1 Action Levels for Air Monitoring

PPE can remain at Level D if breathing zone VOC concentrations are less than 5 ppm and benzene is non detect. Personnel are required to evacuate the Site when breathing zone VOC readings exceed 25 ppm.

The following tables include summaries of the air monitoring, work practices, and action levels for the expected contaminants. The action levels to initiate testing with colorimetric tubes for airborne volatiles is 1 ppm (PID reading) and is based on the Permissible Exposure Limit (PEL) for benzene (1 ppm). The colorimetric tubes are used to confirm the presence or absence of specific constituents, and they do not provide a measured concentration.

PPE can remain at Level D if breathing zone VOC concentrations are less than 5 ppm is non-detect. Personnel are required to evacuate the Site when breathing zone VOC readings exceed 25 ppm.

The following tables include summaries of the air monitoring, work practices, and action levels for the expected contaminants.

| Air Monitoring Summary and Action Levels<br>Organic Vapors | |
|---|---|
| **PID Reading in Breathing Zone (ppm)** | **Action** |
| 0-1 ppm above background | Continue monitoring |
| >1-5 ppm sustained 60 seconds | Continue monitoring |
| <5 ppm | Continue Monitoring, ventilate space |
| ≥ 5 ppm - ≤ 25 ppm | Ventilate space until PID reads < 5 ppm. If < 5 ppm cannot be achieved, upgrade to Level C[1]. |
| ≥ 25 ppm | Ventilate space and evacuate area.<br>Consult with CHSD. |

Background concentrations should be established at the beginning of each work day. It may be necessary to re-establish background concentrations and ambient conditions vary through the day.

1 Measured air concentrations of known organic vapors will be reduced by the respirator to one half of the PEL or lower, and the individual and combined compound concentrations shall be within the service limit of the respirator cartridge.

### 8.6.2 Explosive Hazard

Methane is a common gas constituent that is generated during the decomposition of organics. Methane, a non-toxic compound, does not have an OSHA PEL or other exposure limits based on toxicity; however, methane is explosive under certain conditions. The Lower Explosive Limit (LEL) for methane is 5% in air, or 50,000 parts per million by volume. At high concentrations, methane can also displace oxygen and cause asphyxiation. Since methane is lighter than air, it does not tend to accumulate in low-lying areas and will rapidly mix with atmospheric air if encountered during drilling activities. Methane concentrations will be continuously monitored using a pre-calibrated gas meter while excavation takes place.

### 8.6.3 Air Monitoring Equipment and Calibration

A PID calibrated to an appropriate calibration mixture will be used to detect organic vapors in and around the work areas. Monitoring will be conducted in and around all work areas and at the workers breathing zone before activities commence to establish a background level, then at 15-minute intervals throughout the day. All equipment will be calibrated according to the manufacturer's recommendation. A calibration log will be maintained and will include the name of the person who performed the calibration, the date and time calibrated, and the instrument reading at the time of calibration. A manual bellows pump or equivalent with colorimetric tubes for specific compounds will be utilized to determine the course of action related to upgrading or downgrading the level of respiratory protection, as applicable.

If air monitoring data indicate safe levels of potentially harmful constituents at consistent intervals (5-minute intervals), then monitoring can be conducted less frequently (every 30 minutes). This determination will be made by the onsite SHSO. Monitoring data, including background readings and calibration records, will be documented. Work to be performed on-Site will conform to Roux's Standard Operating Procedures (SOPs). Conformance with these guidelines as well as the guidelines described in this HASP will aid in mitigating the physical and chemical hazards mentioned throughout this HASP.

## 8.7 Tailgate Safety Meetings

A designated Site worker will provide daily safety briefings (e.g., tailgate meetings) including, but not limited to, the following scenarios:

- When new operations are to be conducted;
- Whenever changes in work practices must be implemented; and
- When new conditions are identified and/or information becomes available.

Daily safety briefings shall be recorded on the Roux Daily Tailgate Health and Safety Meeting Log/Daily Site Safety Checklist, and all completed forms will become a part of the project file.

## 8.8 Spill Containment

Spill containment equipment and procedures should, at a minimum, meet the requirements of the facility's Spill Prevention, Control and Countermeasure Plan, if applicable. Otherwise, spill containment equipment and procedures must be considered depending on the task, including, but not limited to, chemical/product transfer points and handling.

### 8.8.1 Initial Spill Notification and Response

Any worker who discovers a hazardous substance spill will immediately notify the SHSO. Additionally, the worker/Roux personnel are to report the spill to the NYSDEC Spills Hotline within 2 hours of discovery. The worker will, to their best ability, report the hazardous substance involved, the location of the spill, the estimated quantity of material spilled, the direction/flow of the spill material, related fire/explosion incidents, and any associated injuries without compromising their own safety.

### 8.8.2 Spill Evaluation and Response

The SHSO is responsible for evaluating spills and determining the appropriate response. When this evaluation is being made, the spill area will be isolated and demarcated to the extent possible. If necessary to protect nearby community members, notification of the appropriate authorities is made by the PM as appropriate. On-Site response is limited to small spills (e.g., <10 gallons); large spills require external emergency responders who will be contacted by the SHSO.

## 8.9 Decontamination

The decontamination section of the HASP describes how personnel and equipment are decontaminated when they leave the Exclusion Zone. This section also describes how residual waste from decontamination processes is disposed. The site decontamination procedures are designed to achieve an orderly, controlled removal or neutralization of contaminants that may accumulate on personnel or equipment. These procedures minimize worker contact with contaminants and protect against the transfer of contaminants to clean areas of the site and off-site. They also extend the useful life of PPE by reducing the amount of time that contaminants contact and can permeate PPE surfaces. Decontamination is facilitated within the CRZ at this site, if applicable.

### 8.9.1 Decontamination Procedures for Personnel and PPE

The following are general decontamination procedures established and implemented at this site.

1. Decontamination is required for all workers exiting a contaminated area. Personnel may re-enter the SZ only after undergoing the decontamination procedures described below in the next section.
2. Protective clothing is decontaminated, cleaned, laundered, maintained and/or replaced as needed to ensure its effectiveness.
3. PPE used at this site that requires maintenance or parts replacement is decontaminated prior to repairs, or
4. PPE used at this site is decontaminated or prepared for disposal on the premises. Personnel who handle contaminated equipment have been trained in the proper means to do so to avoid hazardous exposure.
5. This site uses an off-site laundry for decontamination of PPE. The site has informed that facility of the hazards associated with contaminated PPE from this site.
6. The site requires and trains workers that if their permeable clothing is splashed or becomes wetted with a hazardous substance, they will immediately exit the work zone, perform applicable decontamination procedures, shower, and change into uncontaminated clothing.
7. Procedures for disposal of decontamination waste meet applicable local, State, and Federal regulations.

### 8.9.2 Decontamination Procedures for Equipment

All tools, equipment, and machinery from the EZ or CRZ are decontaminated in the CRZ prior to removal to the SZ. Equipment decontamination procedures are designed to minimize the potential for hazardous skin or inhalation exposure and to avoid cross-contamination and chemical incompatibilities.

General Equipment Decontamination Procedures:

1. Decontamination is required for all equipment exiting a contaminated area. Equipment may re-enter the SZ only after undergoing the equipment decontamination procedures.
2. Vehicles that travel regularly between the contaminated and clean areas of the site are carefully decontaminated each time they exit the EZ and the effectiveness of that decontamination is monitored to reduce the likelihood that contamination will be spread to other parts of the site.
3. Particular attention is given to decontaminating tires, scoops, and other parts of heavy equipment that are directly exposed to contaminants and contaminated soil.
4. Procedures for disposal of decontamination waste shall meet applicable local, State, and Federal regulations.

The following items may be used to decontaminate equipment:

- Fresh water rinse;
- Non-phosphorus detergent wash;
- Acetone rinse;
- Distilled water rinse; and
- A steam cleaner or pressure washer (heavy equipment only).

### 8.9.3 Monitoring the Effectiveness of Decontamination Procedures

Visual examination and sampling are used to evaluate the effectiveness of decontamination procedures. Visual examination is used to ensure that procedures are implemented as described and that they appear to control the spread of contaminants under changing site conditions. Visual examination is also used to inspect for signs of residual contamination or for contaminant permeation of PPE.

Personnel who work in contaminated areas of the site, either the Contamination Reduction Zone (CRZ) or the Exclusion Zone, are trained in the principles and practices of decontamination described in this section of the HASP and in related SOPs. If site procedures are changed as a result of inspection and monitoring, all affected employees are notified of these changes.

## 8.10 Confined Space Entry

No confirmed space entry is anticipated to occur.

The following is a list of the safety requirements for confined space entry at the Site:

- **ROUX PERSONNEL ARE NOT AUTHORIZED TO ENTER AN OSHA PERMIT REQUIRED CONFINED SPACE;**
- Currently the scope of work **DOES NOT** require personnel to enter permitted confined space for this project; and
- Any changes to the field activities that may necessitate confined space entry will be reported to the Project Principal and OHSM.

Confined space is defined as any space, depression, or enclosure that:

- Has limited opening for entry and egress;
- Is large enough for and employee to enter and perform assigned work; and
- Is not intended for continuous occupancy.

A permit required confined space is one that meets the definition of a confined space and has one or more of the following characteristics:

- May contain or produce life-threatening atmospheres due to oxygen deficiency the presence of toxic, flammable, or corrosive contaminants;
- Contains a material that has the potential for engulfment;
- Has an internal configuration that may cause an entrant to be trapped or asphyxiated by inwardly converging walls or by a floor that slopes downward and tapers to a smaller cross-section; and
- Contains any other serious safety or health hazards.

Although Roux personnel will not perform confined space entry, it is expected that subcontractors performing cleaning and mitigation and/or remedial measures activities may be required to enter structures that are considered to be a permit required confined space. Permitting of the confined space as well as hazard mitigation for entry will be completed by the subcontractor in accordance with 29 CFR 1910.146 or 1926.1201-1213 and/or applicable local/client requirements.

## 8.11 Client and Site-Specific

In addition to the OSHA-specific procedures discussed above, there may be client and site-specific safety procedures that must be adhered to during the performance of remedial activities at the Site.

## 8.12 Unusual or Significant Risks

Field activities that appear to have unusual or significant risks that cannot be adequately managed with existing risk tools such as LPS, HASPs, traffic safety plans, work permits, design and O&M practices, equipment HAZOPS or other safety tools must be referred to the CHSD to help with the assessment and management of the associated potential safety risks. Examples include the use of explosives for demolition, use of firearms to control wildlife, rappelling, demolition over water, diving, etc.

## 8.13 Activity-Specific Hazards

In addition to the general hazards discussed above, there are activity-specific hazards associated with each work activity planned for the Site. An activity-specific JSA has been completed for each of the activities planned for the Site. JSAs are provided in **Appendix A**. In the event that new work activities or tasks are planned, JSAs will be developed and implemented prior to performing the new activities. In the absence of a JSA, the personnel performing work must prepare a field JSA and receive clearance from a designated competent safety official prior to performing any task with significant risk. In emergency situations where time is critical, Last Minute Risk Assessments (LMRAs) will be utilized to identify the task, associated hazards and mitigative actions to take. For lower risk activities (as deemed by the discretion of a Competent Person) where a JSA is determined to not be needed, the individual(s) conducting the activities must perform LMRAs prior to and during the work.

### 8.13.1 Electrical and Other Utility Assessment and Accommodations

Roux shall perform a site walk to identify any potential overhead electrical or utility lines. All applicable guidelines will be followed in the vicinity of overhead power and utility lines (see Section 8.13.3 below).

Roux has also reviewed all available Site maps showing buried utility lines to identify potential hazards; the review revealed no underground hazards are known to exist in the vicinity of the areas of the Site pertinent to this HASP.

### 8.13.2 Subsurface Work

Subsurface work activities will require adherence to Roux's Corporate Subsurface Utility Clearance Management program found within Appendix E.

### 8.13.3 Heavy Equipment

Use of heavy equipment at the Site will require adherence to Roux's Corporate Heavy Equipment Exclusion Zone Management Program found within Appendix F. Additionally, operation of the drill rig/other heavy equipment will maintain clearances from overhead power lines in accordance with 29 CFR 1926.1408 Table A Minimum Clearance Distances provided below.

**Minimum Required Clearances for Energized Overhead Power Lines**

| Nominal System Voltage of Power Line (K V) | Minimum Required Clearance (feet) |
|---|---|
| 0-50 | 10 |
| 51-100 | 12 |
| 101-200 | 15 |
| 201-300 | 20 |
| 301-500 | 25 |
| 501-750 | 35 |
| 751-1000 | 45 |

1 kilovolt (KV) = 1,000 volts

## 8.14 Traffic Control

If Site operations encroach upon public streets or highways and a hazard exists to Site personnel because of traffic conditions, a traffic control plan will be implemented in accordance with the United States Department of Transportation's (DOT's) "Manual on Uniform Traffic Control Devices."

## 8.15 Sanitation

Sanitation facilities will be provided in accordance with the sanitation standards (29 CFR 1910.141, 29 CFR 1926.51 and 29 CFR 1928.110). Sanitation facilities will be maintained and kept in good conditions at all times.

# 9. Field Team Review

Each person performing work at or visiting this site shall sign this section after site-specific training is completed and before being permitted to access the CRZ or Exclusion Zone.

I have read and understand this Site-Specific Health and Safety Plan. I will comply with the provision contained therein.

**Site/Project: 2030 Fulton Street, Brookyln, NY/Steps at Saratoga**

| Name & Company | Signature | Date |
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# 10. Approvals

By their signature, the undersigned certify that this HASP is approved and will be utilized at the Steps Family Site.

| | |
|---|---|
| TBD – Site Health and Safety Officer | Date |
| Nevin Pahlad – Office Health and Safety Manager | Date |
| Brooke Hildebrand – Project Manager | Date |
| Jessica Taylor – Project Principal | Date |

# TABLE

1. Toxicological Properties of Hazardous Substances Present at the Site

**Table 1. Toxicological, Physical, and Chemical Properties of Compounds Potentially Present at the Site.**

| Compound | CAS # | ACGIH TLV | NIOSH REL | OSHA PEL | IDLH | Routes of Exposure | Toxic Properties | Target Organs | Physical/Chemical Properties |
|---|---|---|---|---|---|---|---|---|---|
| **ORGANOCHLORINE PESTICIDES (OCP)** | | | | | | | | | |
| DDT | 50-29-3 | TWA 1 mg/m3 | TWA 0.5 mg/m3 | TWA 1 mg/m3 | 500 mg/m3 | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin; paresthesia tongue, lips, face; tremor; anxiety, dizziness, confusion, malaise (vague feeling of discomfort), headache, lassitude (weakness, exhaustion); convulsions; paresis hands; vomiting; [potential occupational carcinogen] | Eyes, skin, central nervous system, kidneys, liver, peripheral nervous system | White, odorless and tasteless, very stable, water-insoluble, synthetic<br><br>BP: 260°F<br>Fl.Pt. = 162-171°F<br>LEL: NA<br>UEL: NA |
| Aldrin | 309-00-2 | TWA 0.1 mg/m3 | TWA 0.25 mg/m3 | TWA 0.25 mg/m3 | 25 mg/m3 | Inhalation, ingestion, skin and/or eye contact | headache, dizziness; nausea, vomiting, malaise (vague feeling of discomfort); myoclonic jerks of limbs; clonic, tonic convulsions; coma; hematuria (blood in the urine), azotemia; [potential occupational carcinogen] | Developmemntal, Endocrine, Liver, Immune System, Nervous System, | Colorless to dark-brown crystalline solid with a mild chemical odor.<br><br>BP: 293°F<br>Fl.Pt. = 150°F<br>LEL: NA<br>UEL: NA |
| Lindane (gamma-BHC) | 58-89-9 | TWA 0.5 mg/m3 | TWA 0.5 mg/m3 | TWA 0.5 mg/m3 | 50 mg/m3 | Inhalation, ingestion, skin and/or eye contact | irritation eyes, skin, nose, throat; headache; nausea; clonic convulsions; resp difficulty; cyanosis; aplastic anemia; muscle spasm; In Animals: liver, kidney damage | Eyes, skin, respiratory system, central nervous system, blood, liver, kidneys | White to yellow, crystalline powder with a slight, musty odor.<br><br>BP: 614°F<br>Fl.Pt. = 150°F<br>LEL: NA<br>UEL: NA |
| Dieldrin | 860-57-1□ | TWA 0.1 mg/m3 | TWA 0.25 mg/m3 | TWA 0.25 mg/m3 | 25 mg/m3 | Inhalation, ingestion, skin and/or eye contact | headache, dizziness; nausea, vomiting, malaise (vague feeling of discomfort), sweating; myoclonic limb jerks; clonic, tonic convulsions; coma; ; In Animals: liver, kidney damage [potential occupational carcinogen] | Developmemntal, Endocrine, Liver, Immune System, Nervous System, | Colorless to light-tan crystals with a mild, chemical odor.<br><br>BP: NA (Decomposes)<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| **VOLATILE ORGANIC COMPOUNDS (VOCs)** | | | | | | | | | |
| 1,1,1-Trichloroethane | 71-55-6 | TWA 350 ppm<br>STEL 450 ppm | C 350 ppm (1900 mg/m$^3$) [15-minute] | TWA 350 ppm (1900 mg/m$^3$) | 700 ppm | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin; headache, lassitude (weakness, exhaustion), central nervous system depression, poor equilibrium; dermatitis; cardiac arrhythmias; liver damage | Eyes, skin, central nervous system, cardiovascular system, liver | Colorless liquid with a mild, chloroform-like odor.<br>BP: 165°F<br>Fl.Pt. = NA<br>LEL: 7.5%<br>UEL: 12.5%<br>Combustible Liquid, but burns with difficulty |
| 1,1,2,2-Tetrachloroethane | 79-34-5 | TWA 1 ppm [skin] | Ca TWA 1 ppm (7 mg/m^3) [skin] | TWA 5 ppm (35 mg/m^3) [skin] | Ca [100 ppm] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Nausea, vomiting, abdominal pain; tremor fingers; jaundice, hepatitis, liver tenderness; dermatitis; leukocytosis (increased blood leukocytes); kidney damage; [potential occupational carcinogen] | Skin, liver, kidneys, central nervous system, gastrointestinal tract | Colorless to pale-yellow liquid with a pungent, chloroform-like odor<br>BP: 296°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible Liquid |
| 1,1,2-Trichloro-1,2,2-trifluoroethane | 76-13-1 | TWA 1000 ppm<br>STEL 1250 ppm | TWA 1000 ppm (7600 mg/m3)<br>ST 1250 ppm (9500 mg/m3) | TWA 1000 ppm (7600 mg/m3) | 2000 ppm | Inhalation, ingestion, skin and/or eye contact | Irritation skin, throat, drowsiness, dermatitis; central nervous system depression; In Animals: cardiac arrhythmias, narcosis | Skin, heart, central nervous system, cardiovascular system | Colorless to water-white liquid with an odor like carbon tetrachloride at high concentrations. [Note: A gas above 118°F.]<br>BP: 118°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| 1,1,2-Trichloroethane | 79-00-5 | TWA 10 ppm [skin] | Ca TWA 10 ppm (45 mg/m3) [skin] | TWA 10 ppm (45 mg/m3) [skin] | Ca [100 ppm] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, nose; central nervous system depression; liver, kidney damage; dermatitis; [potential occupational carcinogen] | Eyes, respiratory system, central nervous system, liver, kidneys | Colorless liquid with a sweet, chloroform-like odor<br>BP: 237°F<br>Fl.Pt. = NA<br>LEL: 6%<br>UEL: 15.5%<br>Combustible Liquid, forms dense soot |
| 1,1-Dichloroethane | 75-34-3 | TWA 100 ppm | TWA 100 ppm (400 mg/m^3) | TWA 100 ppm (400 mg/m^3) | 3,000 ppm | Inhalation, ingestion, skin and/or eye contact | Irritation skin; central nervous system depression; liver, kidney, lung damage | Skin, liver, kidneys, lungs, central nervous system | Colorless, oily liquid with a chloroform-like odor.<br>BP: 135°F<br>Fl.Pt. = 2°F<br>LEL: 5.4%<br>UEL: 11.4%<br>Class IB Flammable Liquid Fl.P. below 73°F and BP at or above 100°F. |

**Table 1. Toxicological, Physical, and Chemical Properties of Compounds Potentially Present at the Site.**

| Compound | CAS # | ACGIH TLV | NIOSH REL | OSHA PEL | IDLH | Routes of Exposure | Toxic Properties | Target Organs | Physical/Chemical Properties |
|---|---|---|---|---|---|---|---|---|---|
| 1,1-Dichloroethene | 75-35-4 | TWA 5 ppm | Ca | None | Ca | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin, throat; dizziness, headache, nausea, dyspnea (breathing difficulty); liver, kidney disturbance; pneumonitis; [potential occupational carcinogen] | Eyes, skin, respiratory system, central nervous system, liver, kidneys | Colorless liquid or gas (above 89°F) with a mild, sweet, chloroform-like odor<br>BP: 89°F<br>Fl.Pt. = -2°F<br>LEL: 6.5%<br>UEL: 15.5%<br>Class IA Flammable Liquid: Fl.P. below 73°F and BP below 100°F |
| 1,2,3-Trichlorobenzene | 87-61-6 | Cameo Chemicals Source https://cameochemicals.noaa.gov/chemical/10051 | NA | NA | NA | Inhalation, skin absorption, ingestion, skin and/or eye contact | Inhalation may cause irritation of respiratory tract. Irritating to the eyes. May redden skin on contact. Ingestion may cause liver damage. | Skin, eyes, respiratory tract, liver | A white solid with a sharp chlorobenzene odor. Insoluble in water and denser than water. Hence sinks in water<br>Fl.Pt. = 234.9°F |
| 1,2,4-Trichlorobenzene | 120-82-1 | C 5 ppm | C 5 ppm (40 mg/m3) | None | N.D. | inhalation, skin absorption, ingestion, skin and/or eye contact | irritation eyes, skin, mucous membrane; In Animals: liver, kidney damage; possible teratogenic effects | Eyes, skin, respiratory system, liver, reproductive system | Colorless liquid or crystalline solid (below 63°F) with an aromatic odor<br>BP: 416°F<br>Fl.Pt. = 222°F<br>LEL (302°F): 2.5%<br>UEL (302°F): 6.6%<br>Class IIIB Combustible Liquid: Fl.P. at or above 200°F.<br>Combustible Solid |
| 1,2-Dibromo-3-chloropropane | 96-12-8 | NA | Ca | TWA 0.001 ppm | Ca | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin, nose, throat; drowsiness; nausea, vomiting; pulmonary edema; liver, kidney injury; sterility; [potential occupational carcinogen] | Eyes, skin, respiratory system, central nervous system, liver, kidneys, spleen, reproductive system, digestive system | Dense yellow or amber liquid with a pungent odor at high concentrations. [pesticide] [Note: A solid below 43°F.]<br>BP: 384°F<br>Fl.Pt. = (oc) 170°F<br>LEL: NA<br>UEL: NA<br>Class IIIA Combustible Liquid: Fl.P. at or above 140°F and below 200°F. |
| 1,2-Dibromoethane | 106-93-4 | None listed<br>Skin | Ca TWA 0.045 ppm<br>C 0.13 ppm<br>[15-minute] | TWA 20 ppm<br>C 30 ppm<br>50 ppm<br>[5-minute maximum peak] | Ca [100 ppm] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin, respiratory system; dermatitis with vesiculation; liver, heart, spleen, kidney damage; reproductive effects; [potential occupational carcinogen] | Eyes, skin, respiratory system, liver, kidneys, reproductive system | Colorless liquid or solid (below 50°F) with a sweet odor. [fumigant]<br>BP: 268°F<br>Fl.Pt. = 50°F<br>LEL: NA<br>UEL: NA<br>Noncombustible Liquid |
| 1,2-Dichlorobenzene | 95-50-1 | TWA 25 ppm<br>STEL 50 ppm | C 50 ppm (300 mg/m3) | C 50 ppm (300 mg/m3) | 200 ppm | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, nose; liver, kidney damage; skin blisters | Eyes, skin, respiratory system, liver, kidneys | Colorless to pale-yellow liquid with a pleasant, aromatic odor. [herbicide]<br>BP: 357°F<br>Fl.Pt. = 1°F<br>LEL: 2.2%<br>UEL: 9.2%<br>Class IIIA Combustible Liquid: Fl.P. at or above 140°F and below 200°F. |
| 1,2-Dichloroethane | 107-06-2 | TWA 10 ppm | Ca TWA 1 ppm (4 mg/m3)<br>ST 2 ppm (8 mg/m3) | TWA 50 ppm<br>C 100 ppm<br>200 ppm [5-minute maximum peak in any 3 hours] | Ca [50 ppm] | Inhalation, ingestion, skin absorption, skin and/or eye contact | Irritation eyes, corneal opacity; central nervous system depression; nausea, vomiting; dermatitis; liver, kidney, cardiovascular system damage; [potential occupational carcinogen] | Eyes, skin, kidneys, liver, central nervous system, cardiovascular system | Colorless liquid with a pleasant, chloroform-like odor. [Note: Decomposes slowly, becomes acidic & darkens in color.]<br>BP: 182°F<br>Fl.Pt. = 56°F<br>LEL: 6.2%<br>UEL: 16%<br>Class IB Flammable Liquid Fl.P. below 73°F and BP at or above 100°F. |
| 1,2-Dichloropropane | 78-87-5 | TWA 10 ppm<br>Dermal Sensitizer (DSEN) | Ca | TWA 75 ppm (350 mg/m3) | Ca [400 ppm] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin, respiratory system; drowsiness, dizziness; liver, kidney damage; In Animals: central nervous system depression; [potential occupational carcinogen] | Eyes, skin, respiratory system, liver, kidneys, central nervous system | Colorless liquid with a chloroform-like odor. [pesticide]<br>BP: 206°F<br>Fl.Pt. = 60°F<br>LEL: 3.4%<br>UEL: 14.5%<br>Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F. |
| 1,3-Dichlorobenzene | 541-73-1 | https://cameochemicals.noaa.gov/chemical/8514 | | | | Inhalation, skin absorption, ingestion, skin and/or eye contact | INHALATION: Causes headache, drousiness, unsteadiness. Irritating to mucous membranes. EYES: Severe irritation. SKIN: Severe irritation. INGESTION: Irritation of gastric mucosa, nausea, vomiting, diarrhea, abdominal cramps and cyanosis. | | Colorless liquid. Sinks in water.<br>BP: 343°F<br>Fl.Pt. = 146°F<br>LEL: 2.02%<br>UEL: 9.2% |
| 1,4-Dichlorobenzene | 106-46-7 | TWA 10 ppm | Ca | TWA 75 ppm (450 mg/m3) | Ca [150 ppm] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Eye irritation, swelling periorbital (situated around the eye); profuse rhinitis; headache, anorexia, nausea, vomiting; weight loss, jaundice, cirrhosis; In Animals: liver, kidney injury; [potential occupational carcinogen] | Liver, respiratory system, eyes, kidneys, skin | Colorless or white crystalline solid with a mothball-like odor. [insecticide]<br>BP: 345°F<br>Fl.Pt. = 150°F<br>LEL: 2.5%<br>UEL: NA<br>Combustible Solid, but may take some effort to ignite. |

**Table 1. Toxicological, Physical, and Chemical Properties of Compounds Potentially Present at the Site.**

| Compound | CAS # | ACGIH TLV | NIOSH REL | OSHA PEL | IDLH | Routes of Exposure | Toxic Properties | Target Organs | Physical/Chemical Properties |
|---|---|---|---|---|---|---|---|---|---|
| 1,4-Dioxane | 123-91-1 | TWA 20 ppm [skin] | Ca C 1 ppm (3.6 mg/m3) [30-minute] | TWA 100 ppm (360 mg/m3) [skin] | Ca [500 ppm] | Inhalation, skin absorption, ingestion, skin and/or eye contact | irritation eyes, skin, nose, throat; drowsiness, headache; nausea, vomiting; liver damage; kidney failure; [potential occupational carcinogen] | Eyes, skin, respiratory system, liver, kidneys | Colorless liquid or solid (below 53°F) with a mild, ether-like odor.<br>BP: 214°F<br>Fl.Pt. = 55°F<br>LEL: 2.0%<br>UEL: 22%<br>Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F |
| 2-Butanone | 78-93-3 | TWA 200 ppm<br>STEL 300 ppm | TWA 200 ppm (590 mg/m3)<br>ST 300 ppm (885 mg/m3) | TWA 200 ppm (590 mg/m3) | 3000 ppm | inhalation, ingestion, skin and/or eye contact | irritation eyes, skin, nose; headache; dizziness; vomiting; dermatitis | Eyes, skin, respiratory system, central nervous system | Colorless liquid with a moderately sharp, fragrant, mint- or acetone-like odor<br>BP: 175°F<br>Fl.Pt. = 16°F<br>LEL (200°F): 1.4%<br>UEL (200°F): 11.4%<br>Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F |
| 2-Hexanone | 591-78-6 | TWA 5 ppm<br>STEL 10 ppm<br>[skin] | TWA 1 ppm (4 mg/m3) | TWA 100 ppm (410 mg/m3) | 1600 ppm | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, nose; peripheral neuropathy: lassitude (weakness, exhaustion), paresthesia; dermatitis; headache, drowsiness | Eyes, skin, respiratory system, central nervous system, peripheral nervous system | Colorless liquid with an acetone-like odor<br>BP: 262°F<br>Fl.Pt. = 77°F<br>LEL: NA<br>UEL: 8.0%<br>Class IC Flammable Liquid: Fl.P. at or above 73°F and below 100°F |
| 4-Methyl-2-pentanone | 108-10-1 | TWA 20 ppm<br>STEL 75 ppm | TWA 50 ppm (205 mg/m3)<br>ST 75 ppm (300 mg/m3) | TWA 100 ppm (410 mg/m3) | 500 ppm | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin, mucous membrane; headache, narcosis, coma; dermatitis; In Animals: liver, kidney damage | Eyes, skin, respiratory system, central nervous system, liver, kidneys | Colorless liquid with a pleasant odor<br>BP: 242°F<br>Fl.Pt. = 64°F<br>LEL (200°F): 1.2%<br>UEL (200°F): 8.0%<br>Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F |
| Acetone | 67-64-1 | TWA 250 ppm<br>STEL 500 ppm | TWA 250 ppm (590 mg/m^3) | TWA 1000 ppm (2400 mg/m^3) | 2500 ppm [10% LEL] | Inhalation, ingestion, skin and/or eye contact | irritation eyes, nose, throat; headache, dizziness, central nervous system depression; dermatitis | Eyes, skin, respiratory system, central nervous system | Colorless liquid with a fragrant, mint-like odor BP: 133°F<br>Fl.Pt. = 0°F<br>LEL: 12.8%<br>UEL: 2.5%<br>Class IB Flammable liquid: Fl.P. below 73°F and BP at or above 100°F. |
| Benzene | 71-43-2 | TWA 0.5 ppm<br>STEL 2.5 ppm | Ca TWA 0.1 ppm<br>ST 1 ppm | TWA 1 ppm<br>ST 5 ppm | Ca [500 ppm] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin, nose, respiratory system; dizziness; headache, nausea, staggered gait; anorexia, lassitude (weakness, exhaustion); dermatitis; bone marrow depression; [potential occupational carcinogen] | Eyes, skin, respiratory system, blood, central nervous system, bone marrow | Colorless to light yellow liquid with an aromatic odor [Note: Solid below 42 °F]<br>BP: 176°F<br>Fl.Pt. = 12°F<br>LEL: 1.2%<br>UEL: 7.8%<br>Class IB Flammable liquid. Fl.P. below 73°F and BP at or above 100°F. |
| Bromochloromethane | 74-97-5 | TWA 200 ppm | TWA 200 ppm (1050 mg/m3) | TWA 200 ppm (1050 mg/m3) | 2000 ppm | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin, throat; confusion, dizziness, central nervous system depression; pulmonary edema | Eyes, skin, respiratory system, liver, kidneys, central nervous system | Colorless to pale-yellow liquid with a chloroform-like odor. [Note: May be used as a fire extinguishing agent.]<br>BP: 155°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible Liquid |
| Bromodichloromethane | 75-27-4 | https://cameochemicals.noaa.gov/chemical/16064 | | | | Inhalation, ingestion, skin and/or eye contact | Symptoms of exposure to this compound may include irritation of the skin, eyes, mucous membranes and respiratory tract. It may also cause narcosis. Other symptoms may include nausea, dizziness and headache. | Liver and kidney damage. Central nervous system effects may also occur. | Clear colorless liquid<br>BP: 189°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Bromoform | 75-25-2 | TWA 0.5 ppm | TWA 0.5 ppm (5 mg/m3) [skin] | TWA 0.5 ppm (5 mg/m3) [skin] | 850 ppm | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin, respiratory system; central nervous system depression; liver, kidney damage | Eyes, skin, respiratory system, central nervous system, liver, kidneys | Colorless to yellow liquid with a chloroform-like odor. [Note: A solid below 47°F.]<br>BP: 301°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible Liquid |
| Bromomethane | 74-83-9 | TWA 1 ppm [skin] | Ca | C 20 ppm (80 mg/m3) [skin] | Ca [250 ppm] | Inhalation, skin absorption (liquid), skin and/or eye contact (liquid) | Irritation eyes, skin, respiratory system; muscle weak, incoordination, visual disturbance, dizziness; nausea, vomiting, headache; malaise (vague feeling of discomfort); hand tremor; convulsions; dyspnea (breathing difficulty); skin vesiculation; liquid: frostbite; [potential occupational carcinogen] | Eyes, skin, respiratory system, central nervous system | Colorless gas with a chloroform-like odor at high concentrations. [Note: A liquid below 38°F. Shipped as a liquefied compressed gas.]<br>BP: 38°F<br>Fl.Pt. = NA (Gas)<br>LEL: 10%<br>UEL: 16.0%<br>Flammable Gas, but only in presence of a high energy ignition source. |

**Table 1. Toxicological, Physical, and Chemical Properties of Compounds Potentially Present at the Site.**

| Compound | CAS # | ACGIH TLV | NIOSH REL | OSHA PEL | IDLH | Routes of Exposure | Toxic Properties | Target Organs | Physical/Chemical Properties |
|---|---|---|---|---|---|---|---|---|---|
| Carbon disulfide | 75-15-0 | TWA 1 ppm [skin] | TWA 1 ppm (3 mg/m3)<br>ST 10 ppm (30 mg/m3) [skin] | TWA 20 ppm<br>C 30 ppm<br>100 ppm<br>(30-minute maximum peak) | 500 ppm | Inhalation, skin absorption, ingestion, skin and/or eye contact | Dizziness, headache, poor sleep, lassitude (weakness, exhaustion), anxiety, anorexia, weight loss; psychosis; polyneuropathy; Parkinson-like syndrome; ocular changes; coronary heart disease; gastritis; kidney, liver injury; eye, skin burns; dermatitis; reproductive effects | Central nervous system, peripheral nervous system, cardiovascular system, eyes, kidneys, liver, skin, reproductive system | Colorless to faint-yellow liquid with a sweet ether-like odor. [Note: Reagent grades are foul smelling.]<br>BP: 116°F<br>Fl.Pt. = -22°F<br>LEL: 1.3%<br>UEL: 50.0%<br>Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F. |
| Carbon tetrachloride | 56-23-5 | TWA 5 ppm<br>STEL 10 ppm<br>[skin] | Ca ST 2 ppm (12.6 mg/m3)<br>[60-minute] | TWA 10 ppm<br>C 25 ppm<br>200 ppm (5-minute maximum peak in any 4 hours) | Ca [200 ppm] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin; central nervous system depression; nausea, vomiting; liver, kidney injury; drowsiness, dizziness, incoordination; [potential occupational carcinogen] | Central nervous system, eyes, lungs, liver, kidneys, skin | Colorless liquid with a characteristic ether-like odor<br>BP: 170°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible Liquid |
| Chlorobenzene | 108-90-7 | TWA 10 ppm | NA | TWA 75 ppm (350 mg/m3) | 1000 ppm | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin, nose; drowsiness, incoordination; central nervous system depression; In Animals: liver, lung, kidney injury | Eyes, skin, respiratory system, central nervous system, liver | Colorless liquid with an almond-like odor<br>BP: 270°F<br>Fl.Pt. = 82°F<br>LEL: 1.3%<br>UEL: 9.6%<br>Class IC Flammable Liquid: Fl.P. at or above 73°F and below 100°F. |
| Chloroethane | 75-00-3 | TWA 100 ppm [skin] | Handle with caution in the workplace | TWA 1000 ppm (2600 mg/m3) | 3800 ppm [10%LEL] | Inhalation, skin absorption (liquid), ingestion (liquid), skin and/or eye contact | Incoordination, inebriation; abdominal cramps; cardiac arrhythmias, cardiac arrest; liver, kidney damage | Liver, kidneys, respiratory system, cardiovascular system, central nervous system | Colorless gas or liquid (below 54°F) with a pungent, ether-like odor. [Note: Shipped as a liquefied compressed gas.]<br>BP: 54°F<br>Fl.Pt. = NA (gas), -58°F (liquid)<br>LEL: 3.8%<br>UEL: 15.4%<br>Flammable Gas |
| Chloroform | 67-66-3 | TWA 10 ppm | Ca ST 2 ppm (9.78 mg/m3)<br>[60-minute] | C 50 ppm (240 mg/m3) | Ca [500 ppm] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin; dizziness, mental dullness, nausea, confusion; headache, lassitude (weakness, exhaustion); anesthesia; enlarged liver; [potential occupational carcinogen] | Liver, kidneys, heart, eyes, skin, central nervous system | Colorless liquid with a pleasant odor<br>BP: 143°F<br>Fl.Pt. = -82°F<br>LEL: NA<br>UEL: NA<br>Noncombustible Liquid |
| Chloromethane | 74-87-3 | TWA 50 ppm<br>STEL 100 ppm | Ca | TWA 100 ppm<br>C 200 ppm<br>300 ppm (5-minute maximum peak in any 3 hours) | Ca [2000 ppm] | Inhalation, skin and/or eye contact (liquid) | Dizziness, nausea, vomiting; visual disturbance, stagger, slurred speech, convulsions, coma; liver, kidney damage; liquid: frostbite; reproductive, teratogenic effects; [potential occupational carcinogen] | Central nervous system, liver, kidneys, reproductive system | Colorless gas with a faint, sweet odor which is not noticeable at dangerous concentrations. [Note: Shipped as a liquefied compressed gas.]<br>BP: -12°F<br>Fl.Pt. = NA (Gas)<br>LEL: 8.1%<br>UEL: 17.4%<br>Flammable Gas |
| cis-1,2-Dichloroethene | 156-59-2 | TWA 200 ppm (All isomers) | TWA 200 ppm (790 mg/m3) | TWA 200 ppm (790 mg/m3) | 1000 ppm | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, respiratory system; central nervous system depression | Eyes, respiratory system, central nervous system | Colorless liquid (usually a mixture of the cis & trans isomers) with a slightly acrid, chloroform-like odor.<br>BP: 118-140°F<br>Fl.Pt. = 36-39°F<br>LEL: 5.6%<br>UEL: 12.8%<br>Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F |
| cis-1,3-Dichloropropene | 10061-01-5 | https://cameochemicals.noaa.gov/chemical/20168 | | | | Inhalation, ingestion, skin and/or eye contact | Symptoms of exposure to this compound may include local irritation of the eyes skin and respiratory tract; dermatitis, gasping, coughing, substernal pain, extreme respiratory distress, lacrimation, central nervous system depression, skin irritation, acute gastrointestinal distress with pulmonary congestion and edema. It also may cause injury to the liver, kidneys and heart. | Skin, eyes, mucous membranes, liver, kidney, heart | Colorless to amber liquid with a sweetish odor.<br>BP: 219.7°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Cyclohexane | 110-82-7 | TWA 100 ppm | TWA 300 ppm (1050 mg/m3) | TWA 300 ppm (1050 mg/m3) | 1300 ppm [10%LEL] | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin, respiratory system; drowsiness; dermatitis; narcosis, coma | Eyes, skin, respiratory system, central nervous system | Colorless liquid with a sweet, chloroform-like odor. [Note: A solid below 44°F.]<br>BP: 177°F<br>Fl.Pt. = 0°F<br>LEL: 1.3%<br>UEL: 8.0%<br>Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F. |
| Dibromochloromethane | 124-48-1 | https://cameochemicals.noaa.gov/chemical/16183 | | | | Inhalation, ingestion, skin and/or eye contact | Symptoms of exposure to this compound may include irritation of the skin, eyes, mucous membranes and upper respiratory tract. It may also cause fatigue. Other symptoms may include central nervous system effects, lung and cornea irritation and liver and kidney damage. Prolonged exposure can cause nausea, dizziness, headache and narcosis. | Skin, eyes, mucous membranes, upper respiratory tract | Clear colorless to yellow-orange liquid<br>BP: 246-248°F<br>Fl.Pt. = Greater than 200°F<br>LEL: NA<br>UEL: NA |

**Table 1. Toxicological, Physical, and Chemical Properties of Compounds Potentially Present at the Site.**

| Compound | CAS # | ACGIH TLV | NIOSH REL | OSHA PEL | IDLH | Routes of Exposure | Toxic Properties | Target Organs | Physical/Chemical Properties |
|---|---|---|---|---|---|---|---|---|---|
| Dichlorodifluoromethane | 75-71-8 | TWA 1000 ppm | TWA 1000 ppm (4950 mg/m3) | TWA 1000 ppm (4950 mg/m3) | 15,000 ppm | Inhalation, skin and/or eye contact (liquid) | Dizziness, tremor, asphyxia, unconsciousness, cardiac arrhythmias, cardiac arrest; liquid: frostbite | Cardiovascular system, peripheral nervous system | Colorless gas with an ether-like odor at extremely high concentrations. [Note: Shipped as a liquefied compressed gas.]<br>BP: -22°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Nonflammable Gas |
| Ethyl benzene | 100-41-4 | TWA 20 ppm | TWA 100 ppm (435 mg/m$^3$)<br>ST 125 ppm (545 mg/m^3) | TWA 100 ppm (435 mg/m$^3$) | 800 ppm [10%LEL] | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin, mucous membrane; headache; dermatitis; narcosis, coma | Eyes, skin, respiratory system, central nervous system | Colorless liquid with an aromatic odor.<br>BP: 277°F<br>Fl.Pt. = 55°F<br>LEL: 0.8%<br>UEL: 6.7%<br>Class IB Flammable Liquid below 73°F and BP at or above 100°F |
| Isopropyl benzene | 98-82-8 | TWA 5 ppm | TWA 50 ppm (245 mg/m3) [skin] | TWA 50 ppm (245 mg/m3) [skin] | 900 ppm [10%LEL] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin, mucous membrane; dermatitis; headache, narcosis, coma | Eyes, skin, respiratory system, central nervous system | Colorless liquid with a sharp, penetrating, aromatic odor<br>BP: 306°F<br>Fl.Pt. = 96°F<br>LEL: 0.9%<br>UEL: 6.5%<br>Class IC Flammable Liquid: Fl.P. at or above 73°F and below 100°F |
| Methyl Acetate | 79-20-9 | TWA 200 ppm<br>STEL 250 ppm | TWA 200 ppm (610 mg/m3)<br>ST 250 ppm (760 mg/m3) | TWA 200 ppm (610 mg/m3) | 3100 ppm [10%LEL] | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin, nose, throat; headache, drowsiness; optic nerve atrophy; chest tightness; In Animals: narcosis | Eyes, skin, respiratory system, central nervous system | Colorless liquid with a fragrant, fruity odor<br>BP: 135°F<br>Fl.Pt. = 14°F<br>LEL: 3.1%<br>UEL: 16%<br>Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F. |
| Methylcyclohexane | 108-87-2 | TWA 400 ppm | TWA 400 ppm (1600 mg/m3) | TWA 500 ppm (2000 mg/m3) | 1200 ppm [LEL] | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin, nose, throat; dizziness, drowsiness; In Animals: narcosis | Eyes, skin, respiratory system, central nervous system | Colorless liquid with a faint, benzene-like odor<br>BP: 214°F<br>Fl.Pt. = 25°F<br>LEL: 1.2%<br>UEL: 6.7%<br>Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F. |
| Methylene chloride | 75-09-2 | TWA 50 ppm [skin]<br>STEL 100 ppm | Ca | [1910.1052] TWA 25 ppm<br>ST 125 ppm | Ca [2300 ppm] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin; lassitude (weakness, exhaustion), drowsiness, dizziness; numb, tingle limbs; nausea; [potential occupational carcinogen] | Eyes, skin, cardiovascular system, central nervous system | Colorless liquid with a chloroform-like odor. [Note: A gas above 104°F.]<br>BP: 104°F<br>Fl.Pt. = NA<br>LEL: 13%<br>UEL: 23%<br>Combustible Liquid |
| Methyl-t-butyl ether | 1634-04-4 | TWA 50 ppm | NA | NA | NA | Inhalation, skin absorption, ingestion, skin and/or eye contact | May cause dizziness or suffocation. Contact may irritate or burn eyes or skin. May be harmful if swallowed. | Eyes, skin | A colorless liquid with a distinctive anesthetic-like odor.<br>BP: 131°F<br>Fl.Pt. = -14°F<br>LEL: NA<br>UEL: NA |
| o-Xylene | 95-47-6 | TWA 20 ppm<br><br>(All isomers) | TWA 100 ppm (435 mg/m3)<br>ST 150 ppm (655 mg/m3) | TWA 100 ppm (435 mg/m3) | 900 ppm | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin, nose, throat; dizziness, excitement, drowsiness, incoordination, staggering gait; corneal vacuolization; anorexia, nausea, vomiting, abdominal pain; dermatitis | Eyes, skin, respiratory system, central nervous system, gastrointestinal tract, blood, liver, kidneys | Colorless liquid with an aromatic odor<br>BP: 292°F<br>Fl.Pt. = 90°F<br>LEL: 0.9%<br>UEL: 6.7%<br>Class IC Flammable Liquid: Fl.P. at or above 73°F and below 100°F |
| Styrene | 100-42-5 | TWA 10 ppm<br>STEL 20 ppm<br>OTO (ototoxicant) | TWA 50 ppm (215 mg/m3)<br>ST 100 ppm (425 mg/m3) | TWA 100 ppm<br>C 200 ppm<br>600 ppm (5-minute maximum peak in any 3 hours) | 700 ppm | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, nose, respiratory system; headache, lassitude (weakness, exhaustion), dizziness, confusion, malaise (vague feeling of discomfort), drowsiness, unsteady gait; narcosis; defatting dermatitis; possible liver injury; reproductive effects | Eyes, skin, respiratory system, central nervous system, liver, reproductive system | Colorless to yellow, oily liquid with a sweet, floral odor<br>BP: 293°F<br>Fl.Pt. = 88°F<br>LEL: 0.9%<br>UEL: 6.8%<br>Class IC Flammable Liquid: Fl.P. at or above 73°F and below 100°F |
| Tetrachloroethene | 127-18-4 | TWA 25 ppm<br>STEL 100 ppm | Ca<br>Minimize workplace exposure concentrations | TWA 100 ppm<br>C 200 ppm (for 5 minutes in any 3-hour period), with a maximum peak of 300 ppm | Ca [150 ppm] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin, nose, throat, respiratory system; nausea; flush face, neck; dizziness, incoordination; headache, drowsiness; skin erythema (skin redness); liver damage; [potential occupational carcinogen] | Eyes, skin, respiratory system, liver, kidneys, central nervous system | Colorless liquid with a mild, chloroform-like odor<br>BP: 250°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible Liquid, but decomposes in a fire to hydrogen chloride and phosgene |

**Table 1. Toxicological, Physical, and Chemical Properties of Compounds Potentially Present at the Site.**

| Compound | CAS # | ACGIH TLV | NIOSH REL | OSHA PEL | IDLH | Routes of Exposure | Toxic Properties | Target Organs | Physical/Chemical Properties |
|---|---|---|---|---|---|---|---|---|---|
| Sodium Hydroxide | 1310-73-2 | Ceiling 2 mg/m$^3$ | C 2 mg/m$^3$ | TWA 2 mg/m$^3$ | 10 mg/m$^3$ | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin, mucous membrane; pneumonitis; eye, skin burns; temporary loss of hair | Eyes, skin, respiratory system | Colorless to white, odorless solid (flakes, beads, granular form).<br>BP: 2534°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible Solid, but when in contact with water may generate sufficient heat to ignite combustible materials. |
| Sulfuric Acid | 7664-93-9 | TWA 0.2 mg/m$^3$ (as thoracic particulate mass) | TWA 1 mg/m$^3$ | TWA 1 mg/m$^3$ | 15 mg/m$^3$ | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin, nose, throat; pulmonary edema, bronchitis; emphysema; conjunctivitis; stomatis; dental erosion; eye, skin burns; dermatitis | Eyes, skin, respiratory system, teeth | Colorless to dark-brown, oily, odorless liquid.[Note: Pure compound is a solid below 51°F. Often used in an aqueous solution.]<br>BP = 554°F<br>Fl.Pt. = NA<br>LEL = NA<br>UEL = NA<br>Noncombustible Liquid, but capable of igniting finely divided combustible materials. |
| trans-1,2-Dichloroethene | 156-60-5 | 200 ppm (All isomers) | TWA 200 ppm (790 mg/m3) | TWA 200 ppm (790 mg/m3) | 1000 ppm | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, respiratory system; central nervous system depression | Eyes, respiratory system, central nervous system | Colorless liquid (usually a mixture of the cis & trans isomers) with a slightly acrid, chloroform-like odor.<br>BP: 118-140°F<br>Fl.P: 36-39°F<br>LEL: 5.6%<br>UEL: 12.8%<br>Class IB Flammable Liquid Fl.P. below 73°F and BP at or above 100°F. |
| trans-1,3-Dichloropropene | 10061-02-6 | https://cameochemicals.noaa.gov/chemical/18110 | | | | Inhalation, ingestion, skin and/or eye contact | Symptoms of exposure to this compound may include local irritation of the eyes skin and respiratory tract, dermatitis, gasping, coughing, substernal pain, extreme respiratory distress, lacrimation, central nervous system depression, acute gastrointestinal distress with pulmonary congestion and edema. It may also cause injury to the liver, kidneys and heart | Skin, eyes, mucous membranes, liver, kidney, heart | A clear colorless liquid with chloroform odor<br>BP: 234°F<br>Fl.P: NA<br>LEL: NA<br>UEL: NA |
| Toluene | 108-88-3 | TWA 20 ppm<br>(ototoxicant) | TWA 100 ppm (375 mg/m$^3$)<br>ST 150 ppm (560 mg/m$^3$) | TWA 200 ppm<br>C 300 ppm<br>500 ppm (10-minute maximum peak) | 500 ppm | Inhalation, skin absorption, ingestion, skin and/or eye contact | irritation eyes, nose; lassitude (weakness, exhaustion), confusion, euphoria, dizziness, headache; dilated pupils, lacrimation (discharge of tears); anxiety, muscle fatigue, insomnia; paresthesia; dermatitis; liver, kidney damage | Eyes, skin, respiratory system, central nervous system, liver, kidneys | Colorless liquid with a sweet, pungent, benzene-like odor.<br>BP: 232°F<br>Fl.P: 40°F<br>LEL: 1.1%<br>UEL: 7.1%<br>Class IB Flammable Liquid Fl.P. below 73°F and BP at or above 100°F. |
| Trichloroethene (TCE) | 79-01-6 | TWA 10 ppm<br>STEL 25 ppm | Ca | TWA 100 ppm<br>C 200 ppm<br>300 ppm (5-minute maximum peak in any 2 hours) | Ca [1000 ppm] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin; headache, visual disturbance, lassitude (weakness, exhaustion), dizziness, tremor, drowsiness, nausea, vomiting; dermatitis; cardiac arrhythmias, paresthesia; liver injury; [potential occupational carcinogen] | Eyes, skin, respiratory system, heart, liver, kidneys, central nervous system | Colorless liquid (unless dyed blue) with a chloroform-like odor.<br>BP: 189°F<br>Fl.Pt. = NA<br>LEL(77°F): 8.0%<br>UEL(77°F): 10.5%<br>Combustible Liquid, but burns with difficulty. |
| Trichlorofluoromethane | 75-69-4 | STEL C 1000 ppm | C 1000 ppm (5600 mg/m3) | TWA 1000 ppm (5600 mg/m3) | 2000 ppm | Inhalation, ingestion, skin and/or eye contact | Incoordination, tremor; dermatitis; cardiac arrhythmias, cardiac arrest; asphyxia; liquid: frostbite | Skin, respiratory system, cardiovascular system | Colorless to water-white, nearly odorless liquid or gas (above 75°F)<br>BP: 75°F<br>Fl.P: NA<br>LEL: NA<br>UEL: NA<br>Noncombustible Liquid Nonflammable Gas |
| Vinyl Chloride (chloroethylene) | 75-01-4 | TWA 1 ppm | Ca | TWA 1 ppm<br>C 5 ppm [15-minute] | Ca (ND) | Inhalation, skin and/or eye contact (liquid) | Lassitude (weakness, exhaustion); abdominal pain, gastrointestinal bleeding; enlarged liver; pallor or cyanosis of extremities; liquid: frostbite; [potential occupational carcinogen] | Liver, central nervous system, blood, respiratory system, lymphatic system | Colorless gas or liquid (below 7°F) with a pleasant odor at high concentrations.[Note: Shipped as a liquefied compressed gas.]<br>BP: 7°F<br>Fl.Pt. = NA (Gas)<br>LEL: 3.6%<br>UEL: 33.0%<br>Flammable Gas |
| Xylene (m, o & p isomers) | 108-38-3,<br>95-47-6,<br>106-42-3 | TWA 20 ppm | NA | NA | NA | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin, nose, throat; dizziness, excitement, drowsiness, incoordination, staggering gait; corneal vacuolization; anorexia, nausea, vomiting, abdominal pain; dermatitis | Eyes, skin, respiratory system, central nervous system, gastrointestinal tract, blood, liver, kidneys | Colorless liquid with an aromatic odor<br>BP: 282°F, 292°F, 281°F<br>Fl. Pt. 82°F, 90°F, 81°F<br>LEL: 1.1%, 0.9%, 1.1%<br>UEL: 7.0%, 6.7%, 7.0%<br>Class IC Flammable Liquid at or above 73°F and below 100°F. |
| Zinc Oxide (dust) | 7440-66-6 | TWA 2 mg/m3 (repsirable)<br>STEL 10 mg/m$^3$ (respirable) | TWA 5 mg/m^3<br>C 15 mg/m^3 | TWA 15 mg/m^3 (total dust)<br>TWA 5 mg/m^3 (resp dust)<br>TWA 5 mg/m^3 (fume) | 500 mg/m^3 | 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 | Respiratory system | White, odorless solid.<br>BP: NA<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible Solid |

**Table 1. Toxicological, Physical, and Chemical Properties of Compounds Potentially Present at the Site.**

| Compound | CAS # | ACGIH TLV | NIOSH REL | OSHA PEL | IDLH | Routes of Exposure | Toxic Properties | Target Organs | Physical/Chemical Properties |
|---|---|---|---|---|---|---|---|---|---|
| **SEMI-VOLATILE ORGANIC COMPOUNDS (SVOCs)** | | | | | | | | | |
| 2-Chloronaphthalene | 91-58-7 | https://cameochemicals.noaa.gov/chemical/16185 | | | | Inhalation, ingestion, skin and/or eye contact | Chloracne, cysts, headache, fatigue, vertigo, anorexia and jaundice | | Monoclinic plates or off-white crystalline powder<br>BP: NA<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| 2-Methylnaphthalene | 91-57-6 | TWA 0.5 ppm<br>TLV-SL 3 mg/100 cm2<br>[skin] | https://cameochemicals.noaa.gov/chemical/20668 | | | Inhalation, ingestion, skin and/or eye contact | Headaches, nausea, vomiting, diarrhea, anemia, jaundice, euphoria, dermatitis, visual disturbances, convulsions and comatose | Skin, eyes, mucous membranes and upper respiratory tract | White crystalline solid<br>Combustible solid<br>BP: 466-468 ° F<br>Fl.Pt. = 208 ° F<br>LEL: NA<br>UEL: NA |
| Acenaphthene | 83-32-9 | https://cameochemicals.noaa.gov/chemical/10358 | | | | Inhalation, ingestion, skin and/or eye contact | Irritation of the skin, eyes, mucous membranes and upper respiratory tract, vomiting | Skin, eyes, mucous membranes and upper respiratory tract | White needles<br>BP: 534 ° F<br>Fl.Pt. = NA<br>LEL: 0.6%<br>UEL: NA |
| Acenaphthylene | 208-96-8 | https://cameochemicals.noaa.gov/chemical/16157 | | | | Inhalation, ingestion, skin and/or eye contact | | | Colorless crystalline solid<br>BP: 509 to 527 ° F at 760 mm Hg<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Anthracene (as coal tar pitch volatiles) | 120-12-7 | TWA 0.2 mg/m3 (as Benzene solubles) | Ca TWA 0.1 mg/m3 (cyclohexane-extractable fraction) | TWA 0.2 mg/m3 (benzene-soluble fraction)<br>[1910.1002] | Ca [80 mg/m3] | Inhalation, skin and/or eye contact | Dermatitis, bronchitis, [potential occupational carcinogen] | Respiratory system, skin, bladder, kidneys | Black or dark-brown amorphous residue<br>BP: NA<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Combustible Solids |
| Benzo[a]anthracene | 56-55-3 | https://cameochemicals.noaa.gov/chemical/16171 | | | | Inhalation, injestion, skin and/or eye contact | | | Colorless leaflets or plates or coarse gold powder with a greenish-yellow fluorescence. May reasonably be expected to be a carcinogen.<br>BP: 815° F at 760 mm Hg<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Benzo[a]pyrene (as coal tar pitch volatiles) | 50-32-8 | TWA 0.2 mg/m3 (as Benzene solubles) | Ca TWA 0.1 mg/m3 (cyclohexane-extractable fraction) | TWA 0.2 mg/m3 (benzene-soluble fraction)<br>[1910.1002] | Ca [80 mg/m3] | Inhalation, skin and/or eye contact | Dermatitis, bronchitis, [potential occupational carcinogen] | Respiratory system, skin, bladder, kidneys | Black or dark-brown amorphous residue<br>BP: NA<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Combustible Solids |
| Benzo[b]fluoranthene | 205-99-2 | None listed | https://cameochemicals.noaa.gov/chemical/16172 | | | Inhalation, injestion, skin and/or eye contact | | | Needles or yellow fluffy powder<br>BP: NA<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Benzo[g,h,i]perylene | 191-24-2 | https://cameochemicals.noaa.gov/chemical/16174 | | | | Inhalation, injestion, skin and/or eye contact | Inhalation of material may be harmful. Contact may cause burns to skin and eyes. Inhalation of Asbestos dust may have a damaging effect on the lungs. Fire may produce irritating, corrosive and/or toxic gases. Some liquids produce vapors that may cause dizziness or suffocation. Runoff from fire control may cause pollution. | Lungs, skin, eyes | Colorless to white crystalline solid. Water insoluble.<br>BP: NA<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Benzo[k]fluoranthene | 207-08-9 | https://cameochemicals.noaa.gov/chemical/16173 | | | | Inhalation, injestion, skin and/or eye contact | When heated to decomposition this compound emits acrid smoke and irritating fumes. | | Pale yellow needles or yellow crystalline solid<br>BP: 896° F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Chrysene (as coal tar pitch volatiles | 218-01-9 | TWA 0.2 mg/m3 (as Benzene solubles) | Ca TWA 0.1 mg/m3 (cyclohexane-extractable fraction) | TWA 0.2 mg/m3 (benzene-soluble fraction)<br>[1910.1002] | Ca [80 mg/m3] | Inhalation, skin and/or eye contact | Dermatitis, bronchitis, [potential occupational carcinogen] | Respiratory system, skin, bladder, kidneys | Black or dark-brown amorphous residue<br>BP: NA<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Combustible Solids |
| Dibenzo(a,h)anthracene | 53-70-3 | https://cameochemicals.noaa.gov/chemical/16192 | | | | Inhalation, injestion, skin and/or eye contact | Symptoms of exposure to this compound may include irritation. This compound is harmful if swallowed or inhaled. It may cause irritation. When heated to decomposition it emits acrid smoke, irritating fumes and toxic fumes of carbon monoxide and carbon dioxide. | Lungs | White crystals or pale yellow solid. Sublimes<br>BP: 975° F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |

**Table 1. Toxicological, Physical, and Chemical Properties of Compounds Potentially Present at the Site.**

| Compound | CAS # | ACGIH TLV | NIOSH REL | OSHA PEL | IDLH | Routes of Exposure | Toxic Properties | Target Organs | Physical/Chemical Properties |
|---|---|---|---|---|---|---|---|---|---|
| Fluoranthene | 206-44-0 | https://cameochemicals.noaa.gov/chemical/16213 | | | | Inhalation, injestion, skin and/or eye contact | When heated to decomposition this compound emits acrid smoke and fumes. | | Light yellow fine crystals<br>BP: 482° F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Fluorene | 86-73-7 | https://cameochemicals.noaa.gov/chemical/16214 | | | | Inhalation, injestion, skin and/or eye contact | | | White leaflets. Sublimes easily under a vacuum. Fluorescent when impure.<br>BP: 563° F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Indeno[1,2,3-cd]pyrene | 193-39-5 | https://cameochemicals.noaa.gov/chemical/16218 | | | | Inhalation, injestion, skin and/or eye contact | | | Yellow crystals<br>BP: 997° F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Naphthalene | 91-20-3 | TWA 10 ppm [skin] | TWA 10 ppm (50 mg/m3)<br>ST 15 ppm (75 mg/m3) | TWA 10 ppm (50 mg/m3) | 250 ppm | 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 | Eyes, skin, blood, liver, kidneys, central nervous system | Colorless to brown solid with an odor of mothballs. [Note: Shipped as a molten solid.]<br>BP: 424°F<br>Fl.P: 174°F<br>LEL: 0.9%<br>UEL: 5.9%<br>Combustible Solid, but will take some effort to ignite |
| Phenanthrene | 85-01-8 | https://cameochemicals.noaa.gov/chemical/16236 | | | | Inhalation, injestion, skin and/or eye contact | Symptoms following exposure to this compound may include skin sensitization, dermatitis, bronchitis, cough, dyspnea, respiratory neoplasm, kidney neoplasm, skin irritation, and respiratory irritation. | Skin, respiratory tract | Colorless monoclinic crystals with a faint aromatic odor. Solutions exhibit a blue fluorescence.<br>BP: 642° F<br>Fl.Pt. = 340° F<br>LEL: NA<br>UEL: NA |
| Pyrene (see coal tar pitch volatiles) | 129-00-0 | TWA 0.2 mg/m3 (as Benzene solubles) | Ca TWA 0.1 mg/m3 (cyclohexane-extractable fraction) | TWA 0.2 mg/m3 (benzene-soluble fraction) [1910.1002] | Ca [80 mg/m3] | Inhalation, skin and/or eye contact | Dermatitis, bronchitis, [potential occupational carcinogen] | Respiratory system, skin, bladder, kidneys | Black or dark-brown amorphous residue<br>BP: NA<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Combustible Solids |
| **METALS** | | | | | | | | | |
| Aluminum | 7429-90-5 | TWA 1 mg/m3 | TWA 10 mg/m^3 (total)<br>TWA 5 mg/m^3 (resp) | TWA 15 mg/m^3 (total)<br>TWA 5 mg/m^3 (resp) | N.D. | Inhalation, skin and/or eye contact | Irritation eyes, skin, respiratory system | Eyes, skin, respiratory system | Silvery-white, malleable, ductile, odorless metal<br>BP: 4221°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Combustible Solid, finely divided dust is easily ignited; may cause explosions. |
| Antimony | 7440-36-0 | TWA 0.5 mg/m3 (as Sb) | TWA 0.5 mg/m3<br>[*Note: The REL also applies to other antimony compounds (as Sb).] | TWA 0.5 mg/m3<br>[*Note: The PEL also applies to other antimony compounds (as Sb).] | 50 mg/m3 (as Sb) | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin, nose, throat, mouth; cough; dizziness; headache; nausea, vomiting, diarrhea; stomach cramps; insomnia; anorexia; unable to smell properly | Eyes, skin, respiratory system, cardiovascular system | Silver-white, lustrous, hard, brittle solid; scale-like crystals; or a dark-gray, lustrous powder<br>BP: 2975°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible Solid in bulk form, but a moderate explosion hazard in the form of dust when exposed to flame. |
| Arsenic | 7440-38-2 | TWA 0.01 mg/m3 | Ca C 0.002 mg/m3 [15-minute] | [1910.1018] TWA 0.010 mg/m3 | Ca [5 mg/m3 (as As)] | Inhalation, skin absorption, skin and/or eye contact, ingestion | Ulceration of nasal septum, dermatitis, gastrointestinal disturbances, peripheral neuropathy, resp irritation, hyperpigmentation of skin, [potential occupational carcinogen] | Liver, kidneys, skin, lungs, lymphatic system | Metal: Silver-gray or tin-white, brittle, odorless solid<br>BP: Sublimes<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Metal: Noncombustible Solid in bulk form, but a slight explosion hazard in the form of dust when exposed to flame |
| Barium | 7440-39-3 | TWA 0.5 mg/m3 | 0.5 mg Ba/m3 TWA | 0.5 mg Ba/m3 TWA | 50 mg Ba/m3 | Inhalation, ingestion, skin and/or eye contact | Inhalation or contact with vapors, substance or decomposition products may cause severe injury or death. Contact may cause burns to skin, eyes, and mucous membranes. May be toxic by ingestion, inhalation and skin absorption. Used to make other chemicals. | Lungs, skin, eyes, and mucous membrane | A silver to white metallic solid<br>BP: 1337°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Beryllium | 7440-41-7 | TWA 0.00005 mg/m$^3$ | Ca C 0.0005 mg/m^3 | TWA 0.002 mg/m^3<br>C 0.005 mg/m$^3$<br>0.025 mg/m3<br>[30-minute maximum peak] | Ca [4 mg/m^3 (as Be)] | Inhalation, skin and/or eye contact | Berylliosis (chronic exposure): anorexia, weight loss, lassitude (weakness, exhaustion), chest pain, cough, clubbing of fingers, cyanosis, pulmonary insufficiency; irritation eyes; dermatitis; [potential occupational carcinogen] | Eyes, skin, respiratory system | Hard, brittle, gray-white solid<br>BP: 4532°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible Solid in bulk form, but a slight explosion hazard in the form of a powder or dust. |

**Table 1. Toxicological, Physical, and Chemical Properties of Compounds Potentially Present at the Site.**

| Compound | CAS # | ACGIH TLV | NIOSH REL | OSHA PEL | IDLH | Routes of Exposure | Toxic Properties | Target Organs | Physical/Chemical Properties |
|---|---|---|---|---|---|---|---|---|---|
| Cadmium | 7440-43-9 | TWA 0.01 mg/m$^3$ total dust<br>TWA 0.002 mg/m^3 (as Cd) respirable fraction | Ca | TWA 0.005 mg/m$^3$ | Ca [9 mg/m3 (as Cd)] | Inhalation, ingestion | Pulmonary edema, dyspnea (breathing difficulty), cough, chest tightness, substernal (occurring beneath the sternum) pain; headache; chills, muscle aches; nausea, vomiting, diarrhea; anosmia (loss of the sense of smell), emphysema, proteinuria, mild anemia; [potential occupational carcinogen] | Respiratory system, kidneys, prostate, blood | Silver-white/blue tinged lustrous, odorless solid.<br>BP: 1409°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible - will burn in powder form |
| Calcium | 7440-70-2 | https://cameochemicals.noaa.gov/chemical/309 | | | | Inhalation, ingestion, skin and/or eye contact | Contact with eyes or skin produces caustic burns. | Eyes, skin | A silvery, soft metal that turns grayish white on exposure to air.<br>BP: 2714°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Chromium | 7440-47-3 | TWA 0.5 mg/m$^3$ (metal)<br>TWA 0.003 mg/m$^3$ (water-soluble Cr III compounds)<br>TWA 0.0002 mg/m$^3$ (water-soluble Cr VI compounds)<br>STEL 0.0005 mg/m3 (water-soluble Cr VI compounds) | TWA 0.5 mg/m$^3$ | TWA 1 mg/m$^3$ | 250 mg/m$^3$ (as Cr) | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin; lung fibrosis (histologic) | Eyes, skin, respiratory system | Blue-white to steel-gray, lustrous, brittle, hard, odorless solid.<br>BP: 4788°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible - will burn in dust form if heated in a flame |
| Cobalt | 7440-48-4 | TWA 0.02 mg/m3 [DSEN] [RSEN] | TWA 0.05 mg/m3 | TWA 0.1 mg/m3 | 20 mg/m3 (as Co) | Inhalation, ingestion, skin and/or eye contact | Cough, dyspnea (breathing difficulty), wheezing, decreased pulmonary function; weight loss; dermatitis; diffuse nodular fibrosis; resp hypersensitivity, asthma | Skin, respiratory system | Odorless, silver-gray to black solid<br>BP: 5612°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible Solid in bulk form, but finely divided dust will burn at high temperatures. |
| Copper | 7440-50-8 | TWA 0.2 mg/m$^3$ (fume)<br>TWA 1 mg/m3 (dusts and mists) | TWA 1 mg/m^3 | TWA 1 mg/m$^3$ | 100 mg/m$^3$ (as Cu) | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, nose, pharynx; nasal septum perforation; metallic taste; dermatitis; In Animals: lung, liver, kidney damage; anemia | Eyes, skin, respiratory system, liver, kidneys (increased risk with Wilson's disease) | Reddish, lustrous, malleable, odorless solid.<br>BP: 4703°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible - powdered form may ignite |
| Iron (as iron oxide) | 7439-89-6 | TWA 5 mg/m3 (respirable particulate mass) | TWA 1 mg/m^3 | NA | NA | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin, mucous membrane; abdominal pain, diarrhea, vomiting; possible liver damage | Eyes, skin, respiratory system, liver, gastrointestinal tract | Appearance and odor vary depending upon the specific soluble iron salt.<br>BP: NA<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible Solids |
| Lead | 7439-92-1 | TWA 0.05 mg/m3 | TWA (8-hour) 0.050 mg/m3 | [1910.1025] TWA 0.050 mg/m3 | 100 mg/m3 (as Pb) | Inhalation, ingestion, skin and/or eye contact | Lassitude (weakness, exhaustion), insomnia; facial pallor; anorexia, weight loss, malnutrition; constipation, abdominal pain, colic; anemia; gingival lead line; tremor; paralysis wrist, ankles; encephalopathy; kidney disease; irritation eyes; hypertension | Eyes, gastrointestinal tract, central nervous system, kidneys, blood, gingival tissue | A heavy, ductile, soft, gray solid<br>BP: 3164°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Noncombustible Solid in bulk form |
| Magnesium | 7439-95-4 | https://cameochemicals.noaa.gov/chemical/6949 | | | | Eye and/or skin contact | Dust irritates eyes in same way as any foreign material. Penetration of skin by fragments of metal is likely to produce local irritation, blisters, and ulcers which may become infected. | Eyes | A light silvery metal<br>BP: 1202°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Manganese | 7439-96-5 | TWA 0.02 mg/m3 [R]<br>TWA 0.1 mg/m3 [I] | TWA 1 mg/m3<br>ST 3 mg/m3 | C 5 mg/m3 | 500 mg/m3 (as Mn) | 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 | respiratory system, central nervous system, blood, kidneys | A lustrous, brittle, silvery solid<br>BP: 3564°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Metal: Combustible Solid |
| Mercury | 7439-97-6 | TWA 0.1 mg/m3, as Hg Aryl compounds<br>TWA 0.025 mg/m3 as Hg, inorganic forms including metallic mercury | Hg Vapor: TWA 0.05 mg/m3 [skin]<br>Other: C 0.1 mg/m3 [skin] | TWA 0.1 mg/m3 | 10 mg/m3 (as Hg) | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin; cough, chest pain, dyspnea (breathing difficulty), bronchitis, pneumonitis; tremor, insomnia, irritability, indecision, headache, lassitude (weakness, exhaustion); stomatitis, salivation; gastrointestinal disturbance, anorexia, weight loss; proteinuria | Eyes, skin, respiratory system, central nervous system, kidneys | Metal: Silver-white, heavy, odorless liquid. [Note: "Other" Hg compounds include all inorganic & aryl Hg compounds except (organo) alkyls.]<br>BP: 674°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Metal: Noncombustible Liquid |

**Table 1. Toxicological, Physical, and Chemical Properties of Compounds Potentially Present at the Site.**

| Compound | CAS # | ACGIH TLV | NIOSH REL | OSHA PEL | IDLH | Routes of Exposure | Toxic Properties | Target Organs | Physical/Chemical Properties |
|---|---|---|---|---|---|---|---|---|---|
| Nickel | 7440-02-0 | TWA 1.5 mg/m^3 [elemental]<br>TWA 0.1 mg/m^3 [soluble inorganic compound]<br>TWA 0.2 mg/m^3 [insoluble inorganic compound]<br>TWA 0.1 mg/m^3 [Nickel subsulfide] | Ca TWA 0.015 mg/m^3 | TWA 1 mg/m^3 | Ca [10 mg/m3 (as Ni)] | Inhalation, ingestion, skin and/or eye contact | Sensitization dermatitis, allergic asthma, pneumonitis; [potential occupational carcinogen] | Nasal cavities, lungs, skin | Lustrous, silvery, odorless solid.<br>BP: 5139°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Combustible Solid; nickel sponge catalyst may ignite spontaneously in air. |
| Potassium | 9/7/7440 | https://cameochemicals.noaa.gov/chemical/4289 | | | | Eye and/or skin contact | Will burn skin and eyes | Skin, eyes | Potassium is a soft silvery metal though normally grayish white due to oxidation<br>BP: 1425°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Selenium | 7782-49-2 | TWA 0.2 mg/m3 | TWA 0.2 mg/m3 | TWA 0.2 mg/m3 | 1 mg/m3 (as Se) | Inhalation, ingestion, skin and/or eye contact | Irritation eyes, skin, nose, throat; visual disturbance; headache; chills, fever; dyspnea (breathing difficulty), bronchitis; metallic taste, garlic breath, gastrointestinal disturbance; dermatitis; eye, skin burns; In Animals: anemia; liver necrosis, cirrhosis; kidney, spleen damage | Eyes, skin, respiratory system, liver, kidneys, blood, spleen | Amorphous or crystalline, red to gray solid. [Note: Occurs as an impurity in most sulfide ores.]<br>BP: 1265°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Combustible Solid |
| Silver | 7440-22-4 | TWA 0.1 mg/m3 [Metal, dust, and fume]<br>TWA 0.01 mg/m3 [Soluble compounds, as Ag] | TWA 0.01 mg/m3 | TWA 0.01 mg/m3 | 10 mg/m3 (as Ag) | Inhalation, ingestion, skin and/or eye contact | Blue-gray eyes, nasal septum, throat, skin; irritation, ulceration skin; gastrointestinal disturbance | Nasal septum, skin, eyes | Metal: White, lustrous solid<br>BP: 3632°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA<br>Metal: Noncombustible Solid, but flammable in form of dust or powder |
| Sodium | 7440-23-5 | https://cameochemicals.noaa.gov/chemical/7794 | | | | Skin contact | Severe burns caused by burning metal or by caustic soda formed by reaction with moisture on skin | Skin | A silvery soft metal that becomes grayish white upon exposure to air<br>BP: 1621°F<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Thallium | 7440-28-0 | 0.02 mg/m3 inhallable particulate matter | TWA 0.1 mg/m3 [skin] | TWA 0.1 mg/m3 [skin] | 15 mg/m3 (as Tl) | Inhalation, skin absorption, ingestion, skin and/or eye contact | Nausea, diarrhea, abdominal pain, vomiting; ptosis, strabismus; peri neuritis, tremor; retrosternal (occurring behind the sternum) tightness, chest pain, pulmonary edema; convulsions, chorea, psychosis; liver, kidney damage; alopecia; paresthesia legs | Eyes, respiratory system, central nervous system, liver, kidneys, gastrointestinal tract, body hair | Appearance and odor vary depending upon the specific soluble thallium compound<br>BP: NA<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Vanadium | 7440-62-2 | https://cameochemicals.noaa.gov/chemical/16147 | | | | Inhalation, skin absorption, ingestion, skin and/or eye contact | Highly toxic, may be fatal if inhaled, swallowed or absorbed through skin. Avoid any skin contact. Effects of contact or inhalation may be delayed. Fire may produce irritating, corrosive and/or toxic gases. Runoff from fire control or dilution water may be corrosive and/or toxic and cause pollution | Skin | Silvery-whitish powder<br>BP: NA<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| Zinc | 7440-66-6 | https://cameochemicals.noaa.gov/chemical/4814 | | | | Inhalation, skin absorption, ingestion, skin and/or eye contact | Inhalation or contact with vapors, substance or decomposition products may cause severe injury or death. May produce corrosive solutions on contact with water. Fire will produce irritating, corrosive and/or toxic gases. Runoff from fire control may cause pollution | Lungs | A grayish powder<br>BP: NA<br>Fl.Pt. = NA<br>LEL: NA<br>UEL: NA |
| **PCBs** | | | | | | | | | |
| PCBs (total) | 11097-69-1, 53469-21-9 | TWA 0.5 mg/m3 [skin]<br>TWA 1 mg/m3 [skin] | Ca TWA 0.001 mg/m3<br>Ca TWA 0.001 mg/m3 | TWA 0.5 mg/m3 [skin]<br>TWA 1 mg/m3 [skin] | Ca [5 mg/m3]<br>Ca [5 mg/m3] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, chloracne; liver damage; reproductive effects; [potential occupational carcinogen] | Skin, eyes, liver, reproductive system | Colorless to pale-yellow, viscous liquid or solid (below 50°F) with a mild, hydrocarbon odor<br>BP: 689-734°F, 617-691°F<br>Fl.Pt. = NA, NA<br>LEL: NA<br>UEL: NA<br>Nonflammable Liquid, but exposure in a fire results in the formation of a black soot containing PCBs, polychlorinated dibenzofurans, and chlorinated dibenzo-p-dioxins. |
| **Petroleum Hydrocarbons** | | | | | | | | | |
| Gasoline | 86290-81-5 | TWA 300 ppm<br>STEL 500 ppm | Ca | None | Ca [N.D.] | Inhalation, skin absorption, ingestion, skin and/or eye contact | Irritation eyes, skin, mucous membrane; dermatitis; headache, lassitude (weakness, exhaustion), blurred vision, dizziness, slurred speech, confusion, convulsions; chemical pneumonitis (aspiration liquid); possible liver, kidney damage; [potential occupational carcinogen] | Eyes, skin, respiratory system, central nervous system, liver, kidneys | Clear liquid with a characteristic odor<br>BP: 102°F<br>Fl.Pt. = -45°F<br>LEL: 1.4%<br>UEL: 7.6% |


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National Oceanic and Atmospheric Administration (NOAA) and Environmental Protection Agency (EPA), 2019. *CAMEO Chemicals Database* . https://cameochemicals.noaa.gov/

U.S. Department of Labor, 1990. *OSHA Regulated Hazardous Substances.* Industrial Exposure and Control Technologies Government Institutes, Inc.

**Table 1. Toxicological, Physical, and Chemical Properties of Compounds Potentially Present at the Site.**

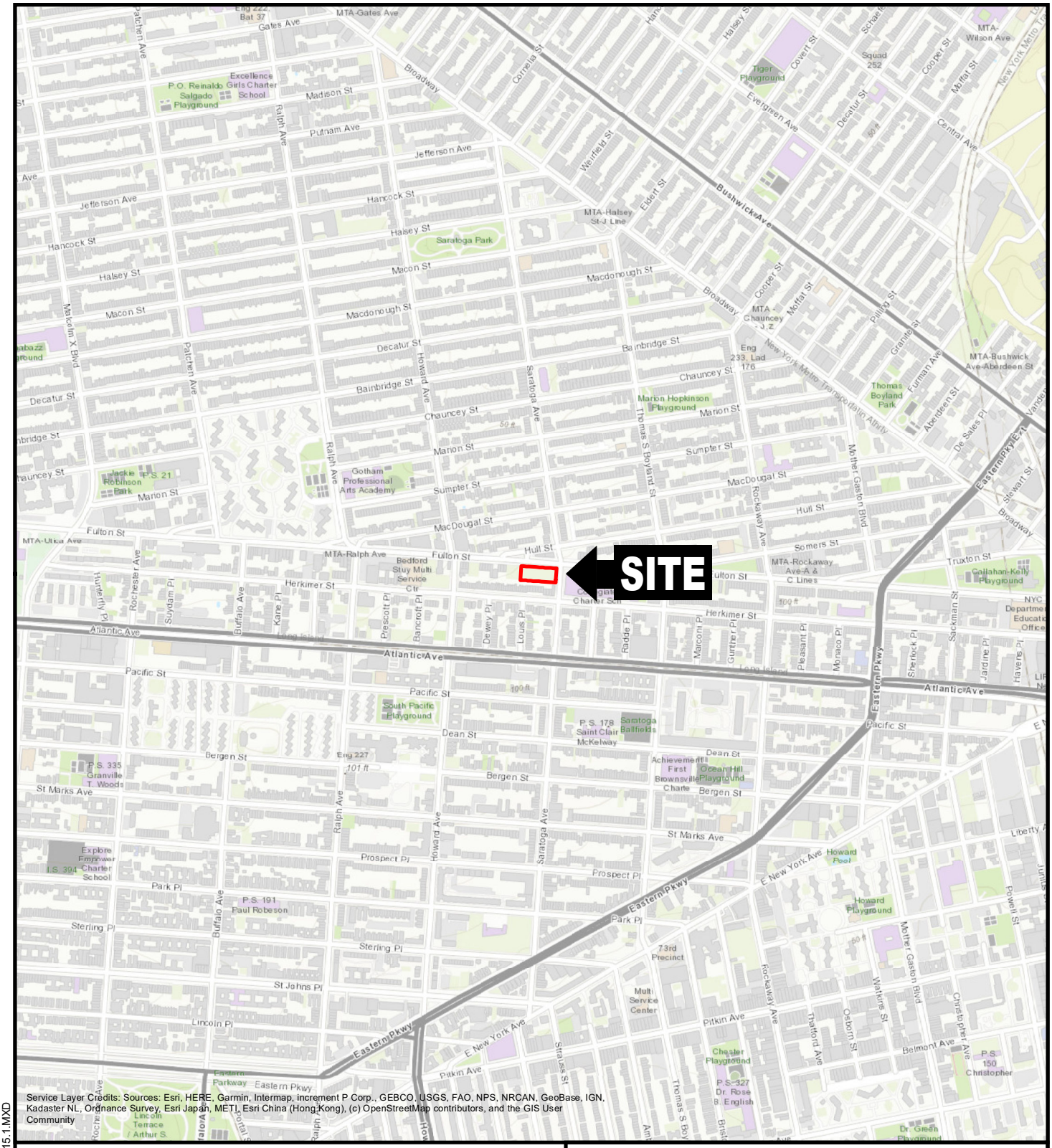
| Compound | CAS # | ACGIH TLV | NIOSH REL | OSHA PEL | IDLH | Routes of Exposure | Toxic Properties | Target Organs | Physical/Chemical Properties |
|---|---|---|---|---|---|---|---|---|---|

Sax, N. Van Nostrand and Reinhold Company, 1987. *Hawley's Condensed Chemical Dictionary, 11th Edition.*
Proctor, N.H., J.P. Hughes and M.L. Fischman, 1989. *Chemical Hazards of the Workplace* . Van Nostrand Reinhold. New York.
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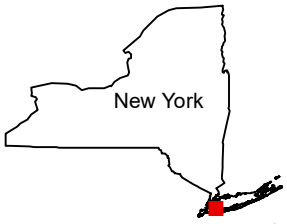
**Abbreviations:**
ACGIH – American Conference of Governmental Industrial Hygienists.
BP – boiling point at 1 atmosphere, °F
C – Ceiling, is a concentration that should not be exceeded during and part of the working exposure.
Ca – Carcinogenic.
CAS# - Chemical Abstracts Service registry number which is unique for each chemical.
DSEN - Dermal Sensitization
Ft Pt. – Flash point
IDLH - Immediately Dangerous to Life and Health concentrations represent the maximum concentration from which, in the event of respirator failure, one could escape within 30 minutes without a respirator and without experiencing any escape-impairing or irreversible health effects.
LEL – Lower explosive (flammable) limit in air, % by volume (at room temperature)
$mg/m^3$ – Milligrams of substance per cubic meter of air
NIOSH - National Institute for Occupational Safety and Health.
OSHA – Occupational Safety and Health Administration
OTO - Ototoxicant
PEL - OSHA Permissible Exposure Limit (usually) a time weighted average concentration that must not be exceeded during any 8 hour work shift of a 40 hr work week.
ppm – parts per million
REL – NIOSH Recommended Limit indicated a time weighted average concentration that must not be exceeded during any 10 hour work shift of a 40 hr work week
RSEN - Respiratory Sensitization
SG - Specific Gravity
STEL – ACGIH Short-term exposure limit (ST)
TLV - ACGIH Threshold Limit Values (usually 8 hour time weighted average concentrations).
TWA – 8-hour, time-weighted average
UEL – Upper explosive (flammable) limit in air, % by volume (at room temperature)
VP - Vapor Pressure

# FIGURES

1. Site Location Map
2. Site Plan with Emergency Muster Area
3. Routes to Urgent Care and Hospital




QUADRANGLE LOCATION




N

1,000 0 1,000'

Title:

# SITE LOCATION MAP

2030 FULTON STREET
BROOKLYN, NEW YORK

Prepared for:

STEPS FAMILY LLC

ROUX

| | |
|---|---|
| Compiled by: B.H. | Date: 10/09/25 |
| Prepared by: M.S.R. | Scale: AS SHOWN |
| Project Mgr: B.H. | Project: 3805.0002Y003 |
| File: 3805.0002Y115.1.mxd | |

FIGURE 1

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N

LEGEND

- EMERGENCY MUSTER AREA
- PROPOSED BCP SITE BOUNDARY

NOTES

1. AERIAL SOURCE: NYS OFFICE OF INFORMATION TECHNOLOGY SERVICES GIS PROGRAM OFFICE (GPO)
2. THE TAX LOTS WERE ALL MERGED INTO LOT 28 ON 08/21/2025

30 0 30'

Title:

# SITE PLAN WITH EMERGENCY MUSTER AREA

2030 FULTON STREET
BROOKLYN, NEW YORK

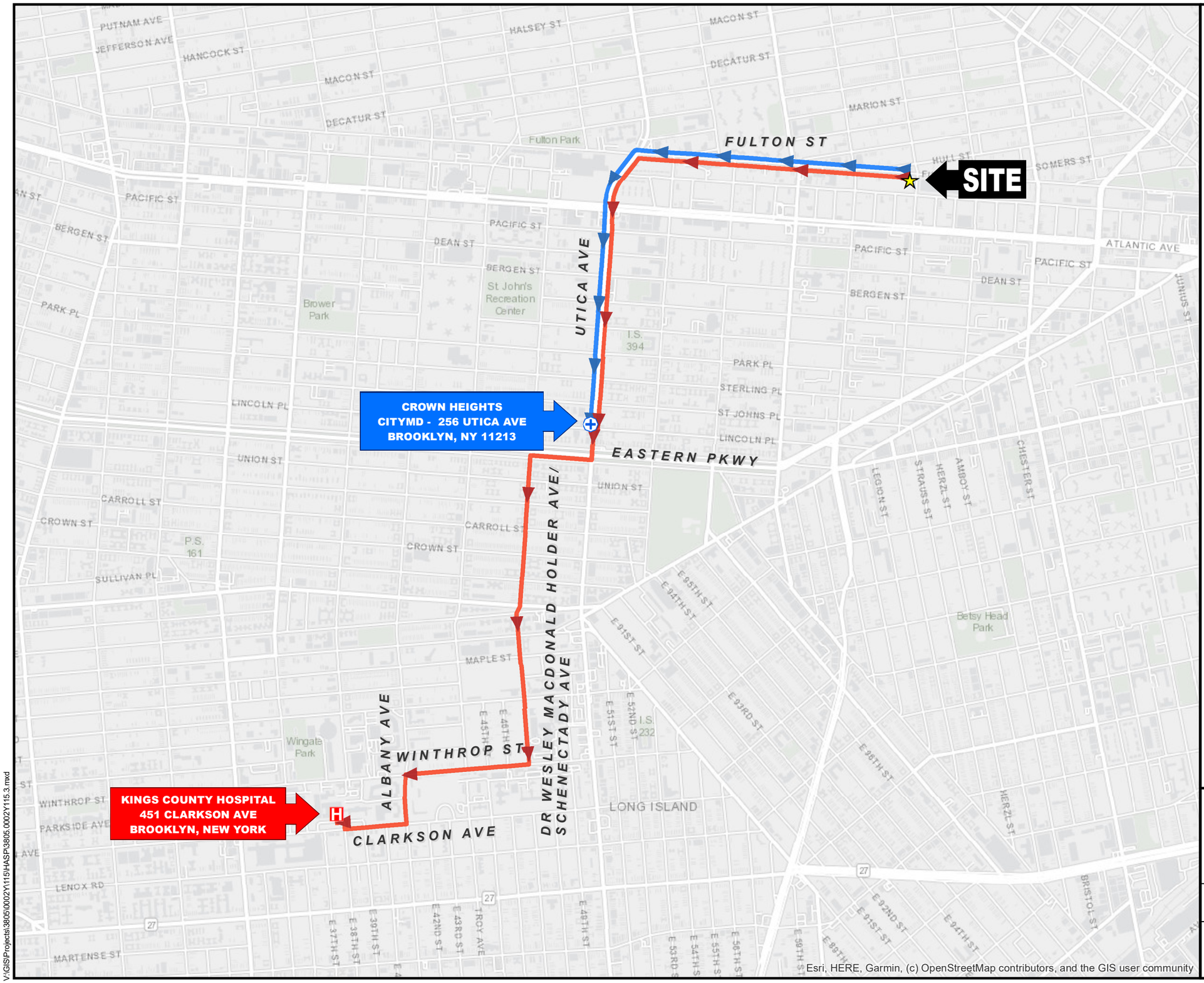
Prepared for:

STEPS FAMILY LLC

ROUX

| | | |
|---|---|---|
| Compiled by: B.H. | Date: 10/09/25 | FIGURE |
| Prepared by: M.S.R. | Scale: AS SHOWN | 2 |
| Project Mgr: B.H. | Project: 3805.0002Y003 | |
| File: 3805.0002Y115.2.mxd | | |

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N

DIRECTIONS TO HOSPITAL

1. HEAD WEST ON FULTON ST TOWARD HULL ST
2. TURN LEFT ONTO UTICA AVE
3. HEAD SOUTH ON UTICA AVE TOWARD LINCOLN PL
4. TURN RIGHT ONTO EASTERN PKWY
5. TURN LEFT ONTO DR WESLEY MACDONALD HOLDER AVE/ SCHENECTADY AVE
6. TURN RIGHT ONTO WINTHROP ST
7. TURN LEFT ONTO ALBANY AVE
8. TURN RIGHT ONTO CLARKSON AVE
9. TURN RIGHT INTO DESTINATION

DIRECTIONS TO URGENT CARE

1. HEAD WEST ON FULTON ST TOWARD HULL ST
2. TURN LEFT ONTO UTICA AVE
3. DESTINATION WILL BE ON THE RIGHT

1,200 0 1,200
Feet

Title:

# ROUTES TO URGENT CARE AND HOSPITAL

2030 FULTON STREET
BROOKLYN, NEW YORK

Prepared for:

STEPS FAMILY LLC

ROUX

| | | |
|---|---|---|
| Compiled by: B.H. | Date: 10/09/25 | FIGURE |
| Prepared by: M.S.R. | Scale: AS SHOWN | 3 |
| Project Mgr: B.H. | Project: 3805.0002Y003 | |
| File: 3805.0002Y115.3.mxd | | |

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# APPENDICES

A. Job Safety Analysis (JSA) Forms
B. SDSs for Chemicals Used
C. Heat Illness Prevention Program
D. Personal Protective Equipment (PPE) Management Program
E. Subsurface Utility Clearance Management Program
F. Heavy Equipment Exclusion Zone Management Program

# APPENDIX A

Job Safety Analysis (JSA) Forms

| JOB SAFETY ANALYSIS | Ctrl. No. GEN-006 | DATE 05/22/2025 | ☐ NEW ☒ REVISED | PAGE 1 of 2 |
|---|---|---|---|---|

| JSA TYPE CATEGORY: | WORK TYPE: | WORK ACTIVITY (Description): |
|---|---|---|
| **Generic** | **Drilling** | **Direct Push Soil Borings / Well Installation** |

**REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**

| | | | |
|---|---|---|---|
| ☐ LIFE VEST<br>☒ HARD HAT<br>☐ LIFELINE / BODY HARNESS<br>☒ SAFETY GLASSES | ☐ GOGGLES<br>☐ FACE SHIELD<br>☒ HEARING PROTECTION: (as needed)<br>☒ SAFETY SHOES: Composite-toe or steel-toe boots | ☐ AIR PURIFYING RESPIRATOR<br>☐ SUPPLIED RESPIRATOR<br>☒ PPE CLOTHING: Fluorescent reflective vest or high visibility clothing, long-sleeved shirt | ☒ GLOVES: Nitrile and cut-resistant<br>☒ OTHER: Insect Repellant, sunscreen (as needed) |

**REQUIRED AND / OR RECOMMENDED EQUIPMENT**

Geoprobe or Truck-Mounted Direct Push Drill Rig, Hand Tools, Photoionization Detector, Multi-Gas Meter (or equivalent), Macrocore liners, Liner Opening Tool, 20 lb. Type ABC Fire Extinguisher, 42" Cones & traffic control paddles, "Work Area" Signs, Water.

**COMMITMENT TO SAFETY-** All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing LMRAs

**EXCLUSION ZONE (EZ): Maintain a Minimum Heavy Equipment Exclusion Zone around equipment and loads while in motion. The HEEZ must be greater than the swing zone of any moving part of the equipment, the tip zone of the equipment, the fall zone of the equipment, and the contents.**

**"SHOW ME YOUR HANDS"**
**The driller and helper should show that their hands are clear of controls and moving parts.**

| Assess [1]JOB STEPS | Analyze [2]POTENTIAL HAZARDS | Act [3]CRITICAL ACTIONS |
|---|---|---|
| **1.** Mobilization of drilling rig (ensure the Subsurface Clearance Protocol and Drill Rig Checklist are completed) | **1a. CONTACT:** Equipment/property damage. | 1a. The drill rig's tower/derrick will be lowered and secured before mobilization.<br>1a. A spotter should be utilized while moving the drill rig. If personnel move into the path of the drill rig, the drill rig will be stopped until the path is again clear. Use a spotter for all required backing operations.<br>1a. Set up the work area and position equipment to eliminate or reduce the need for backing support trucks and trailers.<br>1a. When backing up the truck rig with an attached trailer, use a second spotter if there is tight clearance simultaneously on multiple sides of the equipment or if turning angles limit driver visibility. |
| Secure Work Zone | | 1a. Inspect the driving path for uneven terrain. Level or avoid if needed.<br>1a. Ensure the work area is secure and inform others of work activity.<br>Establish a HEEZ using traffic cones, barrels & snow fencing or telescoping poles.<br>Use flag persons to maintain clear traffic and minimize motorist confusion during set-up.<br>HEEZ is to include the tip/swing radius of the equipment. |
| | **1b. FALL:** Slip/trip/fall hazards. | 1b. Inspect walking path for uneven terrain, weather-related hazards (i.e., ice, puddles, snow, etc.), and obstructions before mobilizing equipment.<br>1b. Do not climb over stored materials/equipment; walk around. Practice good housekeeping.<br>1b. Use established pathways and walk on stable, secure ground. |
| | **1c. CONTACT:** Crushing from a rollover. | 1c Geoprobe should cross all hills/obstructions head-on with the mast down to reduce the risk of rollover. |
| **2.** Raising the tower/derrick of the drill rig | **2a. CONTACT:** Overhead hazards. | 2a. Before raising the tower/derrick, the area above the drilling rig will be inspected for wires, tree limbs, piping, or other structures that could come in contact with the rig's tower and/or drilling rods or tools.<br>2a. Maintain a minimum safe distance of 10' from overhead lines, material, or structures. |
| | **2b. CONTACT:** Pinch Points/Amputation Points when raising the rig, and the instability of the rig | 2b. Inspect the equipment before use and avoid pinch/amputation points.<br>2b. Lower outriggers to ensure stability before raising the rig tower/derrick.<br>2b. If the rig needs to be mounted, use three points of contact. |
| **3.** Advancement of drilling equipment and sound installation | **3a. CONTACT:** Flying debris | 3a. Be aware of and avoid potential line-of-fire hazards and wear required PPE such as eye, ear, and hand protection. |
| | **3b. EXPOSURE:** Noise and dust. | 3b. Wet borehole area with sprayer to minimize dust.<br>3b. Stand upwind and keep your body away from the rig.<br>3b. If conditions warrant, a disposable dust mask (e.g., N95/P100) should be worn.<br>3b. Wear hearing protection when the drill rig is in operation. |

[1] Each Job or Operation consists of a set of tasks / steps. Be sure to list all the steps needed to perform job.
[2] A hazard is a potential danger. Break hazards into six types: Contact - victim is struck by or strikes an object; Caught - victim is caught on, caught in or caught between objects; Fall - victim falls to ground or lower level (includes slips and trips); Exertion - excessive strain or stress / ergonomics / lifting techniques; Exposure - inhalation/skin hazards; Energy Source – electricity, pressure, compression/tension.
[3] Using the first two columns as a guide, decide what actions or procedures are necessary to eliminate or minimize the risk. List the recommended safe operating procedures. Say exactly what needs to be done - such as "use two persons to lift". Avoid general statements such as, "be careful".

| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **3.** Advancement of drilling equipment and well installation (Continued) | **3c. FALL:**<br>Slip/trip/fall hazards. | 3c. Contain drill cuttings and drilling water to prevent fall hazards from developing in the work area.<br>3c. See 1b. |
| | **3d. CAUGHT:**<br>Limb/extremity pinching; abrasion/crushing. | 3d. Ensure all Emergency Safety Stop buttons function properly.<br>3d. Always wear leather gloves when making connections and using hand tools; wear cut-resistant (i.e., Kevlar) gloves when handling cutting tools.<br>3d. Inspect the equipment prior to use for potential pinch/amputation points. Keep hands away from pinch/amputation points, and the use of tools is preferable compared to fingers and hands.<br>3d. Inspect the drill head for worn surface or missing teeth; replace it if damaged or blunt.<br>3d. Ensure all jewelry is removed, loose clothing is secured, and PPE is correctly fitted and secured close to the body.<br>3d. All non-essential personnel should stay away from the immediate work area; position the body out of the line-of-fire of equipment.<br>3d. Drillers and helpers will understand and use the "Show Me Your Hands" Policy.<br>3d. Spinning rods/casing have an **exclusion zone of 24 inches** while in operation. |
| | **3e. CONTACT:**<br>Equipment imbalance during the advancement of drill equipment. | 3e. Drillers will advance the borehole with caution to avoid causing the rig to become imbalanced and/or tip.<br>3e. The blocking and leveling devices used to secure the rig will be inspected by drillers and Roux personnel regularly to see if shifting has occurred.<br>3e. In addition, personnel and equipment that are non-essential to the advancement of the borehole will be positioned away from the rig at a distance that is at least as far as the boom is high (**minimum exclusion zone**). |
| | **3f. EXPOSURE:**<br>Inhalation of contamination/vapors. | 3f. Monitor ambient air for dangerous conditions using a calibrated photoionization detector (PID) to monitor the breathing zone of the work area.<br>3f. If a reading of >5ppm is recorded, the Roux field personnel must temporarily cease work, instruct all Site personnel to step away from the area of elevated readings, and inform the Roux PM of the condition. The Roux PM will then recommend additional precautions in accordance with the site-specific health and safety plan.<br>3f. Use a multi-gas meter to monitor ambient air for dangerous conditions (i.e., unsafe levels of carbon monoxide when drilling indoors or the presence of explosive vapors). |
| | **3g. EXERTION:**<br>Potential for muscle strain/injury while lifting and installing well casings, lifting sand bags, and/or lifting rods. | 3g. Keep your back straight and bend at the knees.<br>3g. Utilize team lifting or mechanical means for objects over 40 lbs.<br>3g. Use a mechanical lifting device for odd-shaped objects. |
| **4.** Remove the sample liner. | **4a. EXERTION:**<br>Potential for muscle strain/injury while removing the liner from the probe rod. | 4a Utilize team lifting or mechanical means for objects over 40 lbs.<br>4a. Use the hydraulic liner extruder if available. |
| | **4b. CONTACT:**<br>Pinch points and cuts | 4b. Place the liner on the sturdy surface when opening.<br>4b. Don cut-resistant gloves and use an appropriate liner cutter when opening liners.<br>4b. Always cut away from the body. |
| | **4c. EXPOSURE:**<br>Inhalation and/or dermal contact with contaminants. | 4c. Wear chemical-resistant disposable gloves when handling liners.<br>4c. See 3f. |

[1] Each Job or Operation consists of a set of tasks / steps. Be sure to list all the steps needed to perform job.
[2] A hazard is a potential danger. Break hazards into six types: Contact - victim is struck by or strikes an object; Caught - victim is caught on, caught in or caught between objects; Fall - victim falls to ground or lower level (includes slips and trips); Exertion - excessive strain or stress / ergonomics / lifting techniques; Exposure - inhalation/skin hazards; Energy Source – electricity, pressure, compression/tension.
[3] Using the first two columns as a guide, decide what actions or procedures are necessary to eliminate or minimize the risk. List the recommended safe operating procedures. Say exactly what needs to be done - such as "use two persons to lift". Avoid general statements such as, "be careful".

| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **5.** Decontaminate equipment. | **5a. EXPOSURE/CONTACT**: To contamination (e.g., Separate Phase Hydrocarbons (SPH), contaminated groundwater, vapors). | 5a. Wear chemical-resistant disposable gloves and safety glasses.<br>5a. Contain decontamination water so that it does not spill.<br>5a. Use an absorbent pad to clean spills, if necessary.<br>5a. Spray equipment from a side angle, not straight on, to avoid backsplash.<br>5a. See 3b and 3f. |
| | **5b. EXPOSURE:** The chemicals in the cleaning solution. | 5b. See 4a. Review the SDS to ensure appropriate precautions are taken and understood. |

[1] Each Job or Operation consists of a set of tasks / steps. Be sure to list all the steps needed to perform job.
[2] A hazard is a potential danger. Break hazards into six types: Contact - victim is struck by or strikes an object; Caught - victim is caught on, caught in or caught between objects; Fall - victim falls to ground or lower level (includes slips and trips); Exertion - excessive strain or stress / ergonomics / lifting techniques; Exposure - inhalation/skin hazards; Energy Source – electricity, pressure, compression/tension.
[3] Using the first two columns as a guide, decide what actions or procedures are necessary to eliminate or minimize the risk. List the recommended safe operating procedures. Say exactly what needs to be done - such as "use two persons to lift". Avoid general statements such as, "be careful".

| JOB SAFETY ANALYSIS | Ctrl. No. GEN-007 | DATE 05/22/2025 | ☐ NEW ☒ REVISED | PAGE 1 of 2 |
|---|---|---|---|---|

| JSA TYPE CATEGORY | WORK TYPE | WORK ACTIVITY (Description) |
|---|---|---|
| **GENERIC** | **General Site Activity** | **Driving** |

**REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**

| | | | |
|---|---|---|---|
| ☐ LIFE VEST<br>☐ HARD HAT: when outside vehicle<br>☐ LIFELINE / BODY HARNESS<br>☐ SAFETY GLASSES: when outside vehicle | ☐ GOGGLES<br>☐ FACE SHIELD<br>☐ HEARING PROTECTION<br>☐ SAFETY TOE BOOTS: when outside vehicle | ☐ AIR PURIFYING RESPIRATOR<br>☐ SUPPLIED RESPIRATOR<br>☐ PPE CLOTHING: high visibility vest when outside vehicle | ☐ GLOVES: Leather/ cut-resistant level 2<br>☐ OTHER _____ |

**REQUIRED AND / OR RECOMMENDED EQUIPMENT**

Motor Vehicle (i.e., car, truck, SUV)

**COMMITMENT TO SAFETY-** All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing LMRAs

| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **1.** Driving to/leaving the Site | **1a. CONTACT:**<br>Severe injury/disability, property damage, monetary loss (insurance premiums, deductibles, loss of license/job) caused by collision with or struck by other vehicles, obstructions, pedestrians, animals, etc.<br><br>*Common factors that may lead to a CONTACT incident, but not limited to:<br>• distracted driving (cell phone, GPS, radio, billboards, "rubbernecking")<br>• lack of situational awareness<br>• unfamiliarity with traffic patterns/road layout<br>• weather conditions (wet/icy roads, hydroplaning, black ice)<br>• weariness<br>• high speeds<br>• obstructed vision (solar glare, debris on windshield, blind spots, large vehicle at the front)<br>• changes in travel pathway (construction, snow banks, non-operational signals, potholes, detours, special events)<br>• improper vehicle maintenance (non-operational signal light, worn tires, cracked windshield, ineffective wipers)<br>• loose or unsecure objects | 1a. PLAN AHEAD – review/make yourself familiar with maps and driving directions before beginning the drive to the Site. Do not attempt to drive and review maps/directions at the same time. Pull over and stop your vehicle before looking at maps/directions.<br><br>1a. If a Roux vehicle or a Roux-rented vehicle, complete a vehicle inspection form. If a personal vehicle, complete a basic 360 vehicle inspection before driving.<br><br>Verify Inspection and Registration are current, tires and wipers are in good condition, all lights are functional, all glass/mirrors are undamaged, the horn is functional, roof/hood/trunk are free from accumulated snow, and visibility is not impaired due to snow/ice/frost/fog on windows.<br><br>1a. Do not hang items in the car that can obstruct your view or become projectiles in a collision.<br><br>1a. Do not get distracted using touch screen radios or GPS units built into newer models. Keep your eyes on the road and stay alert.<br><br>1a. Follow posted speed limits and obey traffic signals and roadway signs.<br><br>1a. Always wear your seat belt and shoulder harness when driving.<br><br>1a. When driving around large vehicles and trucks, maintain extra space as these vehicles may not be able to see a smaller car too close.<br><br>1a. Follow the "Rules of the Road," including: using your turn signals, coming to a complete stop, and allowing vehicles the right of way (yield) when required by traffic laws.<br><br>1a. Apply the Smith Five Keys® of safe driving<br>• Aim High in Steering®<br>- Expand eye lead time to a minimum of 15 seconds<br>• Get the Big Picture®<br>- Maintain a proper 4-second minimum following distance at all times<br>- Scan mirrors every 5-8 seconds to achieve a circle of awareness<br>- Position your vehicle so you can see relevant/non-relevant objects<br>• Keep Your Eyes Moving®<br>- Try to maintain about 180 degrees of visibility<br>- Avoid blank and fixed stares. Avoid focusing on one object for more than 2 seconds<br>• Leave Yourself an Out®<br>- Avoid traveling in traffic clusters<br>- Surround yourself with space<br>- Anticipate the actions of others |

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[3] Using the first two columns as a guide, decide what actions or procedures are necessary to eliminate or minimize the risk. List the recommended safe operating procedures. Say exactly what needs to be done - such as "use two persons to lift". Avoid general statements such as, "be careful".

| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **1.** Driving to/leaving Site (cont'd) | **1a. CONTACT:**<br>Severe injury/disability, property damage, monetary loss (insurance premiums, deductibles, loss of license/job) caused by collision with or struck by other vehicles, obstructions, pedestrians, animals, etc. | • Make Sure They See You®<br>- Maintain eye contact with oncoming vehicles/pedestrians<br>- Use warning devices (e.g., hand signals, high-lights, horns, etc.)<br>- Proper timing is essential<br>1a. Do not perform reconnaissance or inspections while driving. Your vehicle should be parked in a safe location when viewing or surveying the Site and vicinity<br>1a. Avoid sudden turns and stops. Do not drive recklessly – be in control of the vehicle at all times.<br>1a. In inclement weather, first determine if work can be POSTPONED. Otherwise, plan according to weather conditions, including checking the forecast along the entirety of the travel route (especially for long distances). Reduce speed as road conditions warrant. Travelling with winter car equipment in the winter is strongly recommended (i.e., shovel, scraper, brush, blanket, extra clothing, flashlight, bag of sand). If your vehicle has 4-wheel drive, review the operator's manual and understand the operating procedure prior to engaging 4-wheel drive. If at any point on your drive, the weather becomes too severe to proceed safely, pull over if safe to do so or seek nearest cover (e.g., overpass)<br>1a. If feeling drowsy or sleepy, do not drive. Pull over in a safe place to rest if you experience any signs of drowsiness. Make sure to get adequate sleep the night before an early drive.<br>1a. Never operate a vehicle under the influence of alcohol, illegal substances, or medications affecting your performance.<br>1a. Keep your eyes on the road. Do not call, text, or talk on cellular phones. Pull over to a safe location if you must answer or make a call.<br>1a. When parking, pull through when possible. If backing is required, visually inspect the area to ensure it is free from obstructions prior to backing in and relying solely on mirrors; use spotters when available. |
| **2.** Entering/Exiting Vehicle. | **2a. CAUGHT:**<br>Personal injury (broken fingers/hand) while entering or exiting vehicles<br><br>**2b. FALL:**<br>Personal injury (twisted ankle, deep contusion, concussion, broken wrist/arm, etc.) from a slip/fall on an uneven, unstable, or slippery surface while exiting/entering the vehicle<br><br>**2c. CONTACT:**<br>Severe injury/disability, property damage, monetary loss (insurance premiums, deductibles, loss of license/job) caused by collision with or struck by other vehicles, obstructions, pedestrians, animals, etc. | 2a. Open and close doors slowly. Never put hands or feet in between the door and the vehicle to avoid pinch points.<br><br>2b. When exiting the vehicle, make sure your feet are on firm footing and weight is evenly distributed before exiting/standing. In inclement weather, use your hands to support yourself by holding the car door and/or steering wheel when exiting the vehicle.<br><br>2c. Check both directions for traffic before opening the door. Do not exit the vehicle if traffic does not permit you to exit safely<br>2c. Check the anticipated path of the door prior to opening. Do not open the door into any obstructions (e.g., bollards, high curbs) |

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| JOB SAFETY ANALYSIS | Ctrl. No. GEN-009 | DATE: 05/23/2025 | ☐ NEW ☒ REVISED | PAGE 1 of 1 |
|---|---|---|---|---|
| JSA TYPE CATEGORY **Generic** | WORK TYPE **O&M** | WORK ACTIVITY (Description) **Movement of 55-Gallon Drums/Drum Handling with Mobile Carrier** | | |

**REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**

| | | | |
|---|---|---|---|
| ☐ LIFE VEST | ☐ GOGGLES | ☐ AIR PURIFYING RESPIRATOR | ☒ GLOVES: Cut-resistant gloves level II |
| ☒ HARD HAT | ☐ FACE SHIELD | ☐ SUPPLIED RESPIRATOR | ☐ OTHER: |
| ☐ LIFELINE / BODY HARNESS | ☐ HEARING PROTECTION | ☒ PPE CLOTHING: Fluorescent long-sleeve shirt or shirt and reflective safety vest. | |
| ☒ SAFETY GLASSES | ☒ SAFETY SHOES: Steel or composite toe | | |

**REQUIRED AND / OR RECOMMENDED EQUIPMENT**

Mobile Drum Carrier, over-pack drum container, safety cones, and caution tape

**COMMITMENT TO SAFETY-** All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing LMRAs

| Assess [1]JOB STEPS | Analyze [2]POTENTIAL HAZARDS | Act [3]CRITICAL ACTIONS |
|---|---|---|
| **1.** Preparing for an Inspection of the Drum | **1a. FALL:** Tripping/falling due to uneven surface. Loose debris/garbage in the work area. | 1a. Clear the area of loose garbage and debris. Inspect 55-gal drums for proper condition, labeling, check drum ring and bolts for tightness, and inspect mobile drum carrier.<br>1a. Do a Test Lift to get a general sense of the weight of the drum.<br>1a. Inspect and use established pathways to avoid uneven terrain, weather-related hazards (i.e., debris, puddles, ice, etc.), and other obstructions.<br>1a. Secure a work area and coordinate and communicate the planned work activities with other personnel working in the area.<br>1a. Delineate work area with safety cones and caution tape |
| | **1b. CONTACT/EXPOSURE:** Drums could potentially be damaged or contain hazardous material. A mobile drum carrier could potentially be in poor working condition, causing malfunctioning during operation. | 1b. Prior to inspecting drums, don cut-resistant gloves. If the drum is not properly labeled, do not open it and cease all drum transport activities. Immediately contact the project manager and inform him/her of the drum situation.<br>1b. Do not continue drum transport activities until the project manager determines further actions.<br>1b. If the drum is properly labeled, but leaking, improperly sealed, or in poor condition, place the drum in an overpack drum.<br>1b. Inspect the mobile drum carrier to ensure its overall integrity. Look for rust marks or potential weak points where the drum carrier could malfunction. Inspect the wheels to ensure that they easily turn and nothing is impeding their movement. |
| | **1c. EXERTION/CAUGHT:** Potential pinching/exertion hazards while securing the ring/ tightening bolts | 1c. Keep back straight and knees slightly bent while securing the drum ring/tightening bolt. Wear cut-resistant gloves. |
| **2.** Position the drum clamp tightly in between the drum ribs, securing the drum clamp to the drum with the chain | **2a. CAUGHT:** Pinching fingers between the drum clamp and the handle/chain. | 2a. Attach drum clamp with chain and tighten until snug. Do not place hands between the drum clamp and the drum as the chain is tightened; wear cut-resistant gloves. Keep face away from the drum when handling in case of escaping vapors. |

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[3] Using the first two columns as a guide, decide what actions or procedures are necessary to eliminate or minimize the risk. List the recommended safe operating procedures. Say exactly what needs to be done - such as "use two persons to lift." Avoid general statements such as, "be careful."

| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **3.** Disengage safety latches on handle, pull handle down until drum is lifted off the ground, and safety latches are reengaged; slightly suspend drum off the ground | **3a. EXERTION/ CONTACT:** Potential muscle strain associated with lifting/engaging the drum/handle. The drum could shift/slip downward and crush toes. | 3a. Ascertain whether the drum is overweight; if it is, then two people are needed to lower the handle while the drum is secured with the clamp so that safety latches can be engaged. Keep body out of the line-of-fire of the handle (do not position the head above the handle) as it is being pushed down. Do not allow feet/toes to be positioned under the drum as it is being lifted; wear steel/composite-toed boots. |
| | **3b. CAUGHT:** Fingers could be pinched while engaging/disengaging the safety latches on the handle | 3b. Wear cut-resistant gloves while disengaging/reengaging safety latches.<br>3b. Avoid placing hands in pinch points. |
| **4.** Transport drums to the designated location and disengage the drum clamp (repeat Step 3 in reverse order) | **4a. FALL:** Tripping/ falling due to obstructions and uneven terrain. Potential for the drum to fall during transport. | 4a. Ensure the transport path is free of potential obstructions that may cause the drum/carrier to become unstable. Position the drum clamp between the ribs on the drum to prevent possible slipping. |

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| JOB SAFETY ANALYSIS Ctrl. No. GEN-013 | DATE 05/28/2025 | ☐ NEW ☒ REVISED | PAGE 1 of 2 |
|---|---|---|---|

| JSA TYPE CATEGORY | WORK TYPE: | WORK ACTIVITY (Description): |
|---|---|---|
| **Generic** | **Gauging and Sampling** | **Gauging and Sampling** |

**REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**

| | | | |
|---|---|---|---|
| ☒ LIFE VEST<br>☒ HARD HAT<br>☐ LIFELINE / BODY HARNESS<br>☒ SAFETY GLASSES | ☐ GOGGLES<br>☐ FACE SHIELD<br>☐ HEARING PROTECTION<br>☒ SAFETY SHOES: Composite-toed or steel-toed boots | ☐ AIR PURIFYING RESPIRATOR<br>☐ SUPPLIED RESPIRATOR<br>☒ PPE CLOTHING: Fluorescent reflective vest or high visibility clothing | ☒ GLOVES: , Nitrile and cut-resistant level II<br>☒ OTHER: Knee pads, Insect Repellant, sunscreen (as needed) |

**REQUIRED AND / OR RECOMMENDED EQUIPMENT**

Traffic Safety Cones, Caution Tape, Interface Probe with Ground Clamp, and/or Water Level Meter, 20-lb. type ABC Fire Extinguisher, Buckets. Tools as needed: Socket Wrench, Screw Driver, Crow Bar, Mallet, and Wire Brush.

**COMMITMENT TO SAFETY-** All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing LMRAs

| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **1.** Mobilization to monitoring well(s). | **1a. FALL:** Personal injury from slip/trip/fall due to uneven terrain and/or obstructions. | 1a. Inspect the pathway and plan for the most suitable designated pathway prior to mobilization.<br>1a. Use established pathways, walk and/or drive on stable, secure ground, and avoid steep hills or uneven terrain.<br>1a. If working near open water with an unguarded edge, wear a personal flotation device (PFD). |
| | **1b. CONTACT:** With traffic/third parties. | 1b. Identify potential traffic sources and delineate the work area with traffic safety cones. Position the vehicle to protect against oncoming traffic. Use caution tape to provide a more visible delineation of the work area if necessary.<br>1b. Wear appropriate PPE, including high visibility clothing and reflective vest, when exposed to traffic<br>1b. Face traffic, maintain eye contact with oncoming vehicles, and establish a safe exit route. |
| | **1c. EXERTION:** Muscle strain from lifting equipment | 1c. Use proper lifting techniques when handling/moving equipment; bend knees and keep back straight.<br>1c. Use mechanical assistance or team lifting techniques when equipment is 40 lbs. or heavier.<br>1c. Make multiple trips to carry equipment. |
| | **1d. EXPOSURE:** To biological hazards. | 1d. Inspect work area for bees and insects.<br>1d. Use insect/tick repellent, as necessary.<br>1d. Treat clothing with permethrin prior to mobilization.<br>1d. Don Tyvek/mosquito nets, as applicable. |
| **2.** Open/close well. | **2a. EXERTION:** Muscle strain. | 2a. Use proper lifting techniques; keep back straight, lift with legs, and bend knees when reaching to open/close the well. |
| | **2b. CAUGHT:** Pinch/crush points associated with removing/replacing manholes and working with hand tools. | 2b. Wear cut-resistant level II gloves when working with well cover and hand tools.<br>2b. Use proper tools (ratchet and pry bar or magnet for well cover) and inspect before use.<br>2b. Do not put fingers under the well cover. |
| | **2c. CAUGHT:** Pinch points associated with placing the J-plug back onto the PVC pipe. | 2c. See 2b.<br>2c. Keep fingers out of line-of-fire when securing cap. |
| | **2d. EXPOSURE:** To potential hazardous vapors. | 2d. No open flames/heat sources.<br>2d. To minimize exposure to vapors, allow the well to vent after opening it and before sampling activities begin.<br>2d. Stand upwind if possible, to avoid inhaling vapors.<br>2d. Monitor atmosphere with multi-gas meter/PID. |
| **3.** Gauge well. | **3a. CONTACT:** With contamination (e.g., contaminated groundwater). | 3a. Wear chemical-resistant disposable gloves (over cut-resistant gloves) and safety glasses when gauging well.<br>3a. Insert and remove the probe slowly to avoid splashing.<br>3a. Use an absorbent pad to clean probe. |
| | **3b. CONTACT:** With traffic. | 3b. See 1b. |
| | **3c. Exposure:** To static electricity, fire, or explosion. | 3c. Ground interface probe to a designated grounding point/rod, well casing, or electrically conductive surface to dissipate static electricity. |

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| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **4.** Purge and sample well | **4a. EXPOSURE/CONTACT:** To contamination (e.g., SPH, contaminated groundwater, vapors) and/or sample preservatives. | 4a. Open and fill sample jars slowly to avoid splashing and contact with preservatives.<br>4a. Wear cut-resistant gloves and chemical-resistant disposable gloves when sampling.<br>4a. Fill sample containers over the purge container to avoid spilling water onto the ground.<br>4a. Use an absorbent pad to clean spills.<br>4a. When using a bailer to purge a well, pull the bailer slowly from the well to avoid splash hazards.<br>4a. When sampling or purging the water using a bailer, pour water slowly to reduce the potential for splash hazards with groundwater.<br>4a. When using a tubing valve, always remove the valve slowly after sample collection to release any pressure and avoid pressurized splash hazards.<br>4a. When collecting a groundwater sample, always point the sampling apparatus (tubing, bailer, etc.) away from the face and body. |
| | **4b. CONTACT:** Personal injury from cuts, abrasions, or punctures by glassware or sharp objects. | 4b. To avoid spills or breakage, place sampleware on an even surface.<br>4b. Do not over-tighten caps on glass sampleware.<br>4b. Wear chemical-resistant nitrile disposable gloves over cut-resistant (i.e., Kevlar) gloves when sampling and handling glassware (i.e., VOA vials) or when using approved cutting tools. |
| | **4c. EXERTION:** Muscle strain while carrying equipment. | 4c. Use proper lifting techniques when handling/moving equipment, bend knees, and keep back straight.<br>4c. Use mechanical assistance or team lifting techniques when equipment is 40 lbs. or heavier.<br>4c. Make multiple trips to carry equipment. |
| | **4d. CONTACT:** With traffic. | 4d. See 1b. |
| | **4e. CONTACT:** Pinch points with groundwater pump components (i.e., wheel, line, clamps). | 4e. Wear cut-resistant gloves when working with groundwater pumps.<br>4e. Never place hands on or near pinch points such as the wheel, clamps, or other moving parts during pump operations.<br>4e. Use the correct mechanisms, such as a pump reel, to lower the pump into the well.<br>4e. Never attempt to manually stop any moving part of equipment, including hose reels and/or tubing. |
| | **4f. EXERTION:** Muscle strain from repetitive motion of bailing and sampling a well. | 4f. See 4c.<br>4f. Include a stretch break when repetitive motions are part of the task. |
| **5.** Management of purge water. | **5a. EXPOSURE/CONTACT:** To contamination (e.g., SPH, contaminated groundwater, vapors). | 5a. Do not overfill the container, and pour liquids slowly so that they do not splash.<br>5a. Properly dispose of used materials/PPE in an appropriate container in the designated storage area. |
| | **5b. EXERTION:** Muscle strain from lifting/carrying, and moving containers. | 5b. Use proper lifting techniques when lifting/carrying or moving container(s) (see 4c.).<br>5b. Do not overfill container(s). |
| **6.** Decontaminate equipment. | **6a. EXPOSURE/CONTACT:** To contamination (e.g., SPH, contaminated groundwater, vapors). | 6a. Work on the upwind side, where possible, of the decon area.<br>6a. Wear chemical-resistant disposable gloves and safety glasses.<br>6a. Use an absorbent pad to clean spills. |
| | **6b. CAUGHT:** Pinch points associated with handling hand tools | 6b. See 2b.<br>6b. Inspect hand tools for sharp edges before decontaminating. |

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| JOB SAFETY ANALYSIS | Ctrl. No. GEN-015 | DATE: 05/28/2025 | ☐NEW ☒REVISED | PAGE 1 of 2 |
|---|---|---|---|---|

| JSA TYPE CATEGORY | WORK TYPE | WORK ACTIVITY (Description) |
|---|---|---|
| **GENERIC** | **Site Recon** | **Mobilization/Demobilization** |

**REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**

| | | | |
|---|---|---|---|
| ☐ LIFE VEST<br>☒ HARD HAT<br>☐ LIFELINE / BODY HARNESS<br>☒ SAFETY GLASSES | ☐ GOGGLES<br>☐ FACE SHIELD<br>☒ HEARING PROTECTION (as needed)<br>☒ SAFETY SHOES: Steel Toe or composite toe | ☐ AIR PURIFYING RESPIRATOR<br>☐ SUPPLIED RESPIRATOR<br>☒ PPE CLOTHING: Fluorescent reflective vest of high-visibility clothing; long-sleeved shirt; long pants | ☒ GLOVES: Cut-resistant Level II, nitrile<br>☐ OTHER |

**REQUIRED AND / OR RECOMMENDED EQUIPMENT**

Required Equipment: Varies

**COMMITMENT TO SAFETY-** All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing LRAs

**EXCLUSION ZONE (EZ): Maintain a Minimum Heavy Equipment Exclusion Zone around equipment and loads while in motion. The HEEZ must be greater than the swing zone of any moving part of the equipment, the tip zone of the equipment, the fall zone of the equipment, and the contents.**

| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| 1. Mobilize/demobilize and establish a work area | **1a. FALL:** Slip/trips/falls from obstructions, uneven terrain, weather conditions, heavy loads, and/or poor housekeeping. | 1a. Use 3 points-of-contact/ensure secure footing when entering and exiting the vehicle.<br>1a. Inspect walking path for uneven terrain, steep hills, obstructions, and/or weather-related hazards (i.e., ice, snow, and puddles) prior to mobilizing equipment. Use established pathways. Walk on stable/secure ground.<br>1a. Do not climb over stored materials/equipment; walk around. Practice good housekeeping; organize and store equipment neatly in one area at its lowest potential energy.<br>1a. Wear boots with adequate treads.<br>1a. Delineate unsafe areas with traffic safety cones, caution tape, and/or flagging. |
| | **1b. CONTACT:** Personal injury and/or property damage caused by being struck by Site traffic or equipment used in Site activities. | 1b. Observe and maintain the posted speed limits.<br>1b. When first arriving onsite, park vehicles in designated parking spaces and/or out of the way locations. Use parking brake on all vehicles and tire chocks on work trucks and trailers.<br>1b. Check in with Site Manager/Supervisor to ensure coordination with other Site activities and to discuss any special hazards. Ensure that short-service employees (SSE) are identified.<br>1b. Identify potential traffic sources.<br>1b. Wear PPE, including high visibility clothing or reflective vest, when around vehicles or operating equipment<br>1b. Use a spotter while moving work vehicles; plan to avoid backing whenever possible.<br>1b. Maintain a minimum exclusion zone when vehicles are in motion (i.e., greater than the swing/tip radius of the equipment). When backing up the truck rig with an attached trailer, use a second spotter if there is tight clearance simultaneously on multiple sides of the equipment or if turning angles limit driver-to-spotter visibility.<br>1b. Delineate work area with traffic cones, flags, caution tape, and/or other barriers.<br>1b. Position “Work Area” signs at Site entrances, if possible, or at either side of the work area.<br>1b. Position the largest vehicle to protect against oncoming traffic.<br>1b. Face traffic, maintain eye contact with oncoming vehicles, use a spotter, and establish a safe exit route. |

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| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| | | 1b. Observe potential overhead and ground surface features that may interfere with moving equipment. Clear the path of physical hazards prior to initiating mobilization. |
| | **1c. CAUGHT:** Personal injury from pinch points and being in the line of fire of the vehicle and/or equipment. | 1c. Make sure the driver has engaged the parking brake and placed wheel chocks in a position to prevent movement. Be sure that the vehicle is parked in front/down gradient (positioned to block oncoming traffic) of the work area.<br>1c. Wear cut-resistant gloves (Kevlar or similar) when handling objects/tools or equipment,<br>1c. Keep body parts away from line-of-fire of equipment.<br>1c. Always carry tools by the handles and/or designated carrier. Ensure sharp-edged tools are sheathed/secure.<br>1c. Remove any loose jewelry. Avoid wearing loose clothing and/or ensure loose clothing is secure.<br>1c. Secure all items on the equipment, tighten up any items or features that have the potential to shift or break during mobilization. |
| | **1d. OVEREXERTION:** Muscle strains while lifting/carrying equipment. | 1d. Use body positioning and lifting techniques that avoid muscle strain; keep back straight, lift with legs, turn with whole body, keep load close to body, and never reach with a load.<br>1d. Ensure that loads are balanced. Use assistance (mechanical or additional person) to carry equipment that is either unwieldy or over 50 lbs. |
| | **1e. EXPOSURE:** Personal injury from exposure to biological and environmental hazards. | 1e. Inspect the area to avoid contact with biological hazards (i.e., poisonous plants, stinging insects, ticks, etc.).<br>1e. Wear long-sleeved clothes that can be treated with Permethrin, apply insect repellent (may contain DEET) to exposed skin, and inspect clothes and skin for ticks during and after work.<br>1e. Apply sunscreen (SPF 15+) if exposure to the sun for 30 minutes or more is expected. |
| | **1f. EXPOSURE:** Weather-related injuries. | 1f. Watch for heat stress symptoms (muscle cramping, exhaustion, dizziness, nausea, rapid and shallow breathing). Take breaks in cool places and hydrate as needed.<br>1f. Watch for cold stress symptoms (severe shivering, slowing body movement, weakness, stumbling or inability to walk, collapse). Take breaks in warm areas as needed.<br>1f. Wear clothing appropriate for weather and temperature conditions (e.g., rain jackets, snow pants, multiple layers).<br>1f. If lightning is observed, wait 30 minutes in a sheltered location (car is acceptable) before resuming work. |
| | **1g. EXPOSURE:** Personal injury from noise hazards. | 1g. Wear hearing protection if sound levels exceed 85 dBA (if you must raise your voice for normal conversation). |

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[3] Using the first two columns as a guide, decide what actions or procedures are necessary to eliminate or minimize the risk. List the recommended safe operating procedures. Say exactly what needs to be done - such as "use two persons to lift". Avoid general statements such as, "be careful".

| JOB SAFETY ANALYSIS | Ctrl. No. GEN-017 | DATE: 05/28/2025 | ☐NEW ☒REVISED | PAGE 1 of 2 |
|---|---|---|---|---|

| JSA TYPE CATEGORY: | WORK TYPE: | WORK ACTIVITY (Description): |
|---|---|---|
| **GENERIC** | **Drilling** | **Monitoring and Recovery Well Development** |

**REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**

| | | | |
|---|---|---|---|
| ☐ LIFE VEST<br>☒ HARD HAT<br>☐ LIFELINE / BODY HARNESS<br>☒ SAFETY GLASSES | ☐ GOGGLES<br>☐ FACE SHIELD<br>☒ HEARING PROTECTION (as needed)<br>☒ SAFETY SHOES: Composite-toe or steel-toe boots | ☐ AIR PURIFYING RESPIRATOR<br>☐ SUPPLIED RESPIRATOR<br>☒ PPE CLOTHING: Fluorescent reflective vest or high visibility clothing | ☒ GLOVES: Cut-resistant level II and Nitrile<br>☒ OTHER: Insect repellent, sunscreen (as needed) |

**REQUIRED AND / OR RECOMMENDED EQUIPMENT**

Required Equipment as needed: Truck Rig or support truck, Trailer, Traffic Safety cones and flags, Caution Tape, Interface Probe, Power Source, Submersible Pump, Surge Block/Plunger, 20 lb. Type ABC Fire Extinguisher, Holding Tanks and/or Buckets, Absorbent Pads, 5-gas meter, Tools as needed: Socket and Pipe Wrench, Screw Driver, Pry Bar, Ratchet, Vault Key, Clamp or Vice Grip.

**COMMITMENT TO SAFETY-** All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing LMRAs

**EXCLUSION ZONE (EZ): Maintain a Minimum Heavy Equipment Exclusion Zone around equipment and loads while in motion. The HEEZ must be greater than the swing zone of any moving part of the equipment, the tip zone of the equipment, the fall zone of the equipment, and the contents.**

**"SHOW ME YOUR HANDS"**
**The driller and helper should show that their hands are clear of controls and moving parts.**

| Assess [1]JOB STEPS | Analyze [2]POTENTIAL HAZARDS | Act [3]CRITICAL ACTIONS |
|---|---|---|
| **1.** Mobilization / Demobilization (**Review Mobilization and Demobilization JSA - GEN-015**) | **1a. CONTACT:** Equipment/property damage. | 1a. The truck rig's tower/derrick will be lowered and secured prior to mobilization.<br>1a. Set up the work area/position equipment in a manner that eliminates or reduces the need for backing of trucks and trailers.<br>1a. All non-essential personnel should **maintain an exclusion zone greater than the swing/tip radius of the equipment.**<br>1a. Beep horn twice before backing up.<br>1a. When backing up with an attached trailer, use a spotter Position if able to avoid backing up and pull through. |
| | **1b. FALL:** Slip/trip/fall hazards. | 1b Inspect walking path for uneven terrain, weather-related hazards (i.e., ice, puddles, snow, etc.), and obstructions prior to mobilizing equipment.<br>1b. Do not climb over stored materials/equipment; walk around. Store equipment at the lowest potential energy. |
| **2.** Open/close well. | **2a. EXERTION:** Muscle strain (some wells have large vault covers). | 2a. Keep back straight, lift with legs, keep the load close to the body, and never reach with a load. Ensure that loads are balanced to reduce the potential for muscle strain. Use the buddy system or mechanical means when lifting objects heavier than 50 lbs or when the shape makes the object difficult to lift. |
| | **2b. CAUGHT:** Pinch points are associated with removing/replacing manholes and working with hand tools. | 2b. Wear cut-resistant gloves when working with well vault/cover and hand tools. Do not put fingers under the well vault/cover.<br>2b. Use ratchet and pry bar for the well cover and inspect before use. |
| | **2c. EXPOSURE:** Potentially hazardous vapors. | 2c. No open flames/heat sources.<br>2c. Allow the well to vent after opening it and before starting development activities to minimize exposure to vapors. Air monitoring must be performed prior to set up and during the well development activities. Work on the upwind side of the well. |
| | **2d. CONTACT:** Traffic. | 2d. Wear required PPE, including high visibility clothing or reflective vest.<br>2d. Delineate work area with safety cones and/or other barriers. Position the vehicle to protect against oncoming traffic.<br>2d. Face traffic, maintain eye contact with oncoming vehicles, and establish a safe exit route. |

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| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **3.** Develop well (mechanical surging). | **3a. CAUGHT:** Cut hazards and finger pinch points. | 3a See 2b.<br>3a. Use required PPE, including cut-resistant level II gloves, when handling development equipment. Identify finger/hand pinch points. Keep hands away from active surge equipment.<br>3a. All non-essential personnel should **maintain an exclusion zone greater than the swing/tip radius of the equipment.** |
| | **3b. CONTACT/EXPOSURE:** Contamination (e.g., SPH, contaminated groundwater, vapors). | 3b. See 2c.<br>3b. Wear Nitrile gloves and safety glasses. Insert and remove the surge block/plunger and line/cable slowly to avoid splashing at the surface.<br>3b. Use an absorbent pad to clean any spills. |
| | **3c. EXERTION:** Muscle strain from lifting equipment. | 3c. See 2a.<br>3c. Use mechanical device to insert and remove surge block/plunger if greater than 40 lb. |
| | **3d. CONTACT:** Injury while handling wench line/cable, or with active surging equipment. | 3d. If using a drill rig, inspect all wench lines/cables for any kinks or fraying prior to use. Replace any damaged lines/cables. Review the **Drill Rig checklist prior to development activities**.<br>3d. See 3a. |
| **4.** Purging well (pumping water to holding tanks/drums/buckets). | **4a. CAUGHT:** Pinch points are associated with connecting the hose to the tank. Pinch points are associated with handling the pump and hoses. | 4a. See 3a.<br>4a. Ensure that fingers are not placed near the coupling when attaching and securing the hose(s). Do not place fingers under the pump/hoses. Wear cut-resistant gloves when handling pump/hose(s).<br>4a. Keep hands clear of any line of fire. |
| | **4b. FALL:** Using a side-mounted ladder when attaching a hose to the tank.<br>Slip, trip, or fall from lines/hoses | 4b. Inspect ladder steps to make sure steps are not bent/damaged and free of debris/fluid.<br>4b. Use three points of contact always when using a ladder.<br>4b. Use a hoist or other mechanical means to secure and move hoses.<br>4b. Utilize anti-whip cords and secure couplings on all pressurized hoses. Keep hoses and lines coiled and organized out of designated walking paths around the work zone. |
| | **4c. CONTACT:** Contamination (e.g., SPH, contaminated groundwater). | 4c. Secure water hose to holding tanks/drums/buckets with clamp, vice grip, or similar tool.<br>4c. Do not overfill tanks, and purge/transfer liquids in such a manner that they do not splash. (See 3b).<br>4c. Dispose of used materials/PPE in the designated impacted PPE container. |
| | **4d. EXERTION:** Muscle strain from lifting/carrying equipment. | 4d. See 2a. |
| | **4e. FALL:** Spilled purge water. | 4e. Clean up any spills using absorbent pads or spill kits. |
| **5.** Decontaminate equipment | **5a. CONTACT/EXPOSURE:** Contamination (e.g., SPH, contaminated groundwater, vapors). | 5a. See 3b. |
| | **5b. EXPOSURE/CONTACT:** Chemicals in the cleaning solution | 5b. Decontaminate equipment in a well-ventilated area. Wear nitrile gloves to avoid skin contact with cleaning solutions. |

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| JOB SAFETY ANALYSIS | Ctrl. No. GEN-019 | DATE: 05/28/2025 | ☐ NEW ☒ REVISED | PAGE 1 of 2 |
|---|---|---|---|---|

| JSA TYPE CATEGORY | WORK TYPE | WORK ACTIVITY (Description) |
|---|---|---|
| **GENERIC** | **Site Reconnaissance** | **Site Walk and Inspection** |

**REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**

| | | | |
|---|---|---|---|
| ☐ LIFE VEST<br>☒ HARD HAT<br>☐ LIFELINE / BODY HARNESS<br>☒ SAFETY GLASSES | ☐ GOGGLES<br>☐ FACE SHIELD<br>☒ HEARING PROTECTION: earplugs/muffs as necessary<br>☒ SAFETY SHOES: Steel or composite toed | ☐ AIR PURIFYING RESPIRATOR<br>☐ SUPPLIED RESPIRATOR<br>☒ PPE CLOTHING: High-visibility vest or high-vis outerwear | ☒ GLOVES: /cut-resistant (level II)/chemical resistant<br>☒ OTHER: Tyvek and rubber boots as necessary, dust mask as necessary |

**REQUIRED AND / OR RECOMMENDED EQUIPMENT**

Required Equipment: Site map, emergency contact list, documentation of urgent care/hospital routes, and/or guide familiar with Site, operating cell phone or walkie-talkie if Site allows, and bug spray.

**Commitment to Safety** – All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing LMRAs

**EXCLUSION ZONE (EZ): Maintain a Minimum Heavy Equipment Exclusion Zone around equipment and loads while in motion. The HEEZ must be greater than the swing zone of any moving part of the equipment, the tip zone of the equipment, the fall zone of the equipment, and the contents.**

**SITE SECURITY: Before site inspection, verify the appropriate method to address Site Security concerns related to potential criminal activity, homeless population, and/or isolation concerns. Work with the Project Principal and/or Project Manager to address appropriately.**

| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **1.** Check in with the Site contact. | **1a. CONTACT/EXPOSURE/FALL:** Personal injury caused by a lack of awareness of site-specific hazards. | 1a. Inquire about hazards and other activities taking place at the Site.<br>1a. Inform Site contact of work scope, timeline, and location(s).<br>1a. Discuss emergency evacuation procedures and muster points with the Site contact. |
| **2.** Traversing the Site | **2a. CONTACT:** Property damage and personal injury caused by obstructions/vehicles or unauthorized personnel at remote Sites. | 2a. All equipment must be stowed and secured prior to moving.<br>2a. Maintain speed limit as posted on-site.<br>2a. When possible, drive on established roadways.<br>2a. Yield to all pedestrians.<br>2a. Use pull-through spots or back into parking spots.<br>2a. Don high-visibility clothing/safety vest. If working at a remote Site, add orange accessories during hunting season. |
| | **2b. FALL:** Uneven terrain and weather conditions. Overgrown shrubs and vines. Equipment in the work zone. | 2b. Inspect walking path for uneven terrain, weather-related hazards (i.e., ice, puddles, snow, etc.), and obstructions prior to mobilizing equipment.<br>2b. Use established pathways and walk on stable, secure ground when possible.<br>2b. Communicate traversing hazards with others. |
| | **2c. OVEREXERTION:** Muscle strain while carrying equipment. | 2c. When carrying equipment to/from the work area, use proper lifting techniques; keep back straight, lift with legs, keep the load close to the body, never reach with a load. Ensure that loads are balanced to reduce the potential for muscle strain. Use the buddy system or mechanical means to maneuver items heavier than 40 lbs. If necessary, make multiple trips to carry equipment. |
| | **2d. EXPOSURE:** Biological hazards – ticks, bees/wasps, poison ivy, insects; (Ticks are most active whenever the temperature is above freezing, typically from March to November.) | 2d. Inspect the area to avoid contact with biological hazards.<br>2d. Ticks:<br>• Treat outer clothing, including pants, shirts, socks, boots, and hats, the evening before with Permethrin (allowing at least two hours before use).<br>• Apply DEET or equivalent to exposed skin before travelling to the Site and reapply after two hours.<br>• Wear light colored clothing and visually check for ticks during and after work.<br>2d. Bees:<br>• Use bee spray as appropriate to deter/eliminate bees.<br>• Protect exposed skin with insect repellent. |

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| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| | | 2d. Poison Ivy:<br>• Identify areas of poison ivy and spray with weed killer. Don Tyvek and rubber boots while traversing poison ivy areas.<br>• If skin comes into contact with poison ivy, wash the skin thoroughly with soap and water. |
| | **2e. EXPOSURE:**<br>Heat Stress & Cold Stress. Personal injury from working in inclement weather conditions. | 2e. Wear sunscreen with SPF 15 or greater on exposed skin whenever 30 minutes or more of sun exposure is expected.<br>2e. Watch for heat stress symptoms (muscle cramping, exhaustion, dizziness, rapid and shallow breathing). Take breaks as needed.<br>2e. Watch for cold stress symptoms (severe shivering, slowing body movement, weakness, stumbling or inability to walk, collapse). Take breaks as needed.<br>2e. Wear appropriate rain gear as needed.<br>2e. Take frequent breaks if tired, wet, or cold/hot. Drink water.<br>2e. If lightning is observed, wait 30 minutes after the last thunder boom/lightning bolt in a sheltered location (car acceptable) before starting work again. |
| **3.** Walking near heavy equipment and machinery. | **3a. CONTACT:**<br>Personal injury from Site and roadway traffic. Personal injury from flying debris | 3a. See 2a.<br>3a. Maintain an exclusion zone of at least 10 feet from all engaged equipment. Increase distance to include swing tip radius.<br>3a. Keep body parts out of the line-of-fire of pinch points.<br>3a. Wear appropriate PPE always. |
| | **3b. OVEREXERTION:**<br>Personal injury from lifting/moving/rotating equipment. | 3b. See 2c. |
| | **3c. EXPOSURE:**<br>Hearing damage from noise-generating equipment/processes.<br>Inhalation/exposure to hazardous vapors and or dust. | 3c. Wear hearing protection if >85 dBA. (i.e., noise levels which require you to raise your voice to communicate)<br>3c. Always wear cut-resistant gloves when handling any tools or equipment.<br>3c. Always wear appropriate PPE based on the chemicals present. |
| | **3d. EXPOSURE:**<br>Working in a remote area. | 3d. Use the "buddy system" whenever possible. If working alone, contact PM upon arrival/departure and during work activities prior to commencing work if applicable.<br>3d. Always carry a communication device (i.e., cell phone, walkie-talkie) or directional (i.e., map, compass, etc.) when traversing remote areas.<br>3d. If available, follow Lone Worker Protocol/Procedure. |
| **4.** Working in adverse weather conditions. | **4a. EXPOSURE:**<br>Heat Stress & Cold Stress. Personal injury from working in inclement weather conditions. | 4a. Watch for heat stress symptoms (muscle cramping, exhaustion, dizziness, rapid and shallow breathing). Take breaks as needed.<br>4a. Watch for cold stress symptoms (severe shivering, slowing body movement, weakness, stumbling or inability to walk, collapse). Take breaks as needed.<br>4a. Wear appropriate rain gear as needed.<br>4a. Take frequent breaks if tired, wet, or cold/hot. Drink water.<br>4a. If lightning is observed, wait 30 minutes after the last thunder boom/lightning bolt in a sheltered location (car acceptable) before starting work again. |
| **5.** Departing Site. | **5a. EXPOSURE:**<br>Exposure to unnecessary hazards should prompt personnel to believe Roux is on-site during an emergency and conduct a search. | 5a. Sign out or notify the Site contact and Roux Project Manager of your departure. |

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| JOB SAFETY ANALYSIS | Ctrl. No. GEN-020 | DATE: 05/29/2025 | ☐ NEW ☒ REVISED | PAGE 1 of 2 |
|---|---|---|---|---|

| JSA TYPE CATEGORY: | WORK TYPE: | WORK ACTIVITY (Description): |
|---|---|---|
| **GENERIC** | **Gauging & Sampling** | **Soil Sampling** |

**REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**

| | | | |
|---|---|---|---|
| ☐ LIFE VEST<br>☒ HARD HAT<br>☐ LIFELINE / BODY HARNESS<br>☒ SAFETY GLASSES<br>☒ FLAME RESISTANT CLOTHING (as needed) | ☐ GOGGLES<br>☐ FACE SHIELD:<br>☒ HEARING PROTECTION: (as needed)<br>☒ SAFETY SHOES: Composite-toe or steel-toe boots | ☐ AIR PURIFYING RESPIRATOR<br>☐ SUPPLIED RESPIRATOR<br>☒ PPE CLOTHING: Fluorescent reflective vest or high visibility clothing | ☒ GLOVES: , Nitrile and cut resistant level II<br>☒ OTHER: Insect repellent, sunscreen (as needed) |

**REQUIRED AND / OR RECOMMENDED EQUIPMENT**

Recommended Equipment: traffic safety cones, caution tape, trowel

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| Assess [1]JOB STEPS | Analyze [2]POTENTIAL HAZARDS | Act [3]CRITICAL ACTIONS |
|---|---|---|
| **1.** Secure location | **1a. CONTACT:** Personnel and vehicular traffic may enter the work area. | 1a. If in an area with foot or vehicle traffic, delineate the work area with traffic cones and/or caution tape to prevent exposure to traffic and inform others of work activity.<br>1a. Wear a reflective vest and/or high visibility clothing.<br>1a. Face the direction of any vehicular traffic. Position the vehicle to protect the worker from traffic.<br>1a. Communicate work activity with adjacent work areas. |
| | **1b. FALL:** Tripping/falling due to uneven terrain or entry/exit from excavations. | 1b. Inspect pathways and work area for uneven terrain, weather-related hazards (i.e., ice, puddles, snow, etc.), and obstructions.<br>1b. Use established pathways and walk on stable, secure ground.<br>1b. Stage equipment and tools in a convenient, stable, and orderly manner. Store equipment at the lowest potential energy.<br>1b. Roux employees should stay 6 feet from in-progress excavations and trenches. Should entry to an excavation be required (when stabilization is complete), ladders must be employed for steep embankments, excavations, pits, and trenches. |
| | **1c. EXPOSURE:**<br>Exposure to sun and excessive heat, possibly causing sunburn, heat exhaustion, or heat stroke.<br>Exposure to cold temperatures may cause cold stress.<br>Skin burn as a result of fire, if applicable.<br>Exposure to explosive vapors due to tank farm operations.<br>Exposure to airborne dust due to high wind speeds.<br>Biological hazards - ticks, bees/wasps, poison ivy, thorns, insects, etc. | 1c. Wear sunscreen with an SPF 15 or greater whenever 30 minutes or more of exposure is expected.<br>1c. Use a tent to shade the work area from direct sunlight, particularly when warm temperatures are expected.<br>1c. Be aware of the location of all Site personnel.<br>1c. Watch for heat stress symptoms (muscle cramping, exhaustion, dizziness, rapid and shallow breathing).<br>1c. Watch for cold stress symptoms (severe shivering, slowing of body movement, weakness, stumbling or inability to walk, collapse).<br>1c. Take breaks for rest and water as necessary. Move to an area that is well shaded or a climate-controlled area (i.e., car, site trailer, etc.).<br>1c. No open flames/heat sources.<br>1c. Conduct air monitoring and ensure that harmful vapor concentrations are within the levels detailed in the Site Specific HASP. Follow the procedures detailed in HASP for exceedances.<br>1c. Flame retardant clothing must be worn when specified by Site policy.<br>1c. Cell phones should be disabled when specified by Site policy.<br>1c. Pre-treat field clothing with Permethrin prior to site visit to kill ticks and insects.<br>1c. Wear long-sleeved shirts and tuck in (or tape) pant legs into socks or boots to prevent ticks from reaching skin.<br>1c. Spray insect repellent containing DEET or equivalent on exposed skin when working in overgrown areas of the Site.<br>1c. Inspect the area to avoid contact with biological hazards.<br>1c. Wear cut-resistant gloves when handling material, equipment, or branches, shrubs, etc. that may lie within the walking path.<br>1c. Wear spoggles if the average wind speeds are above 15 mph.<br>1c. Personnel shall examine themselves and their co-workers' outer clothing for ticks periodically when on-site.<br>1c. If skin comes in contact with poison ivy, wash skin thoroughly with soap and water. If rash persists after washing, immediately notify your supervisor, the OM, and OHSM for possible consultation with a physician at an approved Occupational Health Clinic. |

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| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **2.** Collect Soil Sample | **2a. CONTACT:**<br>Personal injury from pinch points, cuts, and abrasions from sampling equipment, tools, and material within the soil sample.<br>Personal injury from contact with moving equipment while sampling.<br>Personal injury from contact with glass sample jars. | 2a. Wear cut-resistant (i.e., Kevlar) gloves under chemical-resistant (nitrile) disposable gloves when handling soil samples and sampling jars.<br>2a. Where possible, use a trowel or equivalent tool to avoid contact with soil.<br>2a. If sampling from a bucket of heavy equipment, ensure all equipment is off and the operator utilizes the "show me your hands" policy.<br>2a. See 1a. |
| | **2b. EXPOSURE:**<br>Exposure to contamination (impacted soil) and/or lab preservatives. | 2b. Wear chemical-resistant (nitrile) disposable gloves over cut-resistant gloves to protect hands when handling samples; use containment material or plastic sheeting to protect surrounding areas.<br>2b. Wear safety glasses to protect eyes from dust or air-borne contaminants that may result from disturbing the soil.<br>2b. Where possible, remain upgradient from the sample location if collecting soil samples from stockpile, drill rig, etc., to avoid breathing contaminant vapors, if they are present.<br>2b. When collecting a soil sample from a hand auger, put a large zip lock bag over the entire auger to prevent soil from spilling onto the ground.<br>2b. Open sample jars slowly and fill carefully to avoid contact with preservatives. |
| | **2c. EXERTION:**<br>Exertion due to repetitive motion and ergonomics. | 2c. Utilize a table or raised surface for soil sampling if multiple soil samples are going to be taken to minimize repetitive bending motion. |
| **3.** Decontaminate equipment | **3a. EXPOSURE/CONTACT:**<br>Contamination (e.g., Separate Phase Hydrocarbons (SPH), contaminated vapors, and/or soil).<br>**3b. EXPOSURE:**<br>Chemicals in the cleaning solution include ammonia. | 3a. Wear chemical-resistant (nitrile) disposable gloves and safety glasses.<br>3a. Use an absorbent pad to clean spills.<br>3a. Properly dispose of used materials/PPE in the provided drums in the designated drum storage area.<br>3a. Remain upwind of the sample and avoid breathing contaminant vapors, if they are present.<br>3b. Wear chemical-resistant (nitrile) disposable gloves and safety glasses.<br>3b. Work on the upwind side of the decontamination area.<br>3b. Use an absorbent pad to clean spills.<br>3b. Properly dispose of used materials/PPE in the provided drums in the designated drum storage area. Ensure that all drums are properly labeled and secured. |

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[3] Using the first two columns as a guide, decide what actions or procedures are necessary to eliminate or minimize the risk. List the recommended safe operating procedures. Say exactly what needs to be done - such as "use two persons to lift". Avoid general statements such as, "be careful".

| JOB SAFETY ANALYSIS | Ctrl. No. GEN-021 | DATE: 05/29/2025 | ☐ NEW ☒ REVISED | PAGE 1 of 2 |
|---|---|---|---|---|

| JSA TYPE CATEGORY: | WORK TYPE | WORK ACTIVITY (Description) |
|---|---|---|
| **GENERIC** | **Gauging and Sampling** | **Soil Vapor Sampling (Permanent Monitoring Points)** |

**REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**

| | | | |
|---|---|---|---|
| ☐ LIFE VEST<br>☒ HARD HAT<br>☐ LIFELINE / BODY HARNESS<br>☒ SAFETY GLASSES | ☐ GOGGLES<br>☐ FACE SHIELD<br>☐ HEARING PROTECTION<br>☒ SAFETY SHOES: Steel-toe composite safety boots | ☐ AIR PURIFYING RESPIRATOR<br>☐ SUPPLIED RESPIRATOR<br>☒ PPE CLOTHING: Fluorescent reflective vest or high visibility clothing | ☒ GLOVES: Cut-resistant level ii & Nitriles<br>☒ OTHER: Bug Spray, Sun Screen, Knee Pads, or kneeling pad |

**REQUIRED AND / OR RECOMMENDED EQUIPMENT**

9/16" Socket and Wrench, Non-Toxic Clay, Teflon-Lined Tubing, Masterflex Tubing, Air Pump with Low Flow, Dry Cal, Enclosure (Bucket with 2 holes), Helium Gas Canister, Summa Canisters and Flow Controllers, MultiRAE Photo Ionization Detector (PID), Helium Detector, Tubing Cutter, Traffic/Safety Cones, Caution Tape or Retractable Cone Bars

**COMMITMENT TO SAFETY-** All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing LMRAs.

**Work Zone (WZ): A 5-foot exclusion zone will be maintained for non-essential personnel.**

| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **1.** Define and secure a work area. | **1a. FALL:** Potential tripping hazards. | 1a. Ensure work area is secure and inform others (third party) of work activity.<br>1a. Remove tripping hazards and inspect the walking path for uneven terrain, weather-related hazards (i.e., ice, puddles, snow, etc.), and obstructions prior to mobilizing equipment. |
| | **1b. CONTACT:** Potential contact with moving vehicles or pedestrians. | 1b. If working alongside roads, look both ways before entering roadways, face traffic, and utilize the work vehicle to protect employees.<br>1b. Delineate work area (including vehicles) with traffic safety cones and caution tape or retractable cone bars.<br>1b. Maintain a 5-foot exclusion zone.<br>1b. Wear high visibility clothing or a reflective safety vest. |
| | **1c. EXERTION:** Muscle strain while lifting and carrying equipment. | 1c. When carrying equipment to/from the work area, keep back straight, lift with legs, keep the load close to the body, never reach with a load. Ensure that loads are balanced. Use the buddy system or mechanical means to maneuver loads heavier than 40 lbs. If necessary, make multiple trips to carry equipment. |
| **2.** Remove well cover / close well cover. | **2a. CONTACT/CAUGHT:** Pinch points and scrapes associated with hand tools and well covers. | 2a. Keep hands away from pinch points.<br>2a. Use hand tools with extensions or a magnet to remove and replace well covers.<br>2a. Wear cut-resistant gloves.<br>2a. Use knee pads or a kneeling pad when repetitive kneeling on rough ground is anticipated. |
| | **2b. FALL:** Potential tripping hazards associated with installing bolts. | 2b. Place security bolts in a secure location so as not to create tripping hazards. Replace security bolts so that they fit flush with monitoring well covers. |
| | **2c. EXERTION:** Physical exertion is required to remove bolts that were over-torqued or stripped. | 2c. Replace any security bolts that show signs of stripping. Do not over-tighten.<br>2c. Use body positioning and bending techniques that minimize muscle strain; keep back straight, bend at the knees.<br>2c. See 2a. |

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[3] Using the first two columns as a guide, decide what actions or procedures are necessary to eliminate or minimize the risk. List the recommended safe operating procedures. Say exactly what needs to be done - such as "use two persons to lift". Avoid general statements such as, "be careful".

| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **3.** Screen vapor point with PID. | **3a. FALL:**<br>Potential tripping hazards associated with equipment. | 3a. Place the equipment in one area close to the sampling location.<br>3a. Identify an area where the equipment is to be stored within the work area (away from the main walking path).<br>3a. Do not leave equipment on the ground. Return equipment to the storage area between uses. |
| | **3b. EXPOSURE:**<br>Inhalation of soil vapor | 3b. Replace brass caps immediately upon completion to avoid soil vapors migrating to the surface through sample tubing.<br>3b. Stand upwind of the sample point during screening activities. |
| **4.** Remove/replace brass caps at the end of the sample tubing. | **4a. CONTACT:**<br>Pinch points are associated with hand tools and brass caps. | 4a. Use a wrench to remove and replace brass caps.<br>4a. Wear cut-resistant gloves to protect against pinch points and scrapes. |
| | **4b. EXPOSURE:**<br>Potential pathway for vapors to migrate to the land surface. | 4b. See 3b.<br>4b. Stand upwind of the sample point location. |
| **5.** Set up soil vapor sampling equipment and calibration of meters. | **5a. FALL:**<br>Potential tripping hazards associated with equipment and tubing. | 5a. See 3a.<br>5a. Keep tubing slack to a minimum and locate the summa canister as close to the sampling location as possible.<br>5a. Avoid stepping over equipment and tubing. |
| | **5b. CONTACT:**<br>Pinch points associated with handling equipment. | 5b. Do not place fingers/hands under sampling equipment.<br>5b. Make multiple trips when unloading equipment in the work area.<br>5b. Wear cut-resistant gloves to protect against pinch points while handling sampling equipment. |
| | **5c. EXPOSURE:**<br>Inhalation of calibration gas and helium. | 5c. Review the SDS for each type of calibration gas used before calibrating.<br>5c. Calibrate meters in a well-ventilated area and keep the air flow regulator away from your face.<br>5c. Close valve on canisters after use to avoid inhalation of excess helium or calibration gas.<br>5c. Stand upwind of the bucket during the helium tracer gas test. |
| **6.** Cleaning Work Area. | **6a. FALL:**<br>Potential tripping hazards are associated with equipment and tubing. | 6a. See 3a.<br>6a. See 3b. |
| | **6b. CONTACT:**<br>Storing and transport of equipment in car. | 6b. Ensure that the equipment is placed securely in the vehicle. Do not stack equipment on top of each other. Secure the equipment so that it will not slide while being transported.<br>6b. Wear cut-resistant gloves while handling/loading equipment. |

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| JOB SAFETY ANALYSIS | Ctrl. No. GEN-022 | DATE 05/29/2025 | ☐ NEW ☒ REVISED | PAGE 1 of 2 |
|---|---|---|---|---|

| JSA TYPE CATEGORY: | WORK TYPE: | WORK ACTIVITY (Description): |
|---|---|---|
| **General** | **Drilling** | **Rotosonic Soil Borings / Well Installation** |

**REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**

| | | | |
|---|---|---|---|
| ☐ LIFE VEST<br>☒ HARD HAT<br>☐ LIFELINE / BODY HARNESS<br>☒ SAFETY GLASSES | ☐ GOGGLES<br>☐ FACE SHIELD<br>☒ HEARING PROTECTION: (as needed)<br>☒ SAFETY SHOES steel or composite toe | ☐ AIR PURIFYING RESPIRATOR<br>☐ SUPPLIED RESPIRATOR<br>☒ PPE CLOTHING: fluorescent long-sleeve shirt and reflective safety vest. | ☒ GLOVES: Nitrile and cut resistant level II<br>☒ OTHER: Insect Repellant, sunscreen (as needed) |

**REQUIRED AND / OR RECOMMENDED EQUIPMENT**

Truck-Mounted Sonic Drilling Rig or Mini Sonic Rig, Hand Tools, Photoionization Detector, Multi-Gas Meter (or equivalent), Interface Probe, 20 lb. Type ABC Fire Extinguisher, Traffic/Safety Cones & Flags, "Work Area" Signs, Water

**COMMITMENT TO SAFETY-** All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing LMRAs

**EXCLUSION ZONE (EZ): Maintain a Minimum Heavy Equipment Exclusion Zone around equipment and loads while it is in motion. The HEEZ must be greater than the swing zone of any moving part of the equipment, tip zone of the equipment, fall zone of the equipment and contents.**

**"SHOW ME YOUR HANDS"**
**Driller and helper should show that their hands are clear from controls and moving parts**

| Assess [1]JOB STEPS | Analyze [2]POTENTIAL HAZARDS | Act [3]CRITICAL ACTIONS |
|---|---|---|
| **1.** Mobilization of the drilling rig (ensure the Subsurface Clearance Protocol and Drill Rig Checklist are completed).<br><br>**See also Mobilization/Demobilization JSA.** | **1a. CONTACT/CRUSH:** Equipment/property damage during movement. Crush point between the moving rig and other objects. | 1a. The drill rig's tower/derrick will be lowered and secured prior to mobilization.<br><br>1a. A spotter should be utilized while moving the drill rig or backing operations. If personnel move into the path of the drill rig, the drill rig will be stopped until the path is again clear.<br><br>1a. Set up the work area and position equipment in a manner that eliminates or reduces the need for backing of support trucks and trailers.<br><br>1a. When backing up the truck rig with an attached trailer, use a second spotter if there is tight clearance simultaneously on multiple sides of the equipment or if turning angles limit driver/spotter visibility.<br><br>1a. Inspect the driving path for uneven terrain. Level or avoid if needed.<br><br>1a. Drill rig should have a **minimum exclusion zone (i.e., greater than the swing/tip radius of the rig)** for non-essential personnel (i.e., geologist) when the rig is moving/in operation. |
| | **1b. FALL:** Slip/trip/fall hazards. | 1b Inspect walking path for uneven terrain, weather-related hazards (i.e., ice, puddles, snow, etc.), and obstructions prior to mobilizing equipment.<br><br>1b. Do not climb over stored materials/equipment; walk around. Practice good housekeeping.<br><br>1b. Use established pathways and walk on stable, secure ground. |
| **2.** Raising the tower/derrick of the drill rig. | **2a. CONTACT**: Overhead hazards. | 2a. Prior to raising the tower/derrick, the area above the drilling rig will be inspected for overhead utilities, tree limbs, piping, or other structures that could come in contact with the rig's tower and/or drilling rods or tools.<br><br>2a. Site requirements for raising a tower/derrick around overhead utilities must be reviewed prior to drilling. Maintain **a minimum distance of 10 feet** from overhead structures.<br><br>2a. The tower/derrick must not be raised beneath overhead power lines unless approved by both the Roux and Project PMs. |
| | **2b. CONTACT:** Amputation points occur when raising the rig and the instability of the rig. | 2b. Inspect the equipment prior to use and avoid amputation points when engaging the tower/derrick.<br><br>2b. Lower outriggers to ensure stability prior to raising the rig tower/derrick.<br><br>2b. If the rig needs to be mounted, be sure to use three points of contact. |

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| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **3.** Advancement of soil boring and well installation. | | **If possible, operate the drill rig using a remote control to avoid potential hazards.** |
| | **3a. CONTACT**: Flying debris. | 3a. Be aware of and avoid potential line-of-fire hazards and wear required PPE such as eye, ear, and hand protection. |
| **See also Soil Sampling JSA.** | **3b. EXPOSURE**: Noise and dust. | 3b. Wet borehole area with sprayer to minimize dust.<br>3b. Stand upwind and keep your body away from the rig.<br>3b. A disposable dust mask/respiratory protection (depending on site COCs) should be worn if there is visible dust in the breathing zone.<br>3b. Wear hearing protection when the drill rig is in operation. |
| | **3c. CAUGHT**: Limb/extremity amputation points; abrasion/crushing. | 3c. Always wear cut-resistant gloves (Level II) when making connections, using hand tools, or when handling cutting tools.<br>3c. Inspect the equipment prior to use for potential amputation points. Keep hands away from being between connections, and the use of tools is preferable compared to fingers and hands.<br>3c. Inspect the drill head for worn surface or missing teeth; replace it if damaged or blunt.<br>3c. Ensure all jewelry is removed, loose clothing is secured, and PPE is secured close to the body.<br>3c. All non-essential personnel should remain outside the heavy equipment exclusion zone, which is at least as far as the boom is high (**minimum exclusion zone based on swing/tip radius**).<br>3c. Drillers and helpers will understand and use the “Show Me Your Hands” Policy. Operators and helpers will remain a **minimum of 3 feet** from augers/drill rods while in operation. |
| | **3d. CONTACT/CRUSH**: Crushed between equipment due to an imbalance during the advancement of the drill equipment. | 3d. Drillers will advance the borehole with caution to avoid causing the rig to become imbalanced and/or tip.<br>3d. The blocking and leveling devices used to secure the rig will be inspected by drillers and Roux personnel regularly to see if shifting has occurred.<br>3d. In addition, personnel and equipment that are non-essential to the advancement of the borehole will be positioned away from the rig at a distance that is at least as far as the boom is high (**minimum exclusion zone based on swing/tip radius**). |
| | **3e. EXPOSURE**: Inhalation of contamination/vapors. | 3e. Air monitoring using a calibrated photoionization detector (PID) will be used to monitor the breathing zone of the work area.<br>3e. If readings above the site-specific action levels, the Roux field personnel must temporarily cease work, instruct all Site personnel to step away from the area of elevated readings, and inform the Roux PM of the condition. The Roux PM will then recommend additional precautions in accordance with the site-specific health and safety plan. |
| | **3f. FALL**: Slip/trip/fall hazards. | 3f. Contain drill cuttings and drilling water to prevent slip/trip/fall hazards from developing in the work area.<br>3f. See 1b. |
| | **3g. EXERTION:** Potential for muscle strain/injury while installing well casings and/or lifting sonic rods/casings. | 3g. Keep back straight and bend at the knees.<br>3g. Utilize team lifting for objects over 40 lbs.<br>3g. Use a mechanical lifting device for odd-shaped objects. |

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| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **4.** Decontaminate equipment. | **4a. EXPOSURE:** Contamination (e.g., Separate Phase Hydrocarbons (SPH), contaminated groundwater, vapors, chemicals in cleaning supplies). | 4a. Wear chemical-resistant gloves and safety glasses.<br>4a. Contain decontamination water so that it does not spill.<br>4a. Use an absorbent pad to clean spills, if necessary.<br>4a. See 3b. |

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|---|---|---|---|---|

| JSA TYPE CATEGORY | WORK TYPE | WORK ACTIVITY (Description) |
|---|---|---|
| **Generic** | **Construction** | **Spotting Heavy Machinery** |

**REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**

| | | | |
|---|---|---|---|
| ☐ LIFE VEST | ☐ GOGGLES | ☐ Particulate Respirator | ☒ GLOVES: Cut resistant Level II |
| ☒ HARD HAT | ☐ FACE SHIELD | ☐ SUPPLIED RESPIRATOR | ☐ OTHER: |
| ☒ LONG SLEEVED SHIRT | ☒ HEARING PROTECTION | ☒ PPE CLOTHING: Fluorescent reflective clothing/safety vest | |
| ☐ LIFELINE / BODY HARNESS | ☒ SAFETY SHOES: Steel-/Composite-toe boots | | |
| ☒ SAFETY GLASSES | | | |

**REQUIRED AND / OR RECOMMENDED EQUIPMENT**

Heavy Machinery (i.e., excavator, payloader, truck, forklift, etc.), two-way radios.

**COMMITMENT TO SAFETY-** All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing LMRAs

**EXCLUSION ZONE (EZ): Maintain a Minimum Heavy Equipment Exclusion Zone around equipment and loads while it is in motion. The HEEZ must be greater than the swing zone of any moving part of the equipment, tip zone of the equipment, fall zone of the equipment and contents, distance that debris may travel during demolition activities, and/or footprint of a structure to be demolished.**

| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **1.** Prepare for machine activity. | **1a. CONTACT:** Obstructions in the work area may create contact hazards from machinery.<br><br>**1b. Fall :** Slip/Trip/Fall | 1a. Cordon off the work area with safety barrels/cones and a rigid barrier (snow fence, traffic bar, etc.). Ensure that only necessary personnel are in the work area. Spotter and equipment operator shall enforce the Exclusion Zone (**EZ**). The operator will not operate but shall remain in the hands-off mode while personnel are within the exclusion zone.<br><br>1b. Ensure that the work area is flat, level, and clear of any obstructions or debris before setting up the work zone. |
| **2.** Spotting. | **2a. CONTACT:** Machine or load contact with personnel, property, or machinery. | 2a. Discuss the specifics of the work with the operator and be clear about any hand signals that will be used. Clearly discuss the limits of the assigned work area and the machine's **EZ**. Maintain **EZ**. The **EZ** shall be delineated by using traffic cones/barrels and a fixed rigid barrier.<br>2a. The Minimum Heavy Equipment **EZ** is greater than the swing/tip radius of the equipment.<br><br>2a. Both the spotter and equipment operators shall have verbal/ visual contact and/or 2-way radios/cellular devices on their persons to ensure audible communication in the event any changes or new hazards may arise.<br><br>2a. All workers should stay outside of the **EZ** of all equipment unless the operator is stopped and in "Hands Off" mode. **(This includes the spotter unless an exception has been established in the Site-specific JSA.)** If the **EZ** must be reduced due to work area restrictions, then the spotter and operator shall enforce the reduced **EZ**.<br><br>2a. Spotters must make eye contact with the machine operator, or all movement ceases until visual contact can be reestablished.<br><br>2a. Spotter shall keep an eye out for any issues with the machine that the operator may not see and communicate with other work crews and spotters on behalf of the operator.<br><br>2a. If the spotter needs to take a break, he must find a replacement before leaving or have the machine stop operations. **No heavy equipment shall operate without a spotter under any circumstances.**<br><br>2a. Wear fluorescent clothing/safety vest.<br><br>2a. **Do not multitask. Only perform Spotting** |

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| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| | **2b. FALL:**<br>Slip/Trip/Fall | 2b. Look where you are walking to identify and avoid slip/trip/fall hazards. Avoid icy and/or wet surfaces. Remove obstacles if possible.<br>2b. Use designated walkways during spotting whenever possible.<br>2b. **Do not walk backwards. Always face the direction towards which you are walking.** |
| | **2c. CAUGHT:**<br>Caught between machinery and nearby objects. | 2c. **Maintain EZ.** Do not stand between large, loose, or fixed objects or structures and the machinery while it is in motion. Keep the operator in sight at all times while being aware of surrounding structures. |
| | **2d. EXPOSURE:**<br>Inhalation of exhaust from machinery. | 2d. The spotter will position him/herself upwind of the working machinery, when possible. Spotter will also inform others working within the vicinity of the **EZ** of proper positioning, if applicable. |

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| JOB SAFETY ANALYSIS | Ctrl. No. GEN-029 | DATE: 05/29/2025 | ☐ NEW ☒ REVISED | PAGE 1 of 2 |
|---|---|---|---|---|

| JSA TYPE CATEGORY | WORK TYPE | WORK ACTIVITY (Description) |
|---|---|---|
| **GENERIC** | **Preclearing/Sampling** | **Hand Augering** |

**REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**

| | | | |
|---|---|---|---|
| ☐ LIFE VEST<br>☒ HARD HAT<br>☐ LIFELINE / BODY HARNESS<br>SAFETY GLASSES | ☐ GOGGLES<br>☐ FACE SHIELD<br>☐ HEARING PROTECTION<br>☒ SAFETY SHOES: Steel or composite-toed boots | ☐ AIR PURIFYING RESPIRATOR<br>☐ SUPPLIED RESPIRATOR<br>☒ PPE CLOTHING: Long-sleeved high-visibility clothing | ☒ GLOVES: Cut Resistant level II / Nitrile / Chemical resistant<br>☐ OTHER |

**REQUIRED AND / OR RECOMMENDED EQUIPMENT**

Hand Auger Tools (buckets, rods), 5-gallon buckets, hand tools (hammer, etc.)

**COMMITMENT TO SAFETY-** All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing LMRAs.

| Assess [1] JOB STEPS | Analyze [2] POTENTIAL HAZARDS | Act [3] CRITICAL ACTIONS |
|---|---|---|
| **1.** Drive/walk to hand clearing/hand augering location | **1a. CONTACT:** Property damage and personal injury caused by obstructions/vehicles | 1a. Maintain speed limit on-site.<br>1a. All equipment must be stowed and secured prior to moving.<br>1a. Drive on established roadways.<br>1a. Do not back up the vehicle without a spotter where visibility is limited; use pull-through spots or back into parking spots; use an audible signal (horn/back-up alarm) when backing up vehicles. |
| | **1b. FALL:** Personal injury from tripping/falling due to uneven terrain, weather conditions, and materials/equipment stored at portions of the Site | 1b. Inspect walking path for uneven terrain, weather-related hazards (i.e., ice, puddles, snow, etc.), and obstructions prior to mobilizing equipment.<br>1b. Do not climb over stored materials/equipment; walk around. Use established pathways and walk on stable, secure ground.<br>1b. Use the established ramp when descending into/ascending from impoundment areas.<br>1b. Keep tools and equipment in a designated area. When not in use, tools and equipment must be returned to their proper storage location. Keep the work area clear of obstructions. |
| | **1c. EXERTION:** Muscle strain/exhaustion while carrying equipment (i.e., hand auger, post-hole digger, shovel, pry bar) | 1c. When carrying equipment to/from the work area, use proper lifting techniques; keep back straight, lift with legs, keep the load close to the body, never reach with a load. Ensure that loads are balanced to reduce the potential for muscle strain. Use mechanical assistance or make multiple trips to carry equipment. |
| | **1d. EXPOSURE:** Exposure to the sun, possibly causing sunburn. Biological hazards - bees/wasps, poison oak, thorns, insects, etc. | 1d. Wear sunscreen with an SPF of at least 15 whenever 30 minutes or more of exposure is expected.<br>1d. Inspect the area to avoid contact with biological hazards.<br>1d. Wear cut-resistant gloves when handling material, debris, branches, shrubs, etc. that may lie within the walking path.<br>1d. Avoid any areas on-site that have poison oak. |
| **Assess [1] JOB STEPS** | **Analyze [2] POTENTIAL HAZARDS** | **Act [3] CRITICAL ACTIONS** |
| **2.** Secure location | **2a. CONTACT:** Personnel and vehicular traffic may enter the work area. | 2a. Delineate the work area with traffic cones and/or caution tape to prevent exposure to traffic and inform others of work activity.<br>2a. Wear a reflective vest and/or fluorescent clothing.<br>2a. Face the direction of vehicular traffic. Position the vehicle to protect the worker from traffic.<br>2a. Communicate work activity with adjacent work areas. |
| | **2b. FALL:** Tripping/falling due to uneven terrain, and materials /equipment stored within the work area | 2b. Inspect walking path for uneven terrain, weather-related hazards (i.e., ice, puddles, snow, etc.), and obstructions prior to mobilizing equipment to the impoundments.<br>2b. Equipment and tools will be staged in a convenient, stable, and orderly manner.<br>2b. Equipment and tools will be stored at the lowest point of potential energy and out of the walkway and immediate work area (i.e., tools should not be propped against walls or nearby equipment or vehicles).<br>2b. Equipment and tools that are not anticipated to be used will be returned to an appropriate storage area that is out of the immediate work area. |

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[3] Using the first two columns as a guide, decide what actions or procedures are necessary to eliminate or minimize the risk. List the recommended safe operating procedures. Say exactly what needs to be done - such as "use two persons to lift." Avoid general statements such as, "be careful."

| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **3.** Verify pre-clearance protocol; review completed Subsurface Utility Checklist and utility verification site walkthrough. | **3a. ENERGY SOURCE:** Underground utility damage; property damage; personal injury | 3a. Confirm that "Call Before You Dig"/811 and local utility companies were contacted prior to hand augering.<br>3a. Walk the Site to evaluate utility markings and review maps. |
| **4.** Augering/advancing borehole | **4a. EXPOSURE:** Contaminated soil/water/vapor | 4a. Monitor the breathing zone with a PID when VOCs are a concern. If vapors exceed site-specific action levels, upgrade PPE as per the HASP.<br>4a. Wear chemical-resistant disposable gloves and safety glasses when handling impacted materials.<br>4a. Place excavated soil on plastic sheeting and store soil waste in a designated area.<br>4a. Work on the upwind side of the boring. |
| | **4b. EXERTION:** Muscle strain from lifting, bending, repetitive motion. | 4b. Body positioning and rotating with the auger to reduce strain. Do not twist back. |
| | **4c. CAUGHT:** Personal injury as a result of jewelry/loose clothing caught on equipment, well covers, machinery, hand auger, pry bar, etc. | 4c. No form of jewelry should be worn while on-site.<br>4c. Clothing must be appropriately sized so that it is not loose-fitting. |
| | **4d. CONTACT/CAUGHT:** Pinch points, abrasions | 4d. Keep head and upper body clear when lifting hand auger, pry bar/post-hole digger. Ensure not to be overly aggressive when using the pry bar.<br>4d. Ensure all jewelry is removed, loose clothing is secured, and PPE is secured close to the body. |
| **5.** Lithologic observation and soil sampling | **5a. EXPOSURE:** Contact with contamination (impacted soil and/or lab preservatives) | 5a. Wear chemical-resistant disposable gloves to protect hands when handling samples; wear safety glasses when on site and/or goggles when handling any preservatives; use containment material or plastic sheeting to protect surrounding areas.<br>5a. When collecting a soil sample from a hand auger, put a large zip lock bag over the entire auger to prevent the spillage of soil onto the ground.<br>5a. Open sample jars slowly and fill them carefully to avoid contact with preservatives. |
| **6.** Decontaminate equipment | **6a. EXPOSURE:** Contact with contamination (impacted soil and/or lab preservatives, decontamination solution) | 6a. Wear chemical-resistant disposable gloves and safety glasses.<br>6a. Use an absorbent pad to clean spills.<br>6a. Properly dispose of used materials/PPE trash bags.<br>6a. Refer to the SDS for the decontamination solution for proper handling. |
| | **6b. CONTACT/CAUGHT:** pinch points and cuts/abrasions | 6b. Keep fingers/hands out of pinch points when disassembling the hand auger during decontamination. |

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| JOB SAFETY ANALYSIS | Ctrl. No. GEN-032 | DATE: 05/30/2025 | ☐ NEW ☒ REVISED | PAGE 1 of 2 |
|---|---|---|---|---|

| JSA TYPE CATEGORY | WORK TYPE | WORK ACTIVITY (Description) |
|---|---|---|
| **Generic** | **Construction** | **Spotting Third-Party Traffic** |

**REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT**

| | | | |
|---|---|---|---|
| ☐ LIFE VEST | ☐ GOGGLES | ☐ Particulate Respirator | ☒ GLOVES: Cut-resistant Level II |
| ☒ HARD HAT | ☐ FACE SHIELD | ☐ SUPPLIED RESPIRATOR | ☐ OTHER: |
| ☐ LIFELINE / BODY HARNESS | ☐ HEARING PROTECTION | ☒ PPE CLOTHING: Fluorescent reflective clothing | |
| ☒ SAFETY GLASSES | ☒ SAFETY SHOES: Steel-/Composite-toe boots | | |

**REQUIRED EQUIPMENT**

Traffic cones/ barrels, Handheld stop/slow paddles (1 per spotter), two-way radios as needed

**COMMITMENT TO SAFETY-** All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing LMRAs

**EXCLUSION ZONE (EZ): Maintain a Minimum Heavy Equipment Exclusion Zone around equipment, active operations (e.g., excavation, truck loading, etc.), and loads while it is in motion. The HEEZ must be greater than the swing zone of any moving part of the equipment, tip zone of the equipment, fall zone of the equipment and contents, distance that debris may travel during demolition activities, and/or footprint of a structure to be demolished. The HEEZ should not be compromised in any way by third-party traffic.**

| Assess [1]JOB STEPS | Analyze [2]POTENTIAL HAZARDS | Act [3]CRITICAL ACTIONS |
|---|---|---|
| **1.** Prepare for heavy equipment operations and/or job activities that require traffic stoppage. | **1a. CONTACT:** Third-party traffic in and/or adjacent to the work area may create contact hazards with machinery and site personnel. | 1a. Cordon off the work area with safety barrels/cones and a rigid barrier (snow fence, traffic bar, etc.). Ensure that only necessary personnel are in the work area. Spotter and equipment operator shall enforce the **EZ**. The operator will not operate but shall remain in hands-off mode while personnel are within the exclusion zone.<br>• Spotters must agree on communication via voice and hand signals before the activity begins.<br>• Spotters must be visible and maintain contact with other spotters.<br>• If contact is lost with either spotter, work operations should stop until communication has been re-established.<br>• Spotters should never have additional duties while they are spotting.<br>1a. Spotters must analyze traffic patterns adjacent to the site to assess which traffic direction will need to be redirected and what issues must be resolved before spotting traffic.<br>1a. The "Traffic Control Point" is a specific location where the spotter is positioned to direct traffic.<br>1a. **Spotters should select an appropriate Traffic Control Point where visibility, directions, and personal safety will be maximized.** |
| | **1b. Fall :** Slip/Trip/Fall | 1b. Ensure that the Traffic Control Point is flat, level, and clear of any obstructions or debris before beginning spotting operations. |

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| Assess<br>[1]JOB STEPS | Analyze<br>[2]POTENTIAL HAZARDS | Act<br>[3]CRITICAL ACTIONS |
|---|---|---|
| **2.** Spotting third-party traffic during site operations. | **2a. CONTACT:**<br>Machine or load contact with third-party traffic. | 2a. Discuss the specifics of the work, limits of the assigned work area, and the machine's Exclusion Zone. Maintain the Exclusion Zone while spotting third-party traffic.<br><br>2a. **Spotters and equipment operators shall have visual/ verbal contact or 2-way radios/cellular devices on their persons** to ensure audible communication in the event any changes or new hazards may arise.<br><br>2a. Spotters must make eye contact with the machine operations and third-party traffic, or all movement ceases until visual contact can be reestablished.<br><br>2a. Spotter shall keep an eye out for any traffic issues the operator may not see and communicate with other work crews and spotters accordingly.<br><br>2a. If the spotter needs to take a break, he must find a replacement before leaving or stop operations. **Operations that may contact third-party traffic shall not be performed without a spotter under any circumstances.**<br><br>2a. Wear fluorescent clothing/safety vest. **Handheld stop/ slow paddles (1 per spotter) are required for spotting third-party traffic.**<br><br>2a. Establish traffic control by performing all signals/movements with precision and ensuring drivers obey signals.<br>• Break traffic at natural gaps whenever possible to direct opposite sides of traffic<br>• Maintain consistency in the time you allow each direction of traffic to travel. Prioritize major routes over secondary routes.<br>• If breaks do not occur, allow traffic to accumulate on opposite sides of the road before directing it to move.<br>• Allow right turns whenever they do not interfere with traffic and pedestrians.<br>• Make sure opposite lanes are stopped before allowing left turns to be made.<br><br>2a. **Do not multitask. Only perform Spotting.** |
| | **2b. FALL:**<br>Slip/Trip/Fall | 2b. Look where you are walking to identify and avoid slip/trip/fall hazards. Avoid icy and/or wet surfaces. Remove obstacles if possible.<br><br>2b. Use designated walkways during spotting whenever possible.<br><br>2b. **Do not walk backwards. Always face the direction towards which you are walking.** |
| | **2c. CAUGHT:**<br>Caught between machinery and nearby objects. | 2c. **Maintain Exclusion Zone.** Do not stand between large, loose, or fixed objects or structures and the machinery while it is in motion. Keep in sight of the operator at all times while being aware of surrounding structures. |
| | **2d. EXPOSURE:**<br>Inhalation of exhaust from machinery. | 2d. The spotter will position themself upwind of the working machinery, when possible. Spotter will also inform others working within the vicinity of the EZ of proper positioning, if applicable. |

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# APPENDIX B

SDSs for Chemicals Used

# Safety Data Sheet

according to 1907/2006/EC (REACH), 1272/2008/EC (CLP), 29CFR1910/1200 and GHS Rev. 3

**Effective date**: 12.08.2015 **Revision** : 12.10.2015

**Trade Name**: Alconox

## 1 Identification of the substance/mixture and of the supplier

### 1.1 Product identifier

**Trade Name:** Alconox
**Synonyms:**
**Product number:** Alconox

### 1.2 Application of the substance / the mixture : Cleaning material/Detergent

### 1.3 Details of the supplier of the Safety Data Sheet

**Manufacturer**
Alconox, Inc.
30 Glenn Street
White Plains, NY 10603
1-914-948-4040

**Supplier**
Not Applicable

**Emergency telephone number:**
**ChemTel Inc**
North America: 1-800-255-3924
International: 01-813-248-0585

## 2 Hazards identification

### 2.1 Classification of the substance or mixture:

In compliance with EC regulation No. 1272/2008, 29CFR1910/1200 and GHS Rev. 3 and amendments.

**Hazard-determining components of labeling:**
Tetrasodium Pyrophosphate
Sodium tripolyphosphate
Sodium Alkylbenzene Sulfonate

### 2.2 Label elements:

Skin irritation, category 2.
Eye irritation, category 2A.

**Hazard pictograms:**



**Signal word**: Warning

**Hazard statements:**
H315 Causes skin irritation.
H319 Causes serious eye irritation.

**Precautionary statements:**
P264 Wash skin thoroughly after handling.
P280 Wear protective gloves/protective clothing/eye protection/face protection.
P302+P352 If on skin: Wash with soap and water.
P305+P351+P338 If in eyes: Rinse cautiously with water for several minutes. Remove contact lenses if present and easy to do. Continue rinsing.
P321 Specific treatment (see supplemental first aid instructions on this label).
P332+P313 If skin irritation occurs: Get medical advice/attention.
P362 Take off contaminated clothing and wash before reuse.
P501 Dispose of contents and container as instructed in Section 13.

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**Additional information:** None.

**Hazard description**

**Hazards Not Otherwise Classified (HNOC):** None

**Information concerning particular hazards for humans and environment:**

The product has to be labelled due to the calculation procedure of the "General Classification guideline for preparations of the EU" in the latest valid version.

**Classification system:**

The classification is according to EC regulation No. 1272/2008, 29CFR1910/1200 and GHS Rev. 3 and amendments, and extended by company and literature data. The classification is in accordance with the latest editions of international substances lists, and is supplemented by information from technical literature and by information provided by the company.

## 3 Composition/information on ingredients

**3.1 Chemical characterization** : None

**3.2 Description** : None

**3.3 Hazardous components (percentages by weight)**

| Identification | Chemical Name | Classification | Wt. % |
|---|---|---|---|
| **CAS number:** 7758-29-4 | Sodium tripolyphosphate | Skin Irrit. 2 ; H315<br>Eye Irrit. 2; H319 | 12-28 |
| **CAS number:** 68081-81-2 | Sodium Alkylbenzene Sulfonate | Acute Tox. 4; H303<br>Skin Irrit. 2 ; H315<br>Eye Irrit. 2; H319 | 8-22 |
| **CAS number:** 7722-88-5 | Tetrasodium Pyrophosphate | Skin Irrit. 2 ; H315<br>Eye Irrit. 2; H319 | 2-16 |

**3.4 Additional Information** : None.

## 4 First aid measures

**4.1 Description of first aid measures**

**General information:** None.

**After inhalation:**

Maintain an unobstructed airway.
Loosen clothing as necessary and position individual in a comfortable position.

**After skin contact:**

Wash affected area with soap and water.
Seek medical attention if symptoms develop or persist.

**After eye contact:**

Rinse/flush exposed eye(s) gently using water for 15-20 minutes.
Remove contact lens(es) if able to do so during rinsing.
Seek medical attention if irritation persists or if concerned.

**After swallowing:**

Rinse mouth thoroughly.
Seek medical attention if irritation, discomfort, or vomiting persists.

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**4.2 Most important symptoms and effects, both acute and delayed**

None

**4.3 Indication of any immediate medical attention and special treatment needed:**

No additional information.

## 5 Firefighting measures

**5.1 Extinguishing media**

**Suitable extinguishing agents:**

Use appropriate fire suppression agents for adjacent combustible materials or sources of ignition.

**For safety reasons unsuitable extinguishing agents** : None

**5.2 Special hazards arising from the substance or mixture** :

Thermal decomposition can lead to release of irritating gases and vapors.

**5.3 Advice for firefighters**

**Protective equipment:**

Wear protective eye wear, gloves and clothing.
Refer to Section 8.

**5.4 Additional information** :

Avoid inhaling gases, fumes, dust, mist, vapor and aerosols.
Avoid contact with skin, eyes and clothing.

## 6 Accidental release measures

**6.1 Personal precautions, protective equipment and emergency procedures** :

Ensure adequate ventilation.
Ensure air handling systems are operational.

**6.2 Environmental precautions** :

Should not be released into the environment.
Prevent from reaching drains, sewer or waterway.

**6.3 Methods and material for containment and cleaning up** :

Wear protective eye wear, gloves and clothing.

**6.4 Reference to other sections** : None

## 7 Handling and storage

**7.1 Precautions for safe handling** :

Avoid breathing mist or vapor.
Do not eat, drink, smoke or use personal products when handling chemical substances.

**7.2 Conditions for safe storage, including any incompatibilities** :

Store in a cool, well-ventilated area.

**7.3 Specific end use(s):**

No additional information.

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## 8 Exposure controls/personal protection





### 8.1 Control parameters :

7722-88-5, Tetrasodium Pyrophosphate, OSHA TWA 5 mg/m3.

### 8.2 Exposure controls

**Appropriate engineering controls:**

Emergency eye wash fountains and safety showers should be available in the immediate vicinity of use or handling.

**Respiratory protection:**

Not needed under normal conditions.

**Protection of skin:**

Select glove material impermeable and resistant to the substance.

**Eye protection:**

Safety goggles or glasses, or appropriate eye protection.

**General hygienic measures:**

Wash hands before breaks and at the end of work.
Avoid contact with skin, eyes and clothing.

## 9 Physical and chemical properties

| | | | |
|---|---|---|---|
| **Appearance (physical state, color):** | White and cream colored flakes - powder | **Explosion limit lower**: **Explosion limit upper**: | Not determined or not available. Not determined or not available. |
| **Odor**: | Not determined or not available. | **Vapor pressure at 20°C**: | Not determined or not available. |
| **Odor threshold**: | Not determined or not available. | **Vapor density**: | Not determined or not available. |
| **pH-value**: | 9.5 (aqueous solution) | **Relative density**: | Not determined or not available. |
| **Melting/Freezing point**: | Not determined or not available. | **Solubilities**: | Not determined or not available. |
| **Boiling point/Boiling range**: | Not determined or not available. | **Partition coefficient (n-octanol/water)**: | Not determined or not available. |
| **Flash point (closed cup)**: | Not determined or not available. | **Auto/Self-ignition temperature**: | Not determined or not available. |
| **Evaporation rate**: | Not determined or not available. | **Decomposition temperature**: | Not determined or not available. |

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| | | | |
|---|---|---|---|
| **Flammability (solid, gaseous)**: | Not determined or not available. | **Viscosity**: | a. Kinematic: Not determined or not available.<br>b. Dynamic: Not determined or not available. |
| **Density at 20°C**: | Not determined or not available. | | |

## 10 Stability and reactivity

**10.1 Reactivity** : None

**10.2 Chemical stability** : None

**10.3 Possibility hazardous reactions** : None

**10.4 Conditions to avoid** : None

**10.5 Incompatible materials** : None

**10.6 Hazardous decomposition products** : None

## 11 Toxicological information

**11.1 Information on toxicological effects** :

**Acute Toxicity:**

**Oral**:

: LD50 > 5000 mg/kg oral rat - Product .

**Chronic Toxicity:** No additional information.

**Skin corrosion/irritation:**

Sodium Alkylbenzene Sulfonate: Causes skin irritation. .

**Serious eye damage/irritation:**

Sodium Alkylbenzene Sulfonate: Causes serious eye irritation .

Tetrasodium Pyrophosphate: Rabbit - Risk of serious damage to eyes .

**Respiratory or skin sensitization:** No additional information.

**Carcinogenicity:** No additional information.

**IARC (International Agency for Research on Cancer):** None of the ingredients are listed.

**NTP (National Toxicology Program):** None of the ingredients are listed.

**Germ cell mutagenicity:** No additional information.

**Reproductive toxicity:** No additional information.

**STOT-single and repeated exposure:** No additional information.

**Additional toxicological information:** No additional information.

## 12 Ecological information

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**12.1 Toxicity:**

Sodium Alkylbenzene Sulfonate: Fish, LC50 1.67 mg/l, 96 hours.
Sodium Alkylbenzene Sulfonate: Aquatic invertebrates, EC50 Daphnia 2.4 mg/l, 48 hours.
Sodium Alkylbenzene Sulfonate: Aquatic Plants, EC50 Algae 29 mg/l, 96 hours.
Tetrasodium Pyrophosphate: Fish, LC50 - other fish - 1,380 mg/l - 96 h.
Tetrasodium Pyrophosphate: Aquatic invertebrates, EC50 - Daphnia magna (Water flea) - 391 mg/l - 48 h.

**12.2 Persistence and degradability:** No additional information.

**12.3 Bioaccumulative potential:** No additional information.

**12.4 Mobility in soil:** No additional information.

**General notes:** No additional information.

**12.5 Results of PBT and vPvB assessment:**

**PBT:** No additional information.

**vPvB:** No additional information.

**12.6 Other adverse effects:** No additional information.

## 13 Disposal considerations

**13.1 Waste treatment methods (consult local, regional and national authorities for proper disposal)**

**Relevant Information:**

It is the responsibility of the waste generator to properly characterize all waste materials according to applicable regulatory entities. (US 40CFR262.11).

## 14 Transport information

**14.1 UN Number:** None
ADR, ADN, DOT, IMDG, IATA

**14.2 UN Proper shipping name:** None
ADR, ADN, DOT, IMDG, IATA

**14.3 Transport hazard classes:**
ADR, ADN, DOT, IMDG, IATA

**Class:** None
**Label:** None
**LTD. QTY:** None

**US DOT**
**Limited Quantity Exception:** None

**Bulk:**
**RQ (if applicable):** None
**Proper shipping Name:** None
**Hazard Class:** None
**Packing Group:** None
**Marine Pollutant (if applicable):** No additional information.

**Non Bulk:**
**RQ (if applicable):** None
**Proper shipping Name:** None
**Hazard Class:** None
**Packing Group:** None
**Marine Pollutant (if applicable):** No additional information.

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| | | |
|---|---|---|
| | **Comments:** None | **Comments:** None |
| **14.4** | **Packing group:**<br>ADR, ADN, DOT, IMDG, IATA | None |
| **14.5** | **Environmental hazards** : | None |
| **14.6** | **Special precautions for user:** | None |
| | **Danger code (Kemler):** | None |
| | **EMS number:** | None |
| | **Segregation groups:** | None |
| **14.7** | **Transport in bulk according to Annex II of MARPOL73/78 and the IBC Code:** Not applicable. | |
| **14.8** | **Transport/Additional information:** | |
| | **Transport category:** | None |
| | **Tunnel restriction code:** | None |
| | **UN "Model Regulation":** | None |

## 15 Regulatory information

### 15.1 Safety, health and environmental regulations/legislation specific for the substance or mixture.

#### North American

**SARA**

**Section 313 (specific toxic chemical listings)**: None of the ingredients are listed.
**Section 302 (extremely hazardous substances)**: None of the ingredients are listed.

**CERCLA (Comprehensive Environmental Response, Clean up and Liability Act) Reportable**

**Spill Quantity**: None of the ingredients are listed.

**TSCA (Toxic Substances Control Act):**

**Inventory**: All ingredients are listed.
**Rules and Orders**: Not applicable.

**Proposition 65 (California):**

**Chemicals known to cause cancer**: None of the ingredients are listed.

**Chemicals known to cause reproductive toxicity for females**: None of the ingredients are listed.

**Chemicals known to cause reproductive toxicity for males**: None of the ingredients are listed.

**Chemicals known to cause developmental toxicity**: None of the ingredients are listed.

**Canadian**

**Canadian Domestic Substances List (DSL)**:
All ingredients are listed.

#### EU

**REACH Article 57 (SVHC)**: None of the ingredients are listed.

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**Germany MAK**: Not classified.

### Asia Pacific

**Australia**

**Australian Inventory of Chemical Substances (AICS)**: All ingredients are listed.

**China**

**Inventory of Existing Chemical Substances in China (IECSC)**: All ingredients are listed.

**Japan**

**Inventory of Existing and New Chemical Substances (ENCS)**: All ingredients are listed.

**Korea**

**Existing Chemicals List (ECL)**: All ingredients are listed.

**New Zealand**

**New Zealand Inventory of Chemicals (NZOIC)**: All ingredients are listed.

**Philippines**

**Philippine Inventory of Chemicals and Chemical Substances (PICCS)**: All ingredients are listed.

**Taiwan**

**Taiwan Chemical Substance Inventory (TSCI)**: All ingredients are listed.

## 16 Other information

**Abbreviations and Acronyms:** None

**Summary of Phrases**

**Hazard statements:**

H315 Causes skin irritation.
H319 Causes serious eye irritation.

**Precautionary statements:**

P264 Wash skin thoroughly after handling.
P280 Wear protective gloves/protective clothing/eye protection/face protection.
P302+P352 If on skin: Wash with soap and water.
P305+P351+P338 If in eyes: Rinse cautiously with water for several minutes. Remove contact lenses if present and easy to do. Continue rinsing.
P321 Specific treatment (see supplemental first aid instructions on this label).
P332+P313 If skin irritation occurs: Get medical advice/attention.
P362 Take off contaminated clothing and wash before reuse.
P501 Dispose of contents and container as instructed in Section 13.

**Manufacturer Statement:**

The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text.

**NFPA:** 1-0-0

**Safety Data Sheet**

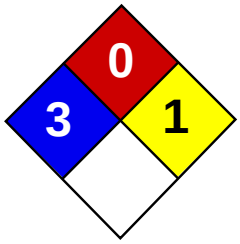
according to 1907/2006/EC (REACH), 1272/2008/EC (CLP), 29CFR1910/1200 and GHS Rev. 3

**Effective date**: 12.08.2015 **Revision** : 12.10.2015

**Trade Name**: Alconox

**HMIS:** 1-0-0







# Material Safety Data Sheet
## Hydrochloric acid MSDS

## Section 1: Chemical Product and Company Identification

**Product Name:** Hydrochloric acid

**Catalog Codes:** SLH1462, SLH3154

**CAS#:** Mixture.

**RTECS:** MW4025000

**TSCA:** TSCA 8(b) inventory: Hydrochloric acid

**CI#:** Not applicable.

**Synonym:** Hydrochloric Acid; Muriatic Acid

**Chemical Name:** Not applicable.

**Chemical Formula:** Not applicable.

**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 |
|---|---|---|
| Hydrogen chloride | 7647-01-0 | 20-38 |
| Water | 7732-18-5 | 62-80 |

**Toxicological Data on Ingredients:** Hydrogen chloride: GAS (LC50): Acute: 4701 ppm 0.5 hours [Rat].

## Section 3: Hazards Identification

**Potential Acute Health Effects:**
Very hazardous in case of skin contact (corrosive, irritant, permeator), of eye contact (irritant, corrosive), of ingestion, . Slightly hazardous in case of inhalation (lung sensitizer). Non-corrosive for lungs. Liquid or spray mist may produce tissue damage particularly on mucous membranes of eyes, mouth and respiratory tract. Skin contact may produce burns. Inhalation of the spray mist may produce severe irritation of respiratory tract, characterized by coughing, choking, or shortness of breath. Severe over-exposure can result in death. Inflammation of the eye is characterized by redness, watering, and itching. Skin inflammation is characterized by itching, scaling, reddening, or, occasionally, blistering.

**Potential Chronic Health Effects:**
Slightly hazardous in case of skin contact (sensitizer). CARCINOGENIC EFFECTS: Classified 3 (Not classifiable for human.) by IARC [Hydrochloric acid]. MUTAGENIC EFFECTS: Not available. TERATOGENIC EFFECTS: Not available. DEVELOPMENTAL TOXICITY: Not available. The substance may be toxic to kidneys, liver, mucous membranes, upper respiratory tract, skin, eyes, Circulatory System, teeth. Repeated or prolonged exposure to the substance can produce target

organs damage. Repeated or prolonged contact with spray mist may produce chronic eye irritation and severe skin irritation. Repeated or prolonged exposure to spray mist may produce respiratory tract irritation leading to frequent attacks of bronchial infection. Repeated exposure to a 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. In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Cold water may be used. Get medical attention immediately.

**Skin Contact:**
In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Cover the irritated skin with an emollient. Cold water may be used.Wash clothing before reuse. Thoroughly clean shoes before reuse. Get medical attention immediately.

**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 immediately.

**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:**
If swallowed, do not induce vomiting unless directed to do so by medical personnel. Never give anything by mouth to an unconscious person. Loosen tight clothing such as a collar, tie, belt or waistband. Get medical attention immediately.

**Serious Ingestion:** Not available.

## Section 5: Fire and Explosion Data

**Flammability of the Product:** Non-flammable.

**Auto-Ignition Temperature:** Not applicable.

**Flash Points:** Not applicable.

**Flammable Limits:** Not applicable.

**Products of Combustion:** Not available.

**Fire Hazards in Presence of Various Substances:** of metals

**Explosion Hazards in Presence of Various Substances:** Non-explosive in presence of open flames and sparks, of shocks.

**Fire Fighting Media and Instructions:** Not applicable.

**Special Remarks on Fire Hazards:**
Non combustible. Calcium carbide reacts with hydrogen chloride gas with incandescence. Uranium phosphide reacts with hydrochloric acid to release spontaneously flammable phosphine. Rubidium acetylene carbides burns with slightly warm hydrochloric acid. Lithium silicide in contact with hydrogen chloride becomes incandescent. When dilute hydrochloric acid is used, gas spontaneously flammable in air is evolved. Magnesium boride treated with concentrated hydrochloric acid produces spontaneously flammble gas. Cesium acetylene carbide burns hydrogen chloride gas. Cesium carbide ignites in contact with hydrochloric acid unless acid is dilute. Reacts with most metals to produce flammable Hydrodgen gas.

**Special Remarks on Explosion Hazards:**

Hydrogen chloride in contact with the following can cause an explosion, ignition on contact, or other violent/vigorous reaction: Acetic anhydride AgClO + CCl4 Alcohols + hydrogen cyanide, Aluminum Aluminum-titanium alloys (with HCl vapor), 2-Amino ethanol, Ammonium hydroxide, Calcium carbide Ca3P2 Chlorine + dinitroanilines (evolves gas), Chlorosulfonic acid Cesium carbide Cesium acetylene carbide, 1,1-Difluoroethylene Ethylene diamine Ethylene imine, Fluorine, HClO4 Hexalithium disilicide H2SO4 Metal acetylides or carbides, Magnesium boride, Mercuric sulfate, Oleum, Potassium permanganate, beta-Propiolactone Propylene oxide Rubidium carbide, Rubidium, acetylene carbide Sodium (with aqueous HCl), Sodium hydroxide Sodium tetraselenium, Sulfonic acid, Tetraselenium tetranitride, U3P4 , Vinyl acetate. Silver perchlorate with carbon tetrachloride in the presence of hydrochloric acid produces trichloromethyl perchlorate which detonates at 40 deg. C.

## Section 6: Accidental Release Measures

**Small Spill:**
Dilute with water and mop up, or absorb with an inert dry material and place in an appropriate waste disposal container. If necessary: Neutralize the residue with a dilute solution of sodium carbonate.

**Large Spill:**
Corrosive liquid. Poisonous liquid. 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. Use water spray curtain to divert vapor drift. Use water spray to reduce vapors. Prevent entry into sewers, basements or confined areas; dike if needed. Call for assistance on disposal. Neutralize the residue with a dilute solution of sodium carbonate. 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 container dry. Do not ingest. Do not breathe gas/fumes/ vapor/spray. Never add water to this product. 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, organic materials, metals, alkalis, moisture. May corrode metallic surfaces. Store in a metallic or coated fiberboard drum using a strong polyethylene inner package.

**Storage:** Keep container tightly closed. Keep container in a cool, well-ventilated area.

## 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:**
Face shield. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Gloves. Boots.

**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:**
CEIL: 5 (ppm) from OSHA (PEL) [United States] CEIL: 7 (mg/m3) from OSHA (PEL) [United States] CEIL: 5 from NIOSH CEIL: 7 (mg/m3) from NIOSH TWA: 1 STEL: 5 (ppm) [United Kingdom (UK)] TWA: 2 STEL: 8 (mg/m3) [United Kingdom (UK)]Consult local authorities for acceptable exposure limits.

## Section 9: Physical and Chemical Properties

**Physical state and appearance:** Liquid.

**Odor:** Pungent. Irritating (Strong.)

**Taste:** Not available.

**Molecular Weight:** Not applicable.

**Color:** Colorless to light yellow.

**pH (1% soln/water):** Acidic.

**Boiling Point:**
108.58 C @ 760 mm Hg (for 20.22% HCl in water) 83 C @ 760 mm Hg (for 31% HCl in water) 50.5 C (for 37% HCl in water)

**Melting Point:**
-62.25°C (-80°F) (20.69% HCl in water) -46.2 C (31.24% HCl in water) -25.4 C (39.17% HCl in water)

**Critical Temperature:** Not available.

**Specific Gravity:**
1.1- 1.19 (Water = 1) 1.10 (20%and 22% HCl solutions) 1.12 (24% HCl solution) 1.15 (29.57% HCl solution) 1.16 (32% HCl solution) 1.19 (37% and 38%HCl solutions)

**Vapor Pressure:** 16 kPa (@ 20°C) average

**Vapor Density:** 1.267 (Air = 1)

**Volatility:** Not available.

**Odor Threshold:** 0.25 to 10 ppm

**Water/Oil Dist. Coeff.:** Not available.

**Ionicity (in Water):** Not available.

**Dispersion Properties:** See solubility in water, diethyl ether.

**Solubility:** Soluble in cold water, hot water, diethyl ether.

## Section 10: Stability and Reactivity Data

**Stability:** The product is stable.

**Instability Temperature:** Not available.

**Conditions of Instability:** Incompatible materials, water

**Incompatibility with various substances:**
Highly reactive with metals. Reactive with oxidizing agents, organic materials, alkalis, water.

**Corrosivity:**
Extremely corrosive in presence of aluminum, of copper, of stainless steel(304), of stainless steel(316). Non-corrosive in presence of glass.

**Special Remarks on Reactivity:**
Reacts with water especially when water is added to the product. Absorption of gaseous hydrogen chloride on mercuric sulfate becomes violent @ 125 deg. C. Sodium reacts very violently with gaseous hydrogen chloride. Calcium phosphide and hydrochloric acid undergo very energetic reaction. It reacts with oxidizers releasing chlorine gas. Incompatible with, alkali metals, carbides, borides, metal oxides, vinyl acetate, acetylides, sulphides, phosphides, cyanides, carbonates. Reacts with most metals to produce flammable Hydrogen gas. Reacts violently (moderate reaction with heat of evolution) with water especially when water is added to the product. Isolate hydrogen chloride from heat, direct sunlight, alkalies (reacts vigorously), organic materials, and oxidizers (especially nitric acid and chlorates), amines, metals, copper and alloys (e.g. brass), hydroxides, zinc (galvanized materials), lithium silicide (incandescence), sulfuric acid(increase in temperature and pressure) Hydrogen chloride gas is emitted when this product is in contact with sulfuric acid. Adsorption of Hydrochloric Acid onto silicon dioxide results in exothmeric reaction. Hydrogen chloride causes aldehydes and epoxides to violently polymerize. Hydrogen chloride or Hydrochloric Acid in contact with the folloiwng can cause explosion or ignition on contact or

**Special Remarks on Corrosivity:**

Highly corrosive. Incompatible with copper and copper alloys. It attacks nearly all metals (mercury, gold, platinium, tantalum, silver, and certain alloys are exceptions). It is one of the most corrosive of the nonoxidizing acids in contact with copper alloys. No corrosivity data on zinc, steel. Severe Corrosive effect on brass and bronze

**Polymerization:** Will not occur.

## Section 11: Toxicological Information

**Routes of Entry:** Absorbed through skin. Dermal contact. Eye contact. Inhalation.

**Toxicity to Animals:**
Acute oral toxicity (LD50): 900 mg/kg [Rabbit]. Acute toxicity of the vapor (LC50): 1108 ppm, 1 hours [Mouse]. Acute toxicity of the vapor (LC50): 3124 ppm, 1 hours [Rat].

**Chronic Effects on Humans:**
CARCINOGENIC EFFECTS: Classified 3 (Not classifiable for human.) by IARC [Hydrochloric acid]. May cause damage to the following organs: kidneys, liver, mucous membranes, upper respiratory tract, skin, eyes, Circulatory System, teeth.

**Other Toxic Effects on Humans:**
Very hazardous in case of skin contact (corrosive, irritant, permeator), of ingestion, . Hazardous in case of eye contact (corrosive), of inhalation (lung corrosive).

**Special Remarks on Toxicity to Animals:**
Lowest Published Lethal Doses (LDL/LCL) LDL [Man] -Route: Oral; 2857 ug/kg LCL [Human] - Route: Inhalation; Dose: 1300 ppm/30M LCL [Rabbit] - Route: Inhalation; Dose: 4413 ppm/30M

**Special Remarks on Chronic Effects on Humans:**
May cause adverse reproductive effects (fetoxicity). May affect genetic material.

**Special Remarks on other Toxic Effects on Humans:**
Acute Potential Health Effects: Skin: Corrosive. Causes severe skin irritation and burns. Eyes: Corrosive. Causes severe eye irritation/conjuntivitis, burns, corneal necrosis. Inhalation: May be fatal if inhaled. Material is extremely destructive to tissue of the mucous membranes and upper respiratory tract. Inhalation of hydrochloric acid fumes produces nose, throat, and larryngeal burning, and irritation, pain and inflammation, coughing, sneezing, choking sensation, hoarseness, laryngeal spasms, upper respiratory tract edema, chest pains, as well has headache, and palpitations. Inhalation of high concentrations can result in corrosive burns, necrosis of bronchial epithelium, constriction of the larynx and bronchi, nasospetal perforation, glottal closure,  occur, particularly if exposure is prolonged. May affect the liver. Ingestion: May be fatal if swallowed. Causes irritation and burning, ulceration, or perforation of the gastrointestinal tract and resultant peritonitis, gastric hemorrhage and infection. Can also cause nausea, vomitting (with "coffee ground" emesis), diarrhea, thirst, difficulty swallowing, salivation, chills, fever, uneasiness, shock, strictures and stenosis (esophogeal, gastric, pyloric). May affect behavior (excitement), the cardiovascular system (weak rapid pulse, tachycardia), respiration (shallow respiration), and urinary system (kidneys- renal failure, nephritis). Acute exposure via inhalation or ingestion can also cause erosion of tooth enamel. Chronic Potential Health Effects: dyspnea, bronchitis. Chemical pneumonitis and pulmonary edema can also

## 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 8: Corrosive material

**Identification:** : Hydrochloric acid, solution UNNA: 1789 PG: II

**Special Provisions for Transport:** Not available.

## Section 15: Other Regulatory Information

**Federal and State Regulations:**
Connecticut hazardous material survey.: Hydrochloric acid Illinois toxic substances disclosure to employee act: Hydrochloric acid Illinois chemical safety act: Hydrochloric acid New York release reporting list: Hydrochloric acid Rhode Island RTK hazardous substances: Hydrochloric acid Pennsylvania RTK: Hydrochloric acid Minnesota: Hydrochloric acid Massachusetts RTK: Hydrochloric acid Massachusetts spill list: Hydrochloric acid New Jersey: Hydrochloric acid New Jersey spill list: Hydrochloric acid Louisiana RTK reporting list: Hydrochloric acid Louisiana spill reporting: Hydrochloric acid California Director's List of Hazardous Substances: Hydrochloric acid TSCA 8(b) inventory: Hydrochloric acid TSCA 4(a) proposed test rules: Hydrochloric acid SARA 302/304/311/312 extremely hazardous substances: Hydrochloric acid SARA 313 toxic chemical notification and release reporting: Hydrochloric acid CERCLA: Hazardous substances.: Hydrochloric acid: 5000 lbs. (2268 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 D-2A: Material causing other toxic effects (VERY TOXIC). CLASS E: Corrosive liquid.

**DSCL (EEC):**
R34- Causes burns. R37- Irritating to respiratory system. S26- In case of contact with eyes, rinse immediately with plenty of water and seek medical advice. S45- In case of accident or if you feel unwell, seek medical advice immediately (show the label where possible).

**HMIS (U.S.A.):**

**Health Hazard:** 3

**Fire Hazard:** 0

**Reactivity:** 1

**Personal Protection:**

**National Fire Protection Association (U.S.A.):**

**Health:** 3

**Flammability:** 0

**Reactivity:** 1

**Specific hazard:**

**Protective Equipment:**
Gloves. Full suit. Vapor respirator. Be sure to use an approved/certified respirator or equivalent. Wear appropriate respirator when ventilation is inadequate. Face shield.

## Section 16: Other Information

**References:**
-Hawley, G.G.. The Condensed Chemical Dictionary, 11e ed., New York N.Y., Van Nostrand Reinold, 1987. -SAX, N.I. Dangerous Properties of Indutrial Materials. Toronto, Van Nostrand Reinold, 6e ed. 1984. -The Sigma-Aldrich Library of Chemical Safety Data, Edition II. -Guide de la loi et du règlement sur le transport des marchandises dangeureuses au canada. Centre de conformité internatinal Ltée. 1986.

**Other Special Considerations:** Not available.

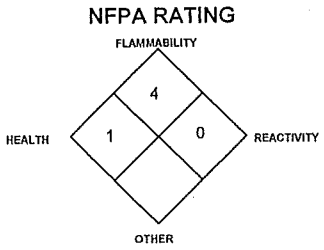
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# MATERIAL SAFETY DATA SHEET

*Prepared to U.S. OSHA, CMA, ANSI and Canadian WHMIS Standards*




## PART I *What is the material and what do I need to know in an emergency?*

### 1. PRODUCT IDENTIFICATION

CHEMICAL NAME; CLASS: **ISOBUTYLENE - $C_4H_8$**
Document Number: Isobutylene

PRODUCT USE: For general analytical/synthetic chemical uses.

SUPPLIER/MANUFACTURER'S NAME: MESA Specialty Gases & Equipment
ADDRESS: 3619 Pendleton Avenue, Suite C
Santa Ana, CA 92704

BUSINESS PHONE: 1-714-434-7102
EMERGENCY PHONE: INFOTRAC: 1-800-535-5053

DATE OF PREPARATION: May 10, 1999

### 2. COMPOSITION and INFORMATION ON INGREDIENTS

| CHEMICAL NAME | CAS # | mole % | EXPOSURE LIMITS IN AIR | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | ACGIH | | OSHA | | | |
| | | | TLV ppm | STEL ppm | PEL ppm | STEL ppm | IDLH ppm | OTHER |
| Isobutylene | 115-11-7 | > 99.0% | There are no specific exposure limits for Isobutylene. Isobutylene is a simple asphyxiant (SA). Oxygen levels should be maintained above 19.5%. | | | | | |
| Maximum Impurities | | < 1.0% | None of the trace impurities in this mixture contribute significantly to the hazards associated with the product. All hazard information pertinent to this product has been provided in this Material Safety Data Sheet, per the requirements of the OSHA Hazard Communication Standard (29 CFR 1910.1200) and State equivalent standards. | | | | | |

NE = Not Established C = Ceiling Limit See Section 16 for Definitions of Terms Used

NOTE: All WHMIS required information is included. It is located in appropriate sections based on the ANSI Z400.1-1993 format.

## 3. HAZARD IDENTIFICATION

**EMERGENCY OVERVIEW:** Isobutylene is a colorless, liquefied, flammable gas with an unpleasant odor similar to burning coal. The liquefied gas rapidly turns into a gas at standard atmospheric temperatures and pressures. Isobutylene is an asphyxiant and presents a significant health hazard by displacing the oxygen in the atmosphere. Rapid evaporation of liquid from the cylinder may cause frostbite. Both the liquid and gas pose a serious fire hazard when accidentally released. The gas is heavier than air and may travel to a source of ignition and flash back to a leak or open container. Flame or high temperature impinging on a localized area of a cylinder of Isobutylene can cause the cylinder to rupture without activating the cylinder's relief devices. Provide adequate fire protection during emergency response situations.

SYMPTOMS OF OVEREXPOSURE BY ROUTE OF EXPOSURE: The most significant route of overexposure for this gas is by inhalation. The following paragraphs describe symptoms of exposure by route of exposure.

INHALATION: High concentrations of this gas can cause an oxygen-deficient environment. Individuals breathing such an atmosphere may experience symptoms which include headaches, ringing in ears, dizziness, drowsiness, unconsciousness, nausea, vomiting, and depression of all the senses. Under some circumstances of overexposure, death may occur. Isobutylene also has some degree of anesthetic action and can be mildly irritating to the mucous membranes. The effects associated with various levels of oxygen are as follows:

| CONCENTRATION | SYMPTOMS OF EXPOSURE |
|---|---|
| 12-16% Oxygen: | Breathing and pulse rate increased, muscular coordination slightly disturbed. |
| 10-14% Oxygen: | Emotional upset, abnormal fatigue, disturbed respiration. |
| 6-10% Oxygen: | Nausea and vomiting, collapse or loss of consciousness. |
| Below 6%: | Convulsive movements, possible respiratory collapse, and death. |

OTHER POTENTIAL HEALTH EFFECTS: Contact with liquid or rapidly expanding gases (which are released under high pressure) may cause frostbite. Symptoms of frostbite include change in skin color to white or grayish-yellow. The pain after such contact can quickly subside.

HAZARDOUS MATERIAL INFORMATION SYSTEM

| | | |
|---|---|---|
| HEALTH | (BLUE) | 1 |
| FLAMMABILITY | (RED) | 4 |
| REACTIVITY | (YELLOW) | 0 |
| PROTECTIVE EQUIPMENT | | B |

| EYES | RESPIRATORY | HANDS | BODY |
|---|---|---|---|
|  | See Section 8 |  | See Section 8 |

For routine industrial applications

**See Section 16 for Definition of Ratings**

HEALTH EFFECTS OR RISKS FROM EXPOSURE: An Explanation in **Lay Terms**. Overexposure to Isobutylene may cause the following health effects:

**ACUTE:** The most significant hazard associated with this gas is inhalation of oxygen-deficient atmospheres. Symptoms of oxygen deficiency include respiratory difficulty, headache, dizziness, and nausea. At high concentrations, unconsciousness or death may occur. Contact with liquefied gas or rapidly expanding gases may cause frostbite.

**CHRONIC:** There are currently no known adverse health effects associated with chronic exposure to Isobutylene.

**TARGET ORGANS:** Respiratory system.

# PART II *What should I do if a hazardous situation occurs?*

## 4. FIRST-AID MEASURES

**RESCUERS SHOULD NOT ATTEMPT TO RETRIEVE VICTIMS OF EXPOSURE TO ISOBUTYLENE WITHOUT ADEQUATE PERSONAL PROTECTIVE EQUIPMENT. At a minimum, Self-Contained Breathing Apparatus and Fire-Retardant Personal Protective equipment should be worn. Adequate fire protection must be provided during rescue situations.**

## 4. FIRST-AID MEASURES (Continued)

Remove victim(s) to fresh air as quickly as possible. Trained personnel should administer supplemental oxygen and/or cardio-pulmonary resuscitation, if necessary. Only trained personnel should administer supplemental oxygen.

In case of frostbite, place the frostbitten part in warm water. DO NOT USE HOT WATER. If warm water is not available, or is impractical to use, wrap the affected parts gently in blankets. Alternatively, if the fingers or hands are frostbitten, place the affected area in the armpit. Encourage victim to gently exercise the affected part while being warmed. Seek immediate medical attention.

Victim(s) must be taken for medical attention. Rescuers should be taken for medical attention, if necessary. Take copy of label and MSDS to physician or other health professional with victim(s).

## 5. FIRE-FIGHTING MEASURES

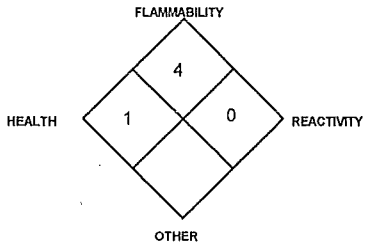
FLASH POINT (Closed Cup): -10°C (< 14°F)
AUTOIGNITION TEMPERATURE: 465°C (869°F)
FLAMMABLE LIMITS (in air by volume, %):

Lower (LEL): 1.8%
Upper (UEL): 9.6%

FIRE EXTINGUISHING MATERIALS: Extinguish Isobutylene fires by shutting off the source of the gas. Use water spray or a foam agent to cool fire-exposed containers, structures, and equipment.

UNUSUAL FIRE AND EXPLOSION HAZARDS: When involved in a fire, this material may ignite and produce toxic gases, including carbon monoxide and carbon dioxide.

**NFPA RATING**




See Section 16 for Definition of Ratings

**DANGER!** Fires impinging (direct flame) on the outside surface of unprotected pressure storage vessels of Isobutylene can be very dangerous. Direct flame exposure on the cylinder wall can cause an explosion either by BLEVE (Boiling Liquid Expanding Vapor Explosion), or by exothermic decomposition. This is a catastrophic failure of the vessel releasing the contents into a massive fireball and explosion. The resulting fire and explosion can result in severe equipment damage and personnel injury or death over a large area around the vessel. For massive fires in large areas, use unmanned hose holder or monitor nozzles; if this is not possible, withdraw from area and allow fire to burn.

Explosion Sensitivity to Mechanical Impact: Not sensitive.
Explosion Sensitivity to Static Discharge: Static discharge may cause Isobutylene to ignite explosively if released.

SPECIAL FIRE-FIGHTING PROCEDURES: Structural firefighters must wear Self-Contained Breathing Apparatus and full protective equipment. The best fire-fighting technique may be simply to let the burning gas escape from the pressurized cylinder, tank car, or pipeline. Stop the leak before extinguishing fire. If the fire is extinguished before the leak is sealed, the leaking gas could explosively re-ignite without warning and cause extensive damage, injury, or fatality. In this case, increase ventilation (in enclosed areas) to prevent flammable or explosive mixture formation. Structural firefighters must wear Self-Contained Breathing Apparatus and full protective equipment. Because of the potential for a BLEVE, evacuation of non-emergency personnel is essential. If water is not available for cooling or protection of vessel exposures, evacuate the area. Refer to the North American Emergency Response Guidebook for additional information. Other information for pre-planning can be found in the American Petroleum Institute Publications 2510 and 2510A.

## 6. ACCIDENTAL RELEASE MEASURES

SPILL AND LEAK RESPONSE: Uncontrolled releases should be responded to by trained personnel using pre-planned procedures. Proper protective equipment should be used. In case of a release, clear the affected area, protect people, and respond with trained personnel. Adequate fire protection must be provided. Minimum Personal Protective Equipment should be **Level B: fire-retardant protective clothing, gloves resistant to tears, and Self-Contained Breathing Apparatus.**

Use only non-sparking tools and equipment. Locate and seal the source of the leaking gas. Protect personnel attempting the shut off with water spray. Allow the gas to dissipate. Monitor the surrounding area for combustible gas levels and oxygen. Combustible gas concentration must be below 10% of the LEL (LEL = 1.8%) prior to entry. The atmosphere must have at least 19.5 percent oxygen before personnel can be allowed in the area without Self-Contained Breathing Apparatus. Attempt to close the main source valve prior to entering the area. If this does not stop the release (or if it is not possible to reach the valve), allow the gas to release in place or remove it to a safe area and allow the gas to be released there.

**THIS IS AN EXTREMELY FLAMMABLE GAS.** Protection of all personnel and the area must be maintained.

# PART III *How can I prevent hazardous situations from occurring?*

## 7. HANDLING and STORAGE

WORK PRACTICES AND HYGIENE PRACTICES: As with all chemicals, avoid getting Isobutylene IN YOU. Do not eat or drink while handling chemicals. Be aware of any signs of dizziness or fatigue; exposures to fatal concentrations of Isobutylene could occur without any significant warning symptoms.

STORAGE AND HANDLING PRACTICES: Cylinders should be stored in dry, well-ventilated areas away from sources of heat. Compressed gases can present significant safety hazards. Store containers away from heavily trafficked areas and emergency exits. Post "No Smoking or Open Flames" signs in storage or use areas.

SPECIAL PRECAUTIONS FOR HANDLING GAS CYLINDERS: Protect cylinders against physical damage. Store in cool, dry, well-ventilated area, away from sources of heat, ignition and direct sunlight. Do not allow area where cylinders are stored to exceed 52°C (125°F). Isolate from oxidizers such as oxygen, chlorine, or fluorine. Use a check valve or trap in the discharge line to prevent hazardous backflow. Post "No Smoking or Open Flame" signs in storage and use areas. Cylinders should be stored upright and be firmly secured to prevent falling or being knocked over. Cylinders can be stored in the open, but in such cases, should be protected against extremes of weather and from the dampness of the ground to prevent rusting. Never tamper with pressure relief devices in valves and cylinders. Electrical equipment should be non-sparking or explosion proof. The following rules are applicable to situations in which cylinders are being used:

**Before Use:** Move cylinders with a suitable hand truck. Do not drag, slide, or roll cylinders. Do not drop cylinders or permit them to strike each other. Secure cylinders firmly. Leave the valve protection cap, if provided, in place until cylinder is ready for use.

**During Use:** Use designated CGA fittings and other support equipment. Do not use adapters. Do not heat cylinder by any means to increase the discharge rate of the product from the cylinder. Use check valve or trap in discharge line to prevent hazardous backflow into the cylinder. Do not use oils or grease on gas-handling fittings or equipment.

**After Use:** Close main cylinder valve. Replace valve protection cap, if provided. Mark empty cylinders "EMPTY".

**NOTE:** Use only DOT or ASME code containers. Earth-ground and bond all lines and equipment associated with Isobutylene. Close valve after each use and when empty. Cylinders must not be recharged except by or with the consent of owner. For additional information refer to the Compressed Gas Association Pamphlet P-1, *Safe Handling of Compressed Gases in Containers*. Additionally, refer to CGA Bulletin SB-2 "*Oxygen Deficient Atmospheres*".

PROTECTIVE PRACTICES DURING MAINTENANCE OF CONTAMINATED EQUIPMENT: Follow practices indicated in Section 6 (Accidental Release Measures). Make certain that application equipment is locked and tagged-out safely. Purge gas handling equipment with inert gas (e.g., nitrogen) before attempting repairs.

## 8. EXPOSURE CONTROLS - PERSONAL PROTECTION

VENTILATION AND ENGINEERING CONTROLS: Use with adequate ventilation. Local exhaust ventilation is preferred, because it prevents Isobutylene dispersion into the work place by eliminating it at its source. If appropriate, install automatic monitoring equipment to detect the presence of potentially explosive air-gas mixtures and level of oxygen.

RESPIRATORY PROTECTION: Maintain oxygen levels above 19.5% in the workplace. Maintain level of gas below the level listed in Section 2 (Composition and Information on Ingredients). Use supplied air respiratory protection if oxygen levels are below 19.5% or during emergency response to a release of Isobutylene. If respiratory protection is required, follow the requirements of the Federal OSHA Respiratory Protection Standard (29 CFR 1910.134) or equivalent State standards.

EYE PROTECTION: Splash goggles or safety glasses, for protection from rapidly expanding gases and splashes of liquid Isobutylene.

HAND PROTECTION: Wear gloves resistant to tears when handling cylinders of Isobutylene. Use low-temperature protective gloves (e.g., Kevlar) when working with containers of liquid Isobutylene.

BODY PROTECTION: Use body protection appropriate for task. Transfer of large quantities under pressure may require protective equipment appropriate to protect employees from splashes of liquefied product, as well as fire retardant items.

## 9. PHYSICAL and CHEMICAL PROPERTIES

VAPOR DENSITY @ 21.1°C (70°F): 2.396 kg/m$^3$ (0.1496 lb/ft$^3$)
SPECIFIC GRAVITY ( air = 1): 1.997
SOLUBILITY IN WATER: Insoluble.
EVAPORATION RATE (nBuAc = 1): Not applicable.
ODOR THRESHOLD: Not established.
COEFFICIENT WATER/OIL DISTRIBUTION: Not applicable.
pH: Not applicable.
FREEZING POINT: -140°C (-220.6°F)
BOILING POINT @ 1 atm: -6.9°C (19.6°F)
EXPANSION RATIO: Not applicable
VAPOR PRESSURE (psia): 39
SPECIFIC VOLUME (ft$^3$/lb): 6.7

APPEARANCE AND COLOR: Colorless gas with the unpleasant odor of burning coal. The liquid is also colorless and has the same unpleasant odor of burning coal.

HOW TO DETECT THIS SUBSTANCE (warning properties): There are no distinct warning properties. In terms of leak detection, fittings and joints can be painted with a soap solution to detect leaks, which will be indicated by a bubble formation.

## 10. STABILITY and REACTIVITY

STABILITY: Stable.

DECOMPOSITION PRODUCTS: When ignited in the presence of oxygen, this gas will burn to produce carbon monoxide and carbon dioxide.

MATERIALS WITH WHICH SUBSTANCE IS INCOMPATIBLE: Strong oxidizers (e.g., chlorine, bromine pentafluoride, oxygen, oxygen difluoride, and nitrogen trifluoride).

HAZARDOUS POLYMERIZATION: Will not occur.

CONDITIONS TO AVOID: Contact with incompatible materials and exposure to heat, sparks, and other sources of ignition. Cylinders exposed to high temperatures or direct flame can rupture or burst.

# PART IV *Is there any other useful information about this material?*

## 11. TOXICOLOGICAL INFORMATION

TOXICITY DATA: The following information is for pure Isobutylene.

**ISOBUTYLENE:**
$LC_{50}$ (rat, inhalation) = 620 g/m$^3$/4 hours
$LC_{50}$ (mouse, inhalation) = 415 g/m$^3$/2 hours

SUSPECTED CANCER AGENT: Isobutylene is not found on the following lists: FEDERAL OSHA Z LIST, NTP, IARC, CAL/OSHA, and therefore is neither considered to be nor suspected to be a cancer-causing agent by these agencies.

IRRITANCY OF PRODUCT: Isobutylene may be mildly irritating to the mucous membranes. In addition, contact with rapidly expanding gases can cause frostbite to exposed tissue.

SENSITIZATION TO THE PRODUCT: Isobutylene is not known to cause sensitization in humans.

REPRODUCTIVE TOXICITY INFORMATION: Listed below is information concerning the effects of Isobutylene on the human reproductive system.

Mutagenicity: No mutagenic effects have been described for Isobutylene.
Embryotoxicity: No embryotoxic effects have been described for Isobutylene.
Teratogenicity: No teratogenic effects have been described for Isobutylene.
Reproductive Toxicity: No reproductive toxicity effects have been described for Isobutylene.

*A mutagen is a chemical which causes permanent changes to genetic material (DNA) such that the changes will propagate through generational lines. An embryotoxin is a chemical which causes damage to a developing embryo (i.e., within the first eight weeks of pregnancy in humans), but the damage does not propagate across generational lines. A teratogen is a chemical which causes damage to a developing fetus, but the damage does not propagate across generational lines. A reproductive toxin is any substance which interferes in any way with the reproductive process.*

MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE: Acute or chronic respiratory conditions may be aggravated by overexposure to Isobutylene.

## 11. TOXICOLOGICAL INFORMATION (Continued)

RECOMMENDATIONS TO PHYSICIANS: Administer oxygen, if necessary. Treat symptoms and eliminate exposure.

BIOLOGICAL EXPOSURE INDICES (BEIs): Currently, Biological Exposure Indices (BEIs) are not applicable for Isobutylene.

## 12. ECOLOGICAL INFORMATION

ENVIRONMENTAL STABILITY: This gas will be dissipated rapidly in well-ventilated areas.

EFFECT OF MATERIAL ON PLANTS or ANIMALS: Any adverse effect on animals would be related to oxygen-deficient environments. No adverse effect is anticipated to occur to plant life, except for frost produced in the presence of rapidly expanding gases. See Section 11, Toxicological Information, for additional information on effects on animals.

EFFECT OF CHEMICAL ON AQUATIC LIFE: No evidence is currently available on the effects of Isobutylene on aquatic life.

## 13. DISPOSAL CONSIDERATIONS

PREPARING WASTES FOR DISPOSAL: Waste disposal must be in accordance with appropriate Federal, State, and local regulations. Return cylinders with any residual product to MESA Specialty Gases & Equipment Do not dispose of locally.

## 14. TRANSPORTATION INFORMATION

THIS MATERIAL IS HAZARDOUS AS DEFINED BY 49 CFR 172.101 BY THE U.S. DEPARTMENT OF TRANSPORTATION.

**For Isobutylene Gas:**

| | |
|---|---|
| PROPER SHIPPING NAME: | Isobutylene |
| HAZARD CLASS NUMBER and DESCRIPTION: | 2.1 (Flammable Gas) |
| UN IDENTIFICATION NUMBER: | UN 1055 |
| PACKING GROUP: | Not Applicable |
| DOT LABEL(S) REQUIRED: | Flammable Gas |

NORTH AMERICAN EMERGENCY RESPONSE GUIDEBOOK NUMBER (1996): 115

**Alternate Description:**

| | |
|---|---|
| PROPER SHIPPING NAME: | Petroleum gases, liquefied |
| HAZARD CLASS NUMBER and DESCRIPTION: | 2.1 (Flammable Gas) |
| UN IDENTIFICATION NUMBER: | UN 1075 |
| PACKING GROUP: | Not Applicable |
| DOT LABEL(S) REQUIRED: | Flammable Gas |

NORTH AMERICAN EMERGENCY RESPONSE GUIDEBOOK NUMBER (1996): 115

MARINE POLLUTANT: Isobutylene is not classified by the DOT as a Marine Pollutant (as defined by 49 CFR 172.101, Appendix B).

TRANSPORT CANADA TRANSPORTATION OF DANGEROUS GOODS REGULATIONS: THIS MATERIAL IS CONSIDERED AS DANGEROUS GOODS. Use the above information for the preparation of Canadian Shipments.

## 15. REGULATORY INFORMATION

U.S. SARA REPORTING REQUIREMENTS: Isobutylene is not subject to the reporting requirements of Sections 302, 304, and 313 of Title III of the Superfund Amendments and Reauthorization Act.

U.S. SARA THRESHOLD PLANNING QUANTITY: Not applicable.

U.S. CERCLA REPORTABLE QUANTITY (RQ): Not applicable.

CANADIAN DSL/NDSL INVENTORY STATUS: Isobutylene is on the DSL Inventory.

U.S. TSCA INVENTORY STATUS: Isobutylene is listed on the TSCA Inventory.

## 15. REGULATORY INFORMATION (Continued)

OTHER U.S. FEDERAL REGULATIONS: Isobutylene is subject to the reporting requirements of Section 112(r) of the Clean Air Act. The Threshold Quantity for this gas is 10,000 lb. Depending on specific operations involving the use of Isobutylene, the regulations of the Process Safety Management of Highly Hazardous Chemicals may be applicable (29 CFR 1910.119). Under this regulation Isobutylene is not listed in Appendix A; however, any process that involves a flammable gas on-site, in one location, in quantities of 10,000 lb (4,553 kg) or greater is covered under this regulation unless it is used as a fuel.

U.S. STATE REGULATORY INFORMATION: Isobutylene is covered under specific State regulations, as denoted below:

**Alaska - Designated Toxic and Hazardous Substances:** Liquefied Petroleum Gas.
**California - Permissible Exposure Limits for Chemical Contaminants:** Liquefied Petroleum Gas.
**Florida - Substance List:** Isobutylene.
**Illinois - Toxic Substance List:** No.
**Kansas - Section 302/313 List:** No.
**Massachusetts - Substance List:** Isobutylene.
**Michigan - Critical Materials Register:** No.
**Minnesota - List of Hazardous Substances:** Liquefied Petroleum Gas.
**Missouri - Employer Information/Toxic Substance List:** No.
**New Jersey - Right to Know Hazardous Substance List:** Isobutylene.
**North Dakota - List of Hazardous Chemicals, Reportable Quantities:** No.
**Pennsylvania - Hazardous Substance List:** Isobutylene.
**Rhode Island - Hazardous Substance List:** Liquefied Petroleum Gas.
**Texas - Hazardous Substance List:** Liquefied Petroleum Gas.
**West Virginia - Hazardous Substance List:** Liquefied Petroleum Gas.
**Wisconsin - Toxic and Hazardous Substances:** Liquefied Petroleum Gas.

CALIFORNIA SAFE DRINKING WATER AND TOXIC ENFORCEMENT ACT (PROPOSITION 65): Isobutylene is not on the California Proposition 65 lists.

LABELING:

**DANGER:**

FLAMMABLE LIQUID AND GAS UNDER PRESSURE.
CAN FORM EXPLOSIVE MIXTURES WITH AIR.
MAY CAUSE FROSTBITE.

Keep away from heat, flames, and sparks.
Store and use with adequate ventilation.
Cylinder temperature should not exceed 52°C (125°F).
Do not get liquid in eyes, on skin, or clothing.
Close valve after each use and when empty.
Use in accordance with the Material Safety Data Sheet.

**FIRST AID:**

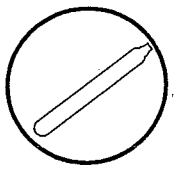
**IF INHALED**, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.

**IN CASE OF FROSTBITE,** obtain immediate medical attention.

DO NOT REMOVE THIS PRODUCT LABEL.

CANADIAN WHMIS SYMBOLS:

**Class A:** Compressed Gas
**Class B1**: Flammable Gas





## 16. OTHER INFORMATION

The information contained herein is based on data considered accurate. However, no warranty is expressed or implied regarding the accuracy of these data or the results to be obtained from the use thereof. MESA Specialty Gases & Equipment assumes no responsibility for injury to the vendee or third persons proximately caused by the material if reasonable safety procedures are not adhered to as stipulated in the data sheet. Additionally, MESA Specialty Gases & Equipment assumes no responsibility for injury to vendee or third persons proximately caused by abnormal use of the material even if reasonable safety procedures are followed. Furthermore, vendee assumes the risk in his use of the material.

## DEFINITIONS OF TERMS

A large number of abbreviations and acronyms appear on a MSDS. Some of these which are commonly used include the following:

**CAS #**: This is the Chemical Abstract Service Number which uniquely identifies each constituent. It is used for computer-related searching.

**EXPOSURE LIMITS IN AIR:**
**ACGIH** - American Conference of Governmental Industrial Hygienists, a professional association which establishes exposure limits. **TLV** - Threshold Limit Value - an airborne concentration of a substance which represents conditions under which it is generally believed that nearly all workers may be repeatedly exposed without adverse effect. The duration must be considered, including the 8-hour Time Weighted Average **(TWA)**, the 15-minute Short Term Exposure Limit, and the instantaneous Ceiling Level **(C)**. Skin absorption effects must also be considered.
**OSHA** - U.S. Occupational Safety and Health Administration. **PEL** - Permissible Exposure Limit - This exposure value means exactly the same as a TLV, except that it is enforceable by OSHA. The OSHA Permissible Exposure Limits are based in the 1989 PELs and the June, 1993 Air Contaminants Rule (Federal Register: 58: 35338-35351 and 58: 40191). Both the current PELs and the vacated PELs are indicated. The phrase, "Vacated 1989 PEL," is placed next to the PEL which was vacated by Court Order.
**IDLH** - Immediately Dangerous to Life and Health - This level represents a concentration from which one can escape within 30-minutes without suffering escape-preventing or permanent injury. **The DFG - MAK** is the Republic of Germany's Maximum Exposure Level, similar to the U.S. PEL. **NIOSH** is the National Institute of Occupational Safety and Health, which is the research arm of the U.S. Occupational Safety and Health Administration **(OSHA)**. NIOSH issues exposure guidelines called Recommended Exposure Levels **(RELs)**. When no exposure guidelines are established, an entry of **NE** is made for reference.

**HAZARD RATINGS:**
**HAZARDOUS MATERIALS IDENTIFICATION SYSTEM:** Health Hazard: **0** (minimal acute or chronic exposure hazard); **1** (slight acute or chronic exposure hazard); **2** (moderate acute or significant chronic exposure hazard); **3** (severe acute exposure hazard; onetime overexposure can result in permanent injury and may be fatal); **4** (extreme acute exposure hazard; onetime overexposure can be fatal). Flammability Hazard: **0** (minimal hazard); **1** (materials that require substantial pre-heating before burning); **2** (combustible liquid or solids; liquids with a flash point of 38-93°C [100-200°F]); **3** (Class IB and IC flammable liquids with flash points below 38°C [100°F]); **4** (Class IA flammable liquids with flash points below 23°C [73°F] and boiling points below 38°C [100°F]. Reactivity Hazard: **0** (normally stable); 1 (material that can become unstable at elevated temperatures or which can react slightly with water); **2** (materials that are unstable but do not detonate or which can react violently with water); **3** (materials that can detonate when initiated or which can react explosively with water); **4** (materials that can detonate at normal temperatures or pressures).
**NATIONAL FIRE PROTECTION ASSOCIATION:** Health Hazard: **0** (material that on exposure under fire conditions would offer no hazard beyond that of ordinary combustible materials); **1** (materials that on exposure under fire conditions could cause irritation or minor residual injury); **2** (materials that on intense or continued exposure under fire conditions could cause temporary incapacitation or possible residual injury); **3** (materials that can on short exposure could cause serious temporary or residual injury); **4** (materials that under very short exposure causes death or major residual injury).

**NATIONAL FIRE PROTECTION ASSOCIATION (Continued):** Flammability Hazard and Reactivity Hazard: Refer to definitions for "Hazardous Materials Identification System".

**FLAMMABILITY LIMITS IN AIR:**
Much of the information related to fire and explosion is derived from the National Fire Protection Association **(NFPA)**. Flash Point - Minimum temperature at which a liquid gives off sufficient vapors to form an ignitable mixture with air. Autoignition Temperature: The minimum temperature required to initiate combustion in air with no other source of ignition. LEL - the lowest percent of vapor in air, by volume, that will explode or ignite in the presence of an ignition source. UEL - the highest percent of vapor in air, by volume, that will explode or ignite in the presence of an ignition source.

**TOXICOLOGICAL INFORMATION:**
Possible health hazards as derived from human data, animal studies, or from the results of studies with similar compounds are presented. Definitions of some terms used in this section are: **$LD_{50}$** - Lethal Dose (solids & liquids) which kills 50% of the exposed animals; **$LC_{50}$** - Lethal Concentration (gases) which kills 50% of the exposed animals; **ppm** concentration expressed in parts of material per million parts of air or water; **$mg/m^3$** concentration expressed in weight of substance per volume of air; **mg/kg** quantity of material, by weight, administered to a test subject, based on their body weight in kg. Data from several sources are used to evaluate the cancer-causing potential of the material. The sources are: **IARC** - the International Agency for Research on Cancer; **NTP** - the National Toxicology Program, **RTECS** - the Registry of Toxic Effects of Chemical Substances, **OSHA** and **CAL/OSHA**. IARC and NTP rate chemicals on a scale of decreasing potential to cause human cancer with rankings from 1 to 4. Subrankings (2A, 2B, etc.) are also used. Other measures of toxicity include **TDLo**, the lowest dose to cause a symptom and **TCLo** the lowest concentration to cause a symptom; **TDo, LDLo**, and **LDo**, or **TC, TCo, LCLo**, and **LCo**, the lowest dose (or concentration) to cause lethal or toxic effects. **BEI** - Biological Exposure Indices, represent the levels of determinants which are most likely to be observed in specimens collected from a healthy worker who has been exposed to chemicals to the same extent as a worker with inhalation exposure to the TLV. Ecological Information: EC is the effect concentration in water.

**REGULATORY INFORMATION:**
This section explains the impact of various laws and regulations on the material. **EPA** is the U.S. Environmental Protection Agency. **WHMIS** is the Canadian Workplace Hazardous Materials Information System. **DOT** and **TC** are the U.S. Department of Transportation and the Transport Canada, respectively. Superfund Amendments and Reauthorization Act **(SARA)**; the Canadian Domestic/Non-Domestic Substances List **(DSL/NDSL)**; the U.S. Toxic Substance Control Act **(TSCA)**; Marine Pollutant status according to the **DOT**; the Comprehensive Environmental Response, Compensation, and Liability Act **(CERCLA or Superfund)**; and various state regulations.

Manufactured for

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***CORPORATION***

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# MATERIAL SAFETY DATA SHEET

*Prepared to U.S. OSHA, CMA, ANSI and Canadian WHMIS Standards*

## 1. PRODUCT IDENTIFICATION

## CHEMICAL NAME; CLASS: NON-FLAMMABLE GAS MIXTURE

**Containing One or More of the Following Components in a Nitrogen Balance Gas: Oxygen, 0.0015-23.5%; Propane, 0-1.1%; n-Pentane, 0-0.75%; n-Hexane; 0-0.48%; Carbon Monoxide, 0.0005-1.0%; Hydrogen Sulfide, 0.001-0.025%**

**NOTE: MIXTURES COMPRISED OF AN AIR BALANCE GAS CONTAIN BETWEEN 19.5-23.5% OXYGEN.**

**SYNONYMS:** Not Applicable

**CHEMICAL FAMILY NAME:** Not Applicable

**FORMULA:** Not Applicable

**Document Number:** 50016 (Replaces ISC MSDS No.1810-2187, 1810-2343, 1810-3366, 1810-3937 1810-7219, 1810-7599, 1810-6179)

**Note:** The Material Safety Data Sheet is for this gas mixture supplied in cylinders with 33 cubic feet (935 liters) or less gas capacity (DOT - 39 cylinders). This MSDS has been developed for various gas mixtures with the composition of components within the ranges listed in Section 2 (Composition and Information on Ingredients). Refer to the product label for information on the actual composition of the product.

| | |
|---|---|
| **PRODUCT USE**: | Calibration of Monitoring and Research Equipment |
| **SUPPLIER/MANUFACTURER'S NAME**: | CALGAZ |
| **ADDRESS**: | 821 Chesapeake Drive<br>Cambridge, MD 21613 |
| **EMERGENCY PHONE**: | CHEMTREC: 1-800-424-9300 |
| **BUSINESS PHONE**: | 1-410-228-6400 |
| General MSDS Information | 1-713/868-0440 |
| Fax on Demand: | 1-800/231-1366 |

## 2. COMPOSITION and INFORMATION ON INGREDIENTS

| CHEMICAL NAME | CAS # | mole % | EXPOSURE LIMITS IN AIR | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | ACGIHTLV | | OSHA | | IDLH | OTHER |
| | | | TWA ppm | STEL ppm | TWA ppm | STEL ppm | ppm | ppm |
| Oxygen | 7782-44-7 | 0.0015 - 23.5% | There are no specific exposure limits for Oxygen. Oxygen levels should be maintained above 19.5%. | | | | | |
| Propane | 74-98-6 | 0 - 1.1% | 2500 | NE | 1000 | NE | 2100 | NIOSH REL: 1000<br>DFG MAK: 1000 ppm |
| n-Pentane | 109-66-0 | 0 - 0.75% | 600 | 750 | 1000<br>600 (Vacated 1989 PEL) | 750 (Vacated 1989 PEL) | 1500 | NIOSH REL:<br>TWA = 120<br>STEL = 610 (ceiling) 15 minutes<br>DFG MAKs:<br>TWA =1000<br>PEAK = 2•MAK, 60 min., momentary value |
| n-Hexane | 110-54-3 | 0 - 0.48% | 50 | NE | 500<br>50 (Vacated 1989 PEL) | NE | 1100 | NIOSH REL: 50<br>DFG MAK: 50 |
| Hydrogen Sulfide | 7783-06-4 | 0.001-0.025 % | 10<br>NIC = 5 | 15<br>NIC = 5 | 10 (Vacated 1989 PEL) | 20 (ceiling), 50 (10 min. peak, once per shift) 15 (Vacated 1989 PEL) | 100 | NIOSH REL:<br>STEL = 10 (ceiling), 10 minutes<br>DFG MAKs:<br>TWA = 10<br>PEAK = 2•MAK, 10 min., momentary value |
| Carbon Monoxide | 630-08-0 | 0.0005 - 1.0% | 25 | NE | 50<br>35 (Vacated 1989 PEL) | 200 (ceiling) (Vacated 1989 PEL) | 1200 | NIOSH RELs:<br>TWA = 35<br>STEL = 200 ceiling<br>DFG MAKs:<br>TWA = 30<br>PEAK = 2•MAK, 15 min., average value, 1 hr interval<br>DFG MAK Pregnancy Risk Classification: B |
| Nitrogen | 7727-37-9 | Balance | There are no specific exposure limits for Nitrogen. Nitrogen is a simple asphyxiant (SA). Oxygen levels should be maintained above 19.5%. | | | | | |

NE = Not Established. NIC = Notice of Intended Change See Section 16 for Definitions of Terms Used.

NOTE: ALL WHMIS required information is included in appropriate sections based on the ANSI Z400.1-1998 format. This gas mixture has been classified in accordance with the hazard criteria of the CPR and the MSDS contains all the information required by the CPR.

# 3. HAZARD IDENTIFICATION

**EMERGENCY OVERVIEW**: This gas mixture is a colorless gas which has a rotten-egg odor (due to the presence of Hydrogen Sulfide). The odor cannot be relied on as an adequate warning of the presence of this gas mixture, because olfactory fatigue occurs after over-exposure to Hydrogen Sulfide. Hydrogen Sulfide and Carbon Monoxide (another component of this gas mixture) are toxic to humans in relatively low concentrations. Over-exposure to this gas mixture can cause skin or eye irritation, nausea, dizziness, headaches, collapse, unconsciousness, coma, and death. The Propane, n-Pentane, and n-Hexane components can cause anesthetic or peripheral neuropathy effects. Additionally, releases of this gas mixture may produce oxygen-deficient atmospheres (especially in small confined spaces or other poorly-ventilated environments); individuals in such atmospheres may be asphyxiated.

**SYMPTOMS OF OVER-EXPOSURE BY ROUTE OF EXPOSURE**: The most significant route of over-exposure for this gas mixture is by inhalation.

**INHALATION:** Due to the small size of an individual cylinder of this gas mixture, no unusual health effects from over-exposure to the product are anticipated under routine circumstances of use. A significant health hazard associated with this gas mixture is the potential of inhalation of Hydrogen Sulfide, a component of this gas mixture. Such over-exposures may occur if this gas mixture is used in a confined space or other poorly-ventilated area. Over-exposures to Hydrogen Sulfide can cause dizziness, headache, and nausea. Exposure to this component can result in respiratory arrest, coma, or unconsciousness. Continuous inhalation of low concentrations of Hydrogen Sulfide may cause olfactory fatigue, so that the odor is no longer an effective warning of the presence of this gas. A summary of exposure concentrations and observed effects are as follows

| CONCENTRATION OF HYDROGEN SULFIDE | OBSERVED EFFECT |
|---|---|
| 0.3-30 ppm | Odor is obvious and unpleasant. |
| 50 ppm | Eye irritation. Dryness and irritation of nose, throat. |
| Slightly higher than 50 ppm | Irritation of the respiratory system. |
| 100-150 ppm | Temporary loss of smell. |
| 200-250 ppm | Headache, vomiting nausea. Prolonged exposure may lead to lung damage. Exposures of 4-8 hours can be fatal. |
| 300-500 | Swifter onset of symptoms. Death occurs in 1-4 hours. |
| 500 ppm | Headache, excitement, staggering, and stomach ache after brief exposure. Death occurs within 0.5 - 1 hour of exposure. |
| > 600 ppm | Rapid onset of unconsciousness, coma, death. |
| > 1000 ppm | Immediate respiratory arrest. |
| NOTE: | This gas mixture contains a maximum of 250 ppm Hydrogen Sulfide. The higher concentration values here are presented to delineate the complete health effects which have been observed for humans after exposure to Hydrogen Sulfide. |

HAZARDOUS MATERIAL IDENTIFICATION SYSTEM

| | | |
|---|---|---|
| HEALTH HAZARD | (BLUE) | 3 |
| FLAMMABILITY HAZARD | (RED) | 0 |
| PHYSICAL HAZARD | (YELLOW) | 0 |

PROTECTIVE EQUIPMENT

| EYES | RESPIRATORY | HANDS | BODY |
|---|---|---|---|
| See Section 8 | | | |

For Routine Industrial Use and Handling Applications

Inhalation over-exposures to atmospheres containing more than the Threshold Limit Value of Carbon Monoxide (25 ppm), another component of this gas mixture, can result in serious health consequences. Carbon Monoxide is classified as a chemical asphyxiant, producing a toxic action by combining with the hemoglobin of the blood and replacing the available oxygen. Through this replacement, the body is deprived of the required oxygen, and asphyxiation occurs.

Since the affinity of Carbon Monoxide for hemoglobin is about 200-300 times that of oxygen, only a small amount of Carbon Monoxide will cause a toxic reaction to occur. Carbon Monoxide exposures in excess of 50 ppm will produce symptoms of poisoning if breathed for a sufficiently long time. If this gas mixture is released in a small, poorly ventilated area (i.e. an enclosed or confined space), symptoms which may develop include the following:

| CONCENTRATION OF CARBON MONOXIDE | OBSERVED EFFECT |
|---|---|
| All exposure levels: .. | Over-exposure to Carbon Monoxide can be indicated by the lips and fingernails turning bright red. |
| 200 ppm: ..... | Slight symptoms (i.e. headache) after several hours of exposure. |
| 400 ppm: | Headache and discomfort experienced within 2-3 hours of exposure. |
| 1,000 -2000 ppm: | Within 30 minutes, slight palpitations of the heart occurs. Within 1.5 hours, there is a tendency to stagger. |
| 200-2500 ppm: | Within 2 hours, there is mental confusion, headaches, and nausea. Unconsciousness within 30 minutes. |
| > 2500 ppm: | Potential for collapse and death before warning symptoms. |

Another hazard associated with this gas mixture is the potential for anesthetic and peripheral neuropathy effects after inhalation over-exposures to the Propane, n-Pentane and n-Hexane components of this gas mixture. Specific human over-exposure data are available for n-Pentane and n-Hexane, as follows:

| CONCENTRATION OF n-PENTANE | OBSERVED EFFECT |
|---|---|
| Brief (10 minute) up to 5,000 ppm: | No symptoms. |
| Higher than 5,000 ppm: | Exhilaration, dizziness and headache can occur. |
| Long term: | Can cause chronic neurological disorder causing damage to the nerves in the hands and feet (peripheral neuropathy) |

| CONCENTRATION OF n-HEXANE | OBSERVED EFFECT |
|---|---|
| Brief (10 minute) at 1,500 ppm: | Irritation of the respiratory tract, nausea and headache. |
| 5000 ppm: | Dizziness and drowsiness can occur. |
| Long term at 500 ppm: | Can affect the nerves in the arms and legs. Effects include numbing or tingling sensations in the fingers and toes, tiredness, muscle weakness, cramps and spasms in the leg, difficulty in holding objects or walking, abdominal pains, loss of appetite, weight loss. More serious exposures can cause damage to the nerves in the hands and feet (peripheral neuropathy). |
| Eyes and Vision: | Abnormal color perception and pigment changes in the eyes have been reported among industrial workers exposed to 423-1280 ppm for 5 years or more. |
| Blood Cells: | Mild forms of anemia have also been associated with exposure to hexane. These are of temporary nature. |

Additionally, if mixtures of this gas mixture contain less than 19.5% Oxygen and are released in a small, poorly ventilated area (i.e. an enclosed or confined space), an oxygen-deficient environment may occur. Individuals breathing such an atmosphere may experience symptoms which include headaches, ringing in ears, dizziness, drowsiness, unconsciousness, nausea, vomiting, and depression of all the senses. Under some circumstances of over-exposure, death may occur. The following effects associated with various levels of oxygen are as follows:

| CONCENTRATION OF OXYGEN | OBSERVED EFFECT |
|---|---|
| 12-16% Oxygen: | Breathing and pulse rate increased, muscular coordination slightly disturbed. |
| 10-14% Oxygen: | Emotional upset, abnormal fatigue, disturbed respiration. |
| 6-10% Oxygen: | Nausea, vomiting, collapse, or loss of consciousness. |
| Below 6%: | Convulsive movements, possible respiratory collapse, and death. |

**SKIN and EYE CONTACT:** The Hydrogen Sulfide component of this gas mixture may be irritating to the skin. Inflammation and irritation of the eyes can occur at very low airborne concentration of Hydrogen Sulfide (less than 10 ppm). Exposure over several hours may result in “gas eyes” or “sore eyes” with symptoms of scratchiness, irritation, tearing and burning. Above 50 ppm of Hydrogen Sulfide, there is an intense tearing, blurring of vision, and pain when looking at light. Over-exposed individuals may see rings around bright lights. Most symptoms disappear when exposure ceases. However, in serious cases, the eye can be permanently damaged.

**HEALTH EFFECTS OR RISKS FROM EXPOSURE: An Explanation in Lay Terms**. Over-exposure to this gas mixture may cause the following health effects:

**ACUTE**: Due to the small size of the individual cylinder of this gas mixture, no unusual health effects from exposure to the product are anticipated under routine circumstances of use. However, the Hydrogen Sulfide and Carbon Monoxide components of this gas mixture are toxic to humans. Over-exposure to this gas mixture can cause nausea, dizziness, headaches, collapse, unconsciousness, coma, and death. Due to the presence of Hydrogen Sulfide, over-exposures to this gas mixture can also irritate the skin and eyes; severe eye contamination can result in blindness.

Inhalation over-exposures to Propane, n-Pentane, and n-Hexane can cause anesthetic effects and motor neuropathy (i.e. pain and tingling in feet and hands).

## 3. HAZARD IDENTIFICATION (Continued)

**CHRONIC**: Abnormal color perception and pigment changes in the eyes have been reported among persons exposed to 420 -1300 ppm of n-Hexane for five years. Additionally, long-term exposure to low levels of n-Hexane or n-Pentane can affect the nerves in the arms and legs. Effects include numbing or tingling sensation, tiredness, cramps, spasms in legs, difficulty holding objects or walking, loss of appetite and weight loss. Pentane isomers, such as n-Pentane, and Propane can cause sensitization of the heart to epinephrine. Refer to Section 11 (Toxicology Information) for additional information on the components of this gas mixture.

**TARGET ORGANS**: ACUTE: Respiratory system, blood system, central nervous system, cardiovascular system. CHRONIC: Reproductive system, cardiovascular system.

## 4. FIRST-AID MEASURES

**RESCUERS SHOULD NOT ATTEMPT TO RETRIEVE VICTIMS OF EXPOSURE TO THIS GAS MIXTURE WITHOUT ADEQUATE PERSONAL PROTECTIVE EQUIPMENT. At a minimum, Self-Contained Breathing Apparatus must be worn.**

No unusual health effects are anticipated after exposure to this gas mixture, due to the small cylinder size. If any adverse symptom develops after over-exposure to this gas mixture, remove victim(s) to fresh air as quickly as possible. Only trained personnel should administer supplemental oxygen and/or cardio-pulmonary resuscitation if necessary.

Victim(s) who experience any adverse effect after over-exposure to this gas mixture must be taken for medical attention. Rescuers should be taken for medical attention if necessary. Take a copy of the label and the MSDS to physician or other health professional with victim(s).

**SKIN EXPOSURE:** If irritation of the skin develops after exposure to this gas mixture, immediately begin decontamination with running water. Minimum flushing is for 15 minutes. Remove exposed or contaminated clothing, taking care not to contaminate eyes. Victim must seek immediate medical attention.

**EYE EXPOSURE**: If irritation of the eye develops after exposure to this gas mixture, open victim's eyes while under gentle running water. Use sufficient force to open eyelids. Have victim "roll" eyes. Minimum flushing is for 15 minutes. Seek medical assistance immediately, preferably an ophthalmologist.

**MEDICAL CONDITIONS AGGRAVATED BY EXPOSURE**: Pre-existing respiratory conditions may be aggravated by over-exposure to this gas mixture. Carbon Monoxide, a component of this gas mixture, can aggravate some diseases of the cardiovascular system, such as coronary artery disease and angina pectoris. Because of the presence of Hydrogen Sulfide, n-Hexane or n-Pentane in this gas mixture, central nervous system conditions, eye disorders, or skin problems may be aggravated by over-exposure to this gas mixture.

**RECOMMENDATIONS TO PHYSICIANS**: Treat symptoms and eliminate over-exposure. Hyperbaric oxygen is the most efficient antidote to Carbon Monoxide poisoning, the optimum range being 2-2.5 atm. A special mask, or, preferably, a compression chamber to utilize oxygen at these pressures is required. Avoid administering stimulant drugs. Be observant for initial signs of pulmonary edema in the event of severe inhalation over-exposures.

## 5. FIRE-FIGHTING MEASURES

**FLASH POINT:** Not applicable.

**AUTOIGNITION TEMPERATURE**: Not applicable.

FLAMMABLE LIMITS (in air by volume, %):

Lower (LEL): Not applicable.

Upper (UEL): Not applicable.

**FIRE EXTINGUISHING MATERIALS**: Non-flammable gas mixture. Use extinguishing media appropriate for surrounding fire.

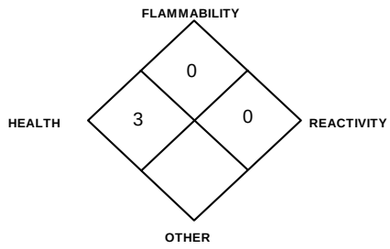
**UNUSUAL FIRE AND EXPLOSION HAZARDS**: This gas mixture contains toxic gases, Hydrogen Sulfide and Carbon Monoxide, and presents an extreme health hazard to firefighters. This gas mixture is not flammable; however, containers, when involved in fire, may rupture or burst in the heat of the fire.

Explosion Sensitivity to Mechanical Impact: Not Sensitive.

Explosion Sensitivity to Static Discharge: Not Sensitive.

**SPECIAL FIRE-FIGHTING PROCEDURES**: Structural firefighters must wear Self-Contained Breathing Apparatus and full protective equipment.

**NFPA RATING**




## 6. ACCIDENTAL RELEASE MEASURES

**LEAK RESPONSE**: Due to the small size and content of the cylinder, an accidental release of this gas mixture presents significantly less risk of over-exposure to Hydrogen Sulfide and Carbon Monoxide, the toxic components of this gas mixture, and other safety hazards related to the remaining components of this gas mixture, than a similar release from a larger cylinder. However, as with any chemical release, extreme caution must be used during emergency response procedures. In the event of a release in which the atmosphere is unknown, and in which other chemicals are potentially involved, evacuate immediate area. Such releases should be responded to by trained personnel using pre-planned procedures. Proper protective equipment should be used. In case of a leak, clear the affected area, protect people, and respond with trained personnel.

For emergency disposal, secure the cylinder and slowly discharge the gas to the atmosphere in a well-ventilated area or outdoors. Allow the gas mixture to dissipate. If necessary, monitor the surrounding area (and the original area of the release) for Hydrogen Sulfide, Carbon Monoxide, and Oxygen. Hydrogen Sulfide and Carbon Monoxide level must be below exposure level listed in Section 2 (Composition and Information on Ingredients) and Oxygen levels must be above 19.5% before non-emergency personnel are allowed to re-enter area.

If leaking incidentally from the cylinder, contact your supplier.

## 7. HANDLING and USE

**WORK PRACTICES AND HYGIENE PRACTICES:** Be aware of any signs of dizziness or fatigue; exposures to fatal concentrations of this gas mixture could occur without any significant warning symptoms, due to olfactory fatigue or oxygen deficiency. Do not attempt to repair, adjust, or in any other way modify cylinders containing a gas mixture with Hydrogen Sulfide or Carbon Monoxide. If there is a malfunction or another type of operational problem, contact nearest distributor immediately. Eye wash stations/safety showers should be near areas where this gas mixture is used or stored. All work operations should be monitored in such a way that emergency personnel can be immediately contacted in the event of a release. All work practices should minimize releases of Hydrogen Sulfide and Carbon Monoxide-containing gas mixtures.

**STORAGE AND HANDLING PRACTICES**: Cylinders should be firmly secured to prevent falling or being knocked-over. Cylinders must be protected from the environment, and preferably kept at room temperature (approximately 21°C, 70°F). Cylinders should be stored in dry, well-ventilated areas, away from sources of heat, ignition, and direct sunlight. Protect cylinders against physical damage.

Full and empty cylinders should be segregated. Use a first-in, first-out inventory system to prevent full containers from being stored for long periods of time. These cylinders are not refillable. **WARNING! Do not refill DOT 39 cylinders. To do so may cause personal injury or property damage.**

**SPECIAL PRECAUTIONS FOR HANDLING GAS CYLINDERS: WARNING!** Compressed gases can present significant safety hazards. During cylinder use, use equipment designed for these specific cylinders. Ensure all lines and equipment are rated for proper service pressure.

**PROTECTIVE PRACTICES DURING MAINTENANCE OF CONTAMINATED EQUIPMENT**: Follow practices indicated in Section 6 (Accidental Release Measures). Make certain that application equipment is locked and tagged-out safely. Always use product in areas where adequate ventilation is provided.

## 8. EXPOSURE CONTROLS - PERSONAL PROTECTION

**VENTILATION AND ENGINEERING CONTROLS**: No special ventilation systems or engineering controls are needed under normal circumstances of use. As with all chemicals, use this gas mixture in well-ventilated areas. If this gas mixture is used in a poorly-ventilated area, install automatic monitoring equipment to detect the levels of Oxygen, Hydrogen Sulfide, and Carbon Monoxide.

**RESPIRATORY PROTECTION:** No special respiratory protection is required under normal circumstances of use. Use supplied air respiratory protection if Carbon Monoxide levels exceed the exposure levels given in Section 2 (Composition and Information on Ingredients) or if oxygen levels are below 19.5%, or if either level is unknown during emergency response to a release of this gas mixture. If respiratory protection is required for emergency response to this gas mixture, follow the requirements of the Federal OSHA Respiratory Protection Standard (29 CFR 1910.134) or equivalent State standards. The following NIOSH respiratory protection recommendations for Hydrogen Sulfide and Carbon Monoxide are provided for further information.

## 8. EXPOSURE CONTROLS - PERSONAL PROTECTION (Continued)

**NIOSH/OSHA RECOMMENDATIONS FOR HYDROGEN SULFIDE CONCENTRATIONS IN AIR:**

Up to 100 ppm: Powered air-purifying respirator with cartridge(s) to protect against hydrogen sulfide; or gas mask with canister to protect against hydrogen sulfide; or SAR; or full-facepiece SCBA.

Emergency or Planned Entry into Unknown Concentration or IDLH Conditions: Positive pressure, full-facepiece SCBA; or positive pressure, full-facepiece SAR with an auxiliary positive pressure SCBA.

Escape: Gas mask with canister to protect against hydrogen sulfide; or escape-type SCBA

NOTE: The IDLH concentration for Hydrogen Sulfide is 100 ppm.

**NIOSH/OSHA RECOMMENDATIONS FOR CARBON MONOXIDE CONCENTRATIONS IN AIR:**

Up to 350 ppm Supplied Air Respirator (SAR)

Up to 875 ppm Supplied Air Respirator (SAR) operated in a continuous flow mode.

Up to 1200 ppm Gas mask with canister to protect against carbon monoxide; or full-facepiece SCBA; or full-facepiece Supplied Air Respirator (SAR).

Emergency or Planned Entry into Unknown Concentration or IDLH Conditions: Positive pressure, full-facepiece SCBA; or positive pressure, full-facepiece Supplied Air Respirator (SAR) with an auxiliary positive pressure SCBA.

Escape: Gas mask with canister to protect against carbon monoxide; or escape-type SCBA.

**NOTE**: End of Service Life Indicator (ESLI) required for gas masks.

**EYE PROTECTION**: Safety glasses. If necessary, refer to U.S. OSHA 29 CFR 1910.133 or appropriate Canadian Standards.

**HAND PROTECTION**: No special protection is needed under normal circumstances of use. If necessary, refer to U.S. OSHA 29 CFR 1910.138 or appropriate Standards of Canada.

**BODY PROTECTION**: No special protection is needed under normal circumstances of use. If a hazard of injury to the feet exists due to falling objects, rolling objects, where objects may pierce the soles of the feet or where employee's feet may be exposed to electrical hazards, use foot protection, as described in U.S. OSHA 29 CFR 1910.136.

## 9. PHYSICAL and CHEMICAL PROPERTIES

**The following information is for Nitrogen, the main component of this gas mixture.**

**GAS DENSITY @ 32°F (0°C) and 1 atm:** 0.072 lbs/ $ft^3$ (1.153 kg/$m^3$)

**FREEZING/MELTING POINT @ 10 psig:** -210°C (-345.8°F)

**BOILING POINT**: -195.8°C (-320.4°F)

**SPECIFIC GRAVITY (air = 1) @ 70°F (21.1°C):** 0.906

**pH**: Not applicable.

**SOLUBILITY IN WATER vol/vol @ 32°F (0°C) and 1 atm:** 0.023

**MOLECULAR WEIGHT:** 28.01

**EVAPORATION RATE (nBuAc = 1):** Not applicable.

**EXPANSION RATIO**: Not applicable.

**ODOR THRESHOLD**: Not applicable.

**SPECIFIC VOLUME ($ft^3$/lb)**: 13.8

**VAPOR PRESSURE @ 70°F (21.1°C) psig**: Not applicable.

**COEFFICIENT WATER/OIL DISTRIBUTION**: Not applicable.

**The following information is for the gas mixture.**

**APPEARANCE AND COLOR**: This gas mixture is a colorless gas which has an rotten egg-like odor, due to the presence of Hydrogen Sulfide.

**HOW TO DETECT THIS SUBSTANCE (warning properties):** Continuous inhalation of low concentrations of Hydrogen Sulfide (a component of this gas mixture) may cause olfactory fatigue, so that there are no distinct warning properties. In terms of leak detection, fittings and joints can be painted with a soap solution to detect leaks, which will be indicated by a bubble formation. Wet lead acetate paper can be used for leak detection. The paper turns black in the presence of Hydrogen Sulfide. Cadmium chloride solutions can also be used. Cadmium solutions will turn yellow upon contact with Hydrogen Sulfide.

## 10. STABILITY and REACTIVITY

**STABILITY**: Normally stable in gaseous state.

**DECOMPOSITION PRODUCTS**: The thermal decomposition products of Propane, n-Hexane, and n-Pentane include carbon oxides. The decomposition products of Hydrogen Sulfide include water and sulfur oxides. The other components of this gas mixture do not decompose, per se, but can react with other compounds in the heat of a fire.

**MATERIALS WITH WHICH SUBSTANCE IS INCOMPATIBLE**: Titanium will burn in Nitrogen (the main component of this gas mixture). Lithium reacts slowly with Nitrogen at ambient temperatures. Components of this gas mixture (Hydrogen Sulfide, Propane, n-Pentane, n-Hexane) are also incompatible with strong oxidizers (i.e. chlorine, bromine pentafluoride, oxygen, oxygen difluoride, and nitrogen trifluoride). Carbon Monoxide is mildly corrosive to nickel and iron (especially at high temperatures and pressures). Hydrogen Sulfide is corrosive to most metals, because it reacts with these substances to form metal sulfides.

**HAZARDOUS POLYMERIZATION**: Will not occur.

**CONDITIONS TO AVOID**: Contact with incompatible materials. Cylinders exposed to high temperatures or direct flame can rupture or burst.

## 11. TOXICOLOGICAL INFORMATION

**TOXICITY DATA**: The following toxicology data are available for the components of this gas mixture:

**NITROGEN:** There are no specific toxicology data for Nitrogen. Nitrogen is a simple asphyxiant, which acts to displace oxygen in the

**n-PENTANE:**

$LD_{50}$ (intravenous, mouse) = 446 mg/kg.

$LC_{50}$ (inhalation, rat) = 364 g/$m^3$/4 hours

LCLo (inhalation, mouse) = 325 g/$m^3$/2 hours

**n-HEXANE:**

Eye, rabbit = 10 mg/ mild

TCLo (inhalation, rat) = 10,000 ppm/7 hr.

TCLo (inhalation, rat) = 5000 ppm/20 hours; teratogenic effects

LD50 (oral, rat) = 28710 mg/kg

LDLo (intraperitoneal, rat ) = 9100 mg/kg

LCLo (inhalation, mouse) = 120,000 mg/kg

LD50 (rat, oral): 28,710 mg/kg

ACUTE INHALATION (mouse): 30,000 ppm, narcosis within 30 to 60 minutes; 35,000-40,000 ppm, convulsions and death.

DERMAL (rabbit): 2 to 5 ml/kg for 4 hours resulted in restlessness and discoordination,; death occurred at 5 ml/kg.

**HYDROGEN SULFIDE:**

LCLo (inhalation, human) = 600 ppm/30 minutes

LDLO (inhalation, man) = 5.7 mg/kg; central nervous system, pulmonary effects

**n-HEXANE (continued):**

CHRONIC INHALATION (rat): 400-600 ppm, 5 days/week, peripheral neuropathy in 45 days; 850 ppm for 143 days, loss of weight and degeneration of the sciatic nerve. (mouse): 250 ppm, peripheral neuropathy within 7 months; no effects at 100 ppm.

**PROPANE:**

**Long-Term Inhalation:** No toxicity or abnormalities were observed when monkeys were exposed to approximately 750 ppm for 90 days. Similar results were obtained when monkeys were exposed to an aerosol spray containing 65% propane and isobutane.

**CARBON MONOXIDE:**

TCLo (inhalation, mouse) = 65 ppm/24 hours (7-18 preg): rep. effects

TCLo (inhalation, mouse) = 8 pph/1 hour (female 8D post): ter. effects

**HYDROGEN SULFIDE (continued):**

LCLo (inhalation, human) = 800 ppm/5 minutes

$LC_{50}$ (inhalation, rat) = 444 ppm

**CARBON MONOXIDE (continued):**

TCLo (inhalation, human) = 600 mg/$m^3$/10 minutes

LCLo (inhalation, man) = 4000 ppm/30 minutes

TCLo (inhalation, man) = 650 ppm/45 minutes: central nervous system and blood system effects.

LCLo (inhalation, human) = 5000 ppm/5 minutes

LCLo (inhalation, dog) = 4000 ppm/46 minutes

LCLo (inhalation, rabbit) = 4000 ppm

$LC_{50}$ (inhalation, rat) = 1811 ppm/4 hours

$LC_{50}$ (inhalation, guinea pig) = 2450 ppm/4 hours

$LC_{50}$ (inhalation, guinea pig) = 5718 ppm/4 hours

LCLo (inhalation, mammal) = 5000 ppm/5 minutes

$LD_{50}$ (inhalation, wild bird) = 1334 ppm

**HYDROGEN SULFIDE (continued):**

$LC_{50}$ (inhalation, mouse) = 673 ppm/1 hour

LCLo (inhalation, mammal) = 800 ppm/5 minutes

**SUSPECTED CANCER AGENT:** The components of this gas mixture are not found on the following lists: FEDERAL OSHA Z LIST, NTP, CAL/OSHA, and IARC; therefore, they are not considered to be, nor suspected to be, cancer-causing agents by these agencies.

**IRRITANCY OF PRODUCT:** The Hydrogen Sulfide component of this gas mixture, is irritating to the eyes, and may be irritating to the skin.

**SENSITIZATION OF PRODUCT**: The components of this gas mixture are not known to be skin or respiratory sensitizers. Pentane isomers (i.e. n-Pentane) and Propane can cause cardiac sensitization to epinephrine.

**REPRODUCTIVE TOXICITY INFORMATION**: Listed below is information concerning the effects of this gas mixture on the human reproductive system.

Mutagenicity: No mutagenicity effects have been described for the components of this gas mixture.

Embryotoxicity: This gas mixture contains components that may cause embryotoxic effects in humans; however, due to the small total amount of the components, embryotoxic effects are not expected to occur.

Teratogenicity: This gas mixture is not expected to cause teratogenic effects in humans due to the small cylinder size and small total amount of all components. The Carbon Monoxide component of this gas mixture which exists up to 1%, can cause teratogenic effects in humans. Severe

## 11. TOXICOLOGICAL INFORMATION (continued)

exposure to Carbon Monoxide during pregnancy has caused adverse effects and the death of the fetus. In general, maternal symptoms are an indicator of the potential risk to the fetus since Carbon Monoxide is toxic to the mother before it is toxic to the fetus.

Reproductive Toxicity: The components of this gas mixture are not expected to cause adverse reproductive effects in humans.

*A mutagen is a chemical which causes permanent changes to genetic material (DNA) such that the changes will propagate through generation lines. An embryotoxin is a chemical which causes damage to a developing embryo (i.e. within the first eight weeks of pregnancy in humans), but the damage does not propagate across generational lines. A teratogen is a chemical which causes damage to a developing fetus, but the damage does not propagate across generational lines. A reproductive toxin is any substance which interferes in any way with the reproductive process.*

**BIOLOGICAL EXPOSURE INDICES (BEIs)**: Biological Exposure Indices (BEIs) have been determined for the components of this gas mixture, as follows:

| CHEMICAL DETERMINANT | SAMPLING TIME | BEI |
|---|---|---|
| CARBON MONOXIDE<br>• Carboxyhemoglobin in blood<br>• Carbon monoxide in end-exhaled air | <br>• End of shift<br>• End of shift | <br>• 3.5% of hemoglobin<br>• 20 ppm |
| n-HEXANE<br>• 2,5-Hexanedione in urine<br>• n-Hexane in end-exhaled air | <br>• End of shift | <br>• 5 mg/g creatinine |

## 12. ECOLOGICAL INFORMATION

**ENVIRONMENTAL STABILITY**: The gas will be dissipated rapidly in well-ventilated areas. The following environmental data are applicable to the components of this gas mixture.

**OXYGEN:** Water Solubility = 1 volume Oxygen/32 volumes water at 20°C. Log $K_{ow}$ = -0.65

**PROPANE:** Log $K_{ow}$ = 2.38. Water Solubility = 62.4 ppm, 25°C. Propane is readily degraded by soil bacteria.

**PENTANE:** Log $K_{ow}$ = 3.39. Water Solubility = 38.5 mg/L. LOG BCF (n-pentane) = calculated, 1.90 and 2.35, respectively. Photolysis, hydrolysis, and bioconcentration are not anticipated to be important fate processes. Biodegradation and soil adsorption are anticipated to be more important processes for this compound.

**n-HEXANE:** Log $K_{ow}$ = 3.90-4.11. Water Solubility = 9.5 mg/L. Estimated Bioconcentration Factor =2.24 and 2.89. Bioconcentration in aquatic organisms is low. Hexane is volatile. Rapid volatilization from water and soil is anticipated for this compound. Hexane will float in slick on surface of the water

**HYDROGEN SULFIDE:** Water Solubility = 1 g/242 mL at 20°C.

**CARBON MONOXIDE**: Water solubility = 3.3 ml/100 cc at 0 °C, 2.3 ml at 20°C.

**NITROGEN:** Water Solubility = 2.4 volumes Nitrogen/100 volumes water at 0°C; 1.6 volumes Nitrogen/100 volumes water at 20°C.

**EFFECT OF MATERIAL ON PLANTS or ANIMALS**: No evidence is currently available on this gas mixture's effects on plant and animal life. The Hydrogen Sulfide and Carbon Monoxide components of this gas mixture, can be deadly to exposed animal life, producing symptoms similar to those experienced by humans. This gas mixture may also be harmful to plant life.

**EFFECT OF CHEMICAL ON AQUATIC LIFE**: No evidence is currently available on the effects of this gas effects on aquatic life. The presence of more than a trace of Carbon Monoxide is a hazard to fish. The following aquatic toxicity data are available for the Hydrogen Sulfide component of this gas mixture.

TLm (Asellussp) = 0.111 mg/L/96 hour
TLm (Cranfgonyx sp) =1.07 mg/L/96 hour
TLm (Gammarrus) = 0.84 mg/L/96 hour
$LC_{50}$ (fly inhalation) = 380 mg/m$^3$/960 minutes
$LC_{50}$ (fly inhalation) = 1500 mg/m$^3$/7 minutes
TLm (Lepomis macrochirus, bluegill sunfish) = 0.0478 mg/L/96 hour
TLm (Lepomis macrochirus, bluegill sunfish) = 0.0448 mg/L/96 hour at 21-22 °C
TLm (Pimephlaes promelas, fathead minnow) = 0.0071-0.55 mg/L/96 hour
TLm (Salvenilis foninalis, brook trout) = 0.0216-0.038 mg/L/96 hour at 8-12.5 °C

## 13. DISPOSAL CONSIDERATIONS

**PREPARING WASTES FOR DISPOSAL PREPARING WASTES FOR DISPOSAL**: Waste disposal must be in accordance with appropriate Federal, State, and local regulations. Cylinders with undesired residual product may be safely vented outdoors with the proper regulator. For further information, refer to Section 16 (Other Information).

## 14. TRANSPORTATION INFORMATION

**THIS GAS MIXTURE IS HAZARDOUS AS DEFINED BY 49 CFR 172.101 BY THE U.S. DEPARTMENT OF TRANSPORTATION.**

**PROPER SHIPPING NAME**: Compressed gases, n.o.s. (*Oxygen, Nitrogen)*or the gas component with the next highest concentration next to Nitrogen.

**HAZARD CLASS NUMBER and DESCRIPTION**: 2.2 (Non-Flammable Gas)

**UN IDENTIFICATION NUMBER**: UN 1956

**PACKING GROUP**: Not Applicable

**DOT LABEL(S) REQUIRED**: Class 2.2 (Non-Flammable Gas)

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**MARINE POLLUTANT**: The components of this gas mixture are not classified by the DOT as Marine Pollutants (as defined by 49 CFR 172.101, Appendix B).

**SPECIAL SHIPPING INFORMATION:** Cylinders should be transported in a secure position, in a well-ventilated vehicle. The transportation of compressed gas cylinders in automobiles or in closed-body vehicles can present serious safety hazards. If transporting these cylinders in vehicles, ensure these cylinders are not exposed to extremely high temperatures (as may occur in an enclosed vehicle on a hot day). Additionally, the vehicle should be well-ventilated during transportation.

**Note:** DOT 39 Cylinders ship in a strong outer carton (overpack). Pertinent shipping information goes on the outside of the overpack. DOT 39 Cylinders do not have transportation information on the cylinder itself.

**TRANSPORT CANADA TRANSPORTATION OF DANGEROUS GOODS REGULATIONS**: This gas is considered as Dangerous Goods, per regulations of Transport Canada.

**PROPER SHIPPING NAME:** Compressed gases, n.o.s. (*Oxygen, Nitrogen)*or the gas component with the next highest concentration next to Nitrogen.

**HAZARD CLASS NUMBER and DESCRIPTION:** 2.2 (Non-Flammable Gas)

**UN IDENTIFICATION NUMBER:** UN 1956

**PACKING GROUP:** Not Applicable

**HAZARD LABEL:** Class 2.2 (Non-Flammable Gas)

**SPECIAL PROVISIONS:** None

**EXPLOSIVE LIMIT AND LIMITED QUANTITY INDEX:** 0.12

**ERAP INDEX:** None

**PASSENGER CARRYING SHIP INDEX:** None

**PASSENGER CARRYING ROAD VEHICLE OR PASSENGER CARRYING RAILWAY VEHICLE INDEX:** 75

**NORTH AMERICAN EMERGENCY RESPONSE GUIDEBOOK NUMBER (2000):** 126

**NOTE:** Shipment of compressed gas cylinders via Public Passenger Road Vehicle is a violation of Canadian law (Transport Canada Transportation of Dangerous Goods Act, 1992).

## 15. REGULATORY INFORMATION

**ADDITIONAL U.S. REGULATIONS:**

**U.S. SARA REPORTING REQUIREMENTS**: This gas is not subject to the reporting requirements of Sections 302, 304 and 313 of Title III of the Superfund Amendments and Reauthorization Act., as follows:

| COMPONENT | SARA 302<br>(40 CFR 355, Appendix A) | SARA 304<br>(40 CFR Table 302.4) | SARA 313<br>(40 CFR 372.65) |
|---|---|---|---|
| n-Hexane | NO | YES | YES |
| Hydrogen Sulfide | YES | YES | YES |

## 15. REGULATORY INFORMATION (Continued)

**U.S. SARA THRESHOLD PLANNING QUANTITY:** Section 302 EHS TPQ = Hydrogen Sulfide = 500 lbs (227 kg);
**U.S. TSCA INVENTORY STATUS**: The components of this gas mixture are listed on the TSCA Inventory.
**U.S. CERCLA REPORTABLE QUANTITY (RQ):** Hexane = 5000 lb (2270 kg); Hydrogen Sulfide = 100 lbs (45.4 lb)
**OTHER U.S. FEDERAL REGULATIONS**:

- Hydrogen Sulfide, Carbon Monoxide, Propane, n-Pentane and n-Hexane are subject to the reporting requirements of CFR 29 1910.1000.
- Hydrogen Sulfide, Propane and n-Pentane are subject to the reporting requirements of Section 112(r) of the Clean Air Act. The Threshold Quantity for each of these gases is 10,000 pounds and so this mixture will not be affected by the regulation.
- Depending on specific operations involving the use of this gas mixture, the regulations of the Process Safety Management of Highly Hazardous Chemicals may be applicable (29 CFR 1910.119). Hydrogen Sulfide is listed in Appendix A of this regulation. The Threshold Quantity for Hydrogen Sulfide under this regulation is 1500 lbs.
- This gas mixture does not contain any Class I or Class II ozone depleting chemicals (40 CFR part 82).
- Nitrogen, Oxygen and n-Hexane are not listed Regulated Substances, per 40 CFR, Part 68, of the Risk Management for Chemical Releases. Hydrogen Sulfide is listed under this regulation in Table 1 as a Regulated Substance (Toxic Substance), in quantities of 10,000 lbs (4,553 kg) or greater. Carbon Monoxide, Propane and n-Pentane are listed under this regulation in Table 3, as Regulated Substances (Flammable), in quantities of 10,000 lbs (4,553 kg) or greater, and so this mixture will not be affected by the regulation.

**U.S. STATE REGULATORY INFORMATION**: The components of this gas mixture are covered under the following specific State regulations:

**Alaska - Designated Toxic and Hazardous Substances: Carbon** Monoxide, Propane, n-Pentane, n-Hexane, Hydrogen Sulfide.
**California - Permissible Exposure Limits for Chemical Contaminants:** Carbon Monoxide, Nitrogen, Propane, n-Pentane, n-Hexane, Hydrogen Sulfide.
**Florida - Substance List:** Oxygen, Carbon Monoxide, n-Pentane, n-Hexane, Hydrogen Sulfide.
**Illinois - Toxic Substance List:** Carbon Monoxide, Propane, n-Pentane, n-Hexane, Hydrogen Sulfide.
**Kansas - Section 302/313 List:** No.
**Massachusetts - Substance List:** Oxygen, Carbon Monoxide, Propane, n-Pentane, n-Hexane, Hydrogen Sulfide.
**Michigan - Critical Materials Register:** No.
**Minnesota - List of Hazardous Substances:** Carbon Monoxide, Propane, n-Pentane, n-Hexane, Hydrogen Sulfide.
**Missouri - Employer Information/Toxic Substance List t:** n-Pentane, n-Hexane, Propane, Hydrogen Sulfide.
**New Jersey - Right to Know Hazardous Substance List:** Oxygen, Carbon Monoxide, Nitrogen, Propane, n-Pentane, n-Hexane.
**North Dakota - List of Hazardous Chemicals, Reportable Quantities:** Hydrogen Sulfide.
**Pennsylvania - Hazardous Substance List:** Oxygen, Carbon Monoxide, Nitrogen, Propane, n-Pentane, n-Hexane, Hydrogen Sulfide.
**Rhode Island - Hazardous Substance List:** Oxygen, Carbon Monoxide, Nitrogen, Propane, n-Pentane, n-Hexane, Hydrogen Sulfide.
**Texas - Hazardous Substance List:** n-Pentane, n-Hexane, Propane, Hydrogen Sulfide.
**West Virginia - Hazardous Substance List:** n-Pentane, n-Hexane, Propane, Hydrogen Sulfide.
**Wisconsin - Toxic and Hazardous Substances:** n-Pentane, n-Hexane, Propane, Hydrogen Sulfide

**CALIFORNIA SAFE DRINKING WATER AND TOXIC ENFORCEMENT ACT (PROPOSITION 65):** The Carbon Monoxide component of this gas mixture is on the California Proposition 65 lists as a chemical known to the State of California to cause birth defects or other reproductive harm.
**ADDITIONAL CANADIAN REGULATIONS:**

**CANADIAN DSL/NDSL INVENTORY STATUS:** The components of this gas mixture are on the Canadian DSL Inventory.
**CANADIAN ENVIRONMENTAL PROTECTION ACT (CEPA) PRIORITIES SUBSTANCES LISTS:** The components of this gas mixture are not on the CEPA Priorities Substances List.
**CANADIAN WHMIS CLASSIFICATION:** This gas mixture is categorized as a Controlled Product, Hazard Classes A and D2A, as per the Controlled Product Regulations.

## 16. OTHER INFORMATION

### INFORMATION ABOUT DOT-39 NRC (Non-Refillable Cylinder) PRODUCTS

DOT 39 cylinders ship as hazardous materials when full. Once the cylinders are relieved of pressure (empty) they are not considered hazardous material or waste. Residual gas in this type of cylinder is not an issue because toxic gas mixtures are prohibited. Calibration gas mixtures typically packaged in these cylinders are Nonflammable n.o.s., UN 1956. A small percentage of calibration gases packaged in DOT 39 cylinders are flammable or oxidizing gas mixtures.

For disposal of used DOT-39 cylinders, it is acceptable to place them in a landfill if local laws permit. Their disposal is no different than that employed with other DOT containers such as spray paint cans, household aerosols, or disposable cylinders of propane (for camping, torch etc.). When feasible, we recommended recycling for scrap metal content. CALGAZ will do this for any customer that wishes to return cylinders to us prepaid. All that is required is a phone call to make arrangements so we may anticipate arrival. Scrapping cylinders involves some preparation before the metal dealer may accept them. We perform this operation as a service to valued customers who want to participate.

**MIXTURES:** When two or more gases or liquefied gases are mixed, their hazardous properties may combine to create additional, unexpected hazards. Obtain and evaluate the safety information for each component before you produce the mixture. Consult an Industrial Hygienist or other trained person when you make your safety evaluation of the end product. Remember, gases and liquids have properties which can cause serious injury or death.

Further information about the handling of compressed gases can be found in the following pamphlets published by: Compressed Gas Association Inc. (CGA), 1725 Jefferson Davis Highway, Suite 1004, Arlington, VA 22202-4102. Telephone: (703) 412-0900.

*P-1* *"Safe Handling of Compressed Gases in Containers"*
*AV-1* *"Safe Handling and Storage of Compressed Gases"*
*"Handbook of Compressed Gases"*

**PREPARED BY:** CHEMICAL SAFETY ASSOCIATES, Inc.
PO Box 3519, La Mesa, CA 91944-3519
619/670-0609

Fax on Demand: 1-800/231-1366

AIR LIQUIDE™

This Material Safety Data Sheet is offered pursuant to OSHA's Hazard Communication Standard, 29 CFR, 1910.1200. Other government regulations must be reviewed for applicability to this gas mixture. To the best of CALGAZ knowledge, the information contained herein is reliable and accurate as of this date; however, accuracy, suitability or completeness are not guaranteed and no warranties of any type, either express or implied, are provided. The information contained herein relates only to this specific product. If this gas mixture is combined with other materials, all component properties must be considered. Data may be changed from time to time. Be sure to consult the latest edition.

# APPENDIX C

Heat Illness Prevention Program

# HEAT ILLNESS
# PREVENTION PROGRAM

| | | |
|---|---|---|
| **CORPORATE HEALTH AND SAFETY DIRECTOR** | : | **Brian Hobbs, CIH, CSP** |
| **EFFECTIVE DATE** | : | **10/2019** |
| **REVISION DATE** | : | **03/2025** |
| **REVISION NUMBER** | : | **4** |

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## 1. PURPOSE

Roux Associates, Inc. and its affiliated companies, Roux Environmental Engineering and Geology, D.P.C, and Remedial Engineering (collectively, "Roux"), have established this heat illness prevention program to assist in preventing workplace accidents, injuries, and illnesses associated with temperature extremes. Roux is committed to taking every precaution to protect employees who might be exposed to heat stress, including establishing safe work practices, heat illness prevention controls, and emergency preparedness, which will be detailed in this plan.

## 2. SCOPE AND APPLICABILITY

Roux's Heat Illness Prevention Program applies to all employees and subcontractors who may be exposed to heat stress in the field.

## 3. ROLES AND RESPONSIBILITIES

The Heat Illness Prevention Program Administrator for Roux is the Corporate Health and Safety Director (CHSD), Brian Hobbs, CIH, CSP. The daily administration of the program is delegated to the Operations Manager (OM) , Office Health and Safety Manager (OHSM), and Site Health and Safety Officer (SHSO). The CHSD has the authority, responsibility, and overall accountability for the comprehensive implementation of this program.

All managers and supervisors shall implement and maintain the heat illness prevention program in work areas where there is a potential for heat stress.

### Management Responsibilities

Management (i.e., Project Managers, Project Principals, OM) must provide the following to promote compliance and foster a good safety culture:

- Comply with applicable standards.
- Inform employees of the provisions of the heat illness prevention program and ensure understanding.
- Provide sufficient water, shade, rest areas, and other heat illness controls for employees.
- Assist with establishing work practices to minimize heat stress risks, such as acclimatization, required rest periods, employee monitoring, and strategic scheduling.
- Encourage employees to report hazardous conditions or heat illness symptoms.
- Provide training to any employee who could possibly be exposed to the risk of heat illness.

### Employee Responsibilities

Employees are expected to actively participate in the organization's heat illness prevention program, which includes the following responsibilities:

- Receive appropriate training with regard to heat stress.
- Understand core concepts of heat illness prevention and follow guidelines to mitigate risks.
- Recognize the signs and symptoms of heat illness and immediately report them.
- Cooperate with workplace inspections and incident investigations.

### Effective Communication

All Roux employees shall be accountable for ensuring there is effective communication with both the field team and their subcontractors. Communication between Roux employees, project leads (e.g., Project Principal, Project

Managers), and subcontractors (if applicable) will be established and maintained so that employees can quickly contact should there be a change in condition or someone at the Site exhibits signs/symptoms of heat stress. These types of incidents would follow Roux's Incident Investigation and Reporting Management Program 2.12 and Roux's Injury Illness Prevention Program 2.22.

## 4. HAZARD IDENTIFICATION AND ASSESSMENT

Prior to scheduled fieldwork, the project team shall ensure a hazard assessment is conducted as it relates to heat stress. This will include a determination of how much heat and exertion employees are likely to be exposed to based on the work activity.

Factors that can increase heat stress include the following:

- Temperatures at the work site;
- Humidity;
- A lack of air movement or exchange;
- The amount of time employees spend working in the heat;
- The time of day work takes place;
- Sources of radiant heat (e.g., sunlight, fire, or hot furnace);
- Work activities that produce heat (e.g., welding);
- Physical contact with warm or hot objects or liquids;
- The clothing and PPE employees are required to wear; and/or
- Physically strenuous work.

Results of the hazard analysis should be used to determine appropriate controls for mitigating heat stress, whenever possible, for planning acclimatization, and for developing work schedules that rotate workers to limit employee exposure. Typically, the Site Supervisor (SS) or Site Health and Safety Officer (SHSO) shall conduct worksite observations to ensure all employees follow heat illness prevention procedures and that hazards are sufficiently controlled. However, all employees should be continuously evaluating work site conditions and be able to recognize heat stressors in order to prevent heat illnesses.

## 5. TRAINING

All Roux employees shall be trained on the topics listed below prior to beginning work, which may potentially expose workers to heat stress.

### Training Topics

- The environmental, behavioral, and personal risk factors for heat illness, such as radiant heat sources, exertion, clothing and PPE, and use of alcohol or drugs.
- Types of heat illnesses, common symptoms, and appropriate emergency response for each.
- The knowledge that mild symptoms may quickly become more severe or life-threatening.
- The importance of immediately reporting any signs or symptoms of heat illness to the supervisor.
- The employer's responsibility to provide shade, water, access to first aid, and cool-down rests during work and the employee's freedom to exercise their rights under this standard without fear of retaliation.

- The employer's heat stress plan and its procedures:
  - Procedures for employees who are newly assigned to work in high-heat areas.
  - The organization's acclimatization methods, as applicable.
  - Heatwave procedures.
  - Heat illness and emergency response, including contact information.
- The importance of frequent consumption of water and the taking of rest breaks.
- How heat illness prevention applies to employees' specific tasks.

**Increasing Training Effectiveness:**

- The SS/SHSO will hold daily tailgates with the field team to go over daily work tasks and basic safety information, including Job Safety Analysis (JSA) review, as well as incorporate elements such as current weather and effects on the work activities for the day.
- If a heat wave or high heat is anticipated, OHSMs and/or OMs will provide communications to Roux employees prior to their shifts and remind them of any special high heat procedures.

## 6. DRINKING WATER

Employees shall be given access to potable drinking water. Access to water will be provided as near as possible to where employees are working, and there shall be enough locations so employees will have sufficient water. Water should be fresh and pure, free of taste or smell that would discourage employees from drinking, comfortably cool, and obtained from an approved source.

The recommendation is that during warm or hot weather, employees drink four 8-ounce glasses of water, or a total of one quart per hour, throughout the entire work shift. Easy access to sufficient potable drinking water throughout the work shift encourages employees to drink.

During a heat wave, water shall be replenished more often to keep it available and cool. Water containers (and all spouts and levers) must be kept clean. If able, provide single-use drinking cups with appropriate waste receptacles. Accessible sanitation facilities shall also be maintained at work Sites as appropriate.

**SS/SHSO is responsible for the following:**

- Drink sufficient water before, during, and after work shifts to maintain hydration.
- Encourage frequent drinking of small amounts of water throughout the shift. In high-heat environments, remind field staff and subcontractors that drinking extra water may be necessary.
- Discourage the choice of drinks with caffeine or sugar that may possibly dehydrate employees instead of water. Also, discourage the drinking of alcohol.
- Monitor the water supply.
- If employees become dehydrated and are unable to alleviate symptoms with the steps below, get them immediate medical attention.

**Field Staff/Subcontractors are responsible for the following:**

- Drink sufficient water before, during, and after work shifts to maintain hydration. Drink up to 4 cups of water per hour, especially during hot weather.
- Be aware that in high-heat situations, you may need to drink more water.

- Monitor yourself and others for signs of dehydration. If you feel dehydrated:
  - Follow Roux's Incident Notification Flowchart;
  - Rest in the shaded resting area; and
  - Drink water in small amounts, but frequently.

## 7. ACCESS TO SHADE

Access to shaded areas will be provided to Roux staff, as needed. Employees are encouraged to use these areas when they feel overheated. Roux's SS/SHSO shall oversee the proper implementation of shaded areas and will communicate these locations on a daily basis with the Field Team.

Depending on the Site, shaded areas can include the following:

- Pop-up Tents
- Canopies
- Umbrellas
- Structures mechanically ventilated or open to air movement
  - Garage
- Conex-mounted RV canopies
- Full and thick tree canopies that block direct sunlight
- Buildings
- Enclosed areas only if they provide cooling comparable to shade in open-air
  - Vehicles with air conditioning

### Shaded Area Requirements

The shade shall be strong enough to cool employees down and other shadows should not be visible in the shade. The shade will be provided by Roux when the air temperature exceeds 80°F. If the temperature is less than 80°F, shade will be available and provided upon request. The SS/SHSO will monitor conditions to determine when the air will exceed (and is exceeding) 80°F. For most Sites, air will be monitored hourly and shade will be set up immediately if 80°F is exceeded.

Shade shall be located as close as practical to areas where employees are working and is easily accessible. These areas will be considered safe and free from other hazards. Shaded areas should be large enough to accommodate all employees who are on a break, resting, or recovering without crowding. Should natural vegetation be used for shade, the SS/SHSO will evaluate shade for effectiveness.

### Field Staff Responsibilities

Field Staff should take ordinary rest breaks in the shaded areas and monitor themselves for signs of heat stress, and go to the shaded area when they need to cool down. Any issues or problems with shaded areas should be reported to the SS/SHSO for the Site.

If shaded areas are used for a preventative cool-down, the SS/SHSO will ensure the affected employees will remain in the shade until they begin to feel better. The SS/SHSO will continue to monitor the employees and ask them if

they are experiencing symptoms of a heat illness. Employees shall not be sent back to work before symptoms have ended and at a minimum at least 10 minutes of rest.

## 8. MONITORING AND SCHEDULING

### Monitoring

The SS/SHSO and Field Team will continue to monitor themselves and others for signs of heat illness. The SS/SHSO will monitor when the air temperature exceeds 80°F. Throughout the summer, weather and temperature will be monitored at least two weeks ahead, and the work schedule will be planned to accommodate the expected weather.

### Scheduling

Scheduling accommodations may include:

- Working during cooler hours of the day
- Working at night
- Stopping work early
- Rescheduling work activities
- Increasing frequency of breaks

In general, strenuous work activities shall be rescheduled to the coolest parts of the day.

Special precautions are required for temperatures above 80°F, 95°F, and heat waves. These precautions are provided in the sections below.

## 9. ACCLIMATIZATION AND NEW EMPLOYEE PROCEDURES

Roux requires employees to be acclimatized in order for them to better tolerate heat in the workplace. Acclimatization is the physical process of adapting to a different thermal environment, allowing a better toleration of heat. Acclimatization procedures require gradual exposure that gives the employee time to adjust to each level of exposure. Acclimatization is essential for new employees but is necessary for all employees when the temperature significantly changes. Heat stress is much more likely if these procedures are not followed.

The SS/SHSO is responsible for observing new employees during their first 14 days of employment in high-heat areas.

Re-acclimatization is necessary if employees are absent from high-heat environments for a week or more or the temperature increases significantly.

## 10. HEATWAVE AND EXTREME HEAT PROCEDURES

### Heat Wave Procedures

A heat wave is defined as consistent temperatures over 80°F or if the temperature is 10° higher than the average daily temperatures in the preceding 5 days.

SS/SHSO and Project Management (e.g., PP, PM) shall closely observe and monitor employees during a heat wave. The Field Teams should institute a ratio of one SS to 20 or fewer employees, a mandatory buddy system, or

a consistent practice for supervisors to check on employees. Pre-shift meetings to review high-heat procedures with employees will be carried out to emphasize work-rest schedules, drinking water, shade, etc.

### Extreme Heat Procedures

When work-site temperatures equal or exceed 95°F, the employer will enact extreme heat procedures:

- Employees will be closely observed by the SS/SHSO for signs of heat illness. New employees will be supervised for acclimatization.
- Effective communication and monitoring will be ensured through the use of periodic check-ins via phone with the SS/SHSO. Communications between employees and Project Management (e.g., PP/PM) will be established and maintained. Mandatory 10-minute break periods are required for every two hours worked. SS/SHSO must enforce this rule.
- Pre-shift safety tailgate meetings will occur to review procedures and to remind employees to drink water and take cool-down rests if needed. SS/SHSO shall remind employees to rest and drink water. Employees should drink more water than usual.

## 11. EMERGENCY RESPONSE PROCEDURES

Roux is dedicated to providing prompt appropriate care for all employees who report or show symptoms of heat illness. If an employee shows signs of heat illness, they will be monitored and shall not be left alone or sent home without being offered first aid or emergency medical services. If on-site personnel require any medical treatment, the following steps will be taken:

1. Notify Roux's Project and Corporate Management Team for any work-related injury and/or illness occurrence, and communicate with the contracted Occupational Health Care Management Provider, AllOne Health (AOH), immediately following the notifications provided above.
2. Based on discussions with the Project Team, Corporate Management, and the AOH evaluation, if medical attention beyond onsite First Aid is warranted, transport the injured/ill person (IP) to the Urgent Care Center, or notify the Fire Department or Ambulance Emergency service and request an ambulance or transport the victim to the hospital, and continue communications with Corporate Management Team. An Urgent Care/Hospital Route map with locations and directions is provided within Site Specific Health and Safety Plans.
3. First aid medical support will be provided by onsite personnel trained and certified in First Aid, Cardio Pulmonary Resuscitation (CPR), Automatic External Defibrillation (AED), and Blood-Borne Pathogens (BBP) Awareness, until relieved by emergency medical services (EMS).
4. The SHSO and Project Manager will perform a Loss Investigation (LI), and the Project Team will complete the final Loss Report.

## 12. HEAT-RELATED ILLNESSES

### Heat Stress

Heat stress is a significant potential hazard and can be associated with heavy physical activity and/or the use of personal protective equipment (PPE) in hot-weather environments. For these reasons, the company will provide potable drinking water and access to shade or other areas of relief (i.e., air-conditioned vehicles and work trailers). Supervisors, prior to supervising personnel in the field as well as all personnel involved with the fieldwork of a project, are trained in this HASP that includes preventing heat-related illnesses and the below procedures in response to heat-related symptoms and illness. Since much of our work is dependent upon environmental factors beyond our control, we must closely monitor air temperature and humidity and be aware of avoiding radiant heat sources and providing as much air circulation as possible wherever we work. Physical factors that need to be evaluated as part of our Job Safety Analysis (JSA) reviews include the level of physical activity and duration of work

and the type (i.e., color, weight breathability) of the clothing we select. In addition, personal factors such as age, weight, fitness, drug/alcohol use, and prior history of heat-related illness need to be considered.

Heat cramps are brought on by prolonged exposure to heat. As an individual sweats, water and salts are lost by the body, resulting in painful muscle cramps. The signs and symptoms of heat stress are as follows:

- Severe muscle cramps, usually in the legs and abdomen;
- Exhaustion, often to the point of collapse; and
- Dizziness or periods of faintness.

First aid treatment includes, but is not limited to, shade, rest, and fluid replacement. Typically, the individual should recover within one-half hour while being monitored constantly. If the individual has not improved substantially within 30 minutes and the body temperature has not decreased, the individual should be transported to a hospital for medical attention.

**Heat Exhaustion**

Heat exhaustion may occur in a healthy individual who has been exposed to excessive heat while working or exercising. The circulatory system of the individual fails as blood collects near the skin to rid the body of excess heat through transference. The signs and symptoms of heat exhaustion are as follows:

- Rapid and shallow breathing;
- Weak pulse;
- Cold and clammy skin with heavy perspiration;
- Skin appears pale;
- Fatigue and weakness;
- Dizziness; and
- Elevated body temperature.

First aid treatment includes, but is not limited to, cooling the victim, elevating the feet, and replacing fluids. If the individual is not substantially improved within 30 minutes and the body temperature has not decreased, the individual should be transported to the hospital for medical attention.

**Heat Stroke**

Heat stroke occurs when an individual is exposed to excessive heat and stops sweating. This condition is classified as a MEDICAL EMERGENCY requiring immediate cooling of the victim and transport to a medical facility. The signs and symptoms of heat stroke are as follows:

- Dry, hot red skin;
- Body temperature approaching or above 105 degrees F;
- Confusion, altered mental state, slurred speech;
- Seizures;
- Large (dilated) pupils; and
- Loss of consciousness – the individual may go into a coma.

First aid treatment requires immediate cooling and transportation to a medical facility. Heat stress is a significant hazard if any type of protective equipment (semi-permeable or impermeable) that prevents evaporative cooling is worn in hot-weather environments.

## 13. OTHER HEAT CONTROLS

### Clothing and PPE

Employees should choose clothing that is reflective, light-colored, lightweight, loose-fitting, and breathable. Clothing should cover the exposed parts of the body. In direct sun, hard hats with a brim or bill may be helpful. Should specialized cooling garments be applicable, please consult with your OM/OHSM/CHSD.

### Managing Employee Risk Factors

It is recommended that employees are aware of how their health can affect their risk of heat stress. The following increases personnel's risk for a heat-related illness:

- A poor level of physical fitness
- Obesity
- Chronic or acute illnesses
- Conditions such as diabetes, heart disease, or high blood pressure
- Certain medications, such as diuretics
- Age (60+)

Employees should:

- Maintain their health outside of work
- Be aware of the effects of medications
- Drink adequate amounts of water
- Eat light, cool meals during work shifts and save heavy meals until after the shift is over
- Do not skip meals:  food helps replace electrolytes when sweating
- Take breaks as needed
- Do not consume alcohol prior to working in a hot environment

# APPENDIX D

Personal Protective Equipment (PPE) Management Program

# PERSONAL PROTECTIVE EQUIPMENT
# MANAGEMENT PROGRAM

| | | |
|---|---|---|
| **CORPORATE HEALTH AND SAFETY DIRECTOR** | : | **Brian Hobbs, CIH, CSP** |
| **EFFECTIVE DATE** | : | **01/2019** |
| **REVIEW DATE** | : | **03/2025** |
| **REVISION NUMBER** | : | **6** |

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## 1. PURPOSE

Roux Associates, Inc. and its affiliated companies, Roux Environmental Engineering and Geology, D.P.C., and Remedial Engineering (collectively, “Roux”), have instituted the following program to establish guidelines for the selection of personal protective equipment (PPE) for use by Roux personnel performing field activities in hazardous environments. PPE is not meant to be a substitute for engineering, work practice, and/or administrative controls, but PPE should be used in conjunction with these controls to protect employees in the workplace. Clothing, body coverings, and other accessories designed to prevent worker exposure to workplace hazards are all types of PPE. To ensure adequate PPE,, employee-owned PPE is assessed on a case-by-case basis to ensure its adequacy, maintenance, and sanitation.

## 2. SCOPE AND APPLICABILITY

These guidelines apply to all PPE selection decisions to implement the Roux program. The foundations for this program are the numerous Occupational Health and Safety Administration (OSHA) standards related to PPE cited in 29 CFR 1910 Subpart I, 29 CFR 1926 Subpart E, and the hazardous environment work employee protection requirements under the OSHA Hazardous Waste Operations and Emergency Response (HAZWOPER) standard at 29 CFR 1910.120 and 1926.65 as well as California OSHA (Cal/OSHA) Title 8 5192 HAZWOPER. To ensure hazard assessments are documented, the levels of protection, types of protection, and tasks requiring protection are covered in site-specific Health and Safety Plans (HASPs) and Job Safety Analyses (JSAs).

## 3. PROCEDURES

Due to the varied nature of site activities and the different potential hazards associated with different sites, several aspects must be considered when selecting PPE. The following text describes PPE selection logic and provides guidelines and requirements for the appropriate selection and use of PPE.

### 3.1 Introduction

To harm the body, chemicals must first gain entrance. The intact skin and respiratory tract are usually the first body tissues that are exposed to chemical contaminants. These tissues provide barriers to some chemicals but, in many cases, are damaged themselves or are highly permeable by certain chemical compounds. PPE, therefore, is used to minimize or eliminate chemical compounds from coming into contact with these first barrier tissues.

Proper PPE selection is essential in preventing exposure. The Project Manager (PM) or health and safety personnel making the selection will have to consider several factors. The level of protection, type, and kind of equipment selected depends on the hazardous conditions. In some cases, additional selection factors include cost, availability, compatibility with other equipment, and performance. All these factors must be accurately assessed before work can be safely carried out.

#### 3.1.1 Training

Training shall be provided to all field-based employees on how to properly use and care for PPE. Training shall include, but not be limited to, the initial 40-hour HAZWOPER training, annual 8-hour HAZWOPER refresher training, and site-specific PPE training. The training program includes when PPE is necessary, what types of PPE are required; how to don, doff, adjust, and wear PPE; the limitations of PPE; the proper care, maintenance, useful life, signs of PPE failure and emergency procedures to follow in the event of PPE failure and disposal of PPE. In addition, retraining shall be conducted when workplace changes require a change in PPE when an employee demonstrates a lack of knowledge or improper use of PPE is identified.

### 3.2 Types of PPE

The type and selection of PPE must meet certain general criteria and requirements as required under OSHA 29 CFR 1910.132 (General Industry) and 1926.95 (Construction). In addition to these general requirements, specific requirements and specifications exist for some types of PPE that form the basis of the protective clothing scheme. All manufacturer's recommendations for PPE care and use shall be followed. All PPE shall be maintained in a sanitary and reliable condition. Additionally, proper fit and size shall be evaluated to ensure adequate employee protection. Should PPE become defective, it shall be immediately removed from service and discarded.

The following is a list of the common types of specific PPE and the specific requirements for the PPE type, where applicable:

1. Hard Hats - Regulated by 29 CFR 1910.135 and 1926.100; and specified in ANSI Z89.1.
2. Face Shields and Safety Glasses - Regulated by 29 CFR 1910.133 and 1926.102; and specified in ANSI Z87.1.
3. Respiratory Protection - Regulated by 29 CFR 1910.134 and 1926.103.
4. Hand Protection - Regulated by 29 CFR 1910.138
5. Foot Protection - Regulated by 29 CFR 1910.136 and 1926.96; and specified in ANSI Z41.1.
6. Protective Clothing (e.g., fully encapsulated suits, aprons) - Specified in NFPA 1991/1992/1994.

#### 3.2.1 PPE Inspection, Cleaning Maintenance, and Storage

The Office Health and Safety Manager (OHSM) or their designee is responsible for procuring PPE, in accordance with this management program. Single-use/disposable PPE should be purchased and used whenever practicable.

##### 3.2.1.1 PPE Inspection

Roux employees must inspect PPE regularly, before and after each use, to check for defects and damage. Garments and other PPE in central storage must be inspected at least annually or as recommended by the manufacturer by the OHSM, Equipment Manager, and/or their designee. Defective or damaged equipment must be tagged as out-of-service, immediately removed from the work site, and discarded.

##### 3.2.1.2 PPE Cleaning and Maintenance

All Roux employees are responsible for routine cleaning of their assigned PPE. Decontamination of PPE is addressed within Roux's Site Control and Decontamination Program as specified within site-specific health and safety plans. As appropriate, Roux employees should be issued single-use PPE when practicable to minimize the requirement for decontamination. When performing routine cleaning of reusable PPE:

- Always follow the manufacturer's recommendations to avoid damaging the PPE. For example, some cleaning solutions compromise the integrity of protective helmets shell.
- Clean and maintain PPE according to the manufacturer's recommendations. Before being re-issued, PPE should be thoroughly sanitized.
- Disassemble, wash and sanitize reusable respirators after each use. For more information, refer to Roux's Respiratory Protection Management Program.

Employees are not authorized to repair PPE without prior approval from the Health and Safety Department. Reusable PPE may require maintenance by the manufacturer to maintain integrity and certification. Many

manufacturers specify which repairs, if performed by the end-user, will void the product warranty. Therefore, Roux employees are not authorized to perform such maintenance or repairs.

#### 3.2.1.3 PPE Storage

Certain equipment failures can be directly attributed to improper storage. The OHSM or Equipment Manager (or another designated person) is responsible for implementing procedures for appropriate PPE storage, for equipment that is centrally stored. Both central storage and dedicated PPE provided to employees should be stored using the following:

- Store goggles and safety glasses in protective cases.
- Store PPE (such as helmets, boots, etc.) in clean, dust-proof containers or bags out of direct sunlight.
- PPE should ideally be stored under climate-controlled conditions. PPE, stocked gear bags, and/or go-kits should not be stored in hot, cold, or other extreme environmental conditions, and they should not be stored in vehicles (government-owned or rentals) for even short periods of time. Do not store helmets on the rear window shelf of a vehicle.
- Store different kinds of clothing and gloves separately to prevent errors in selection.
- Hang or fold protective clothing in accordance with manufacturers' recommendations.
- Never store contaminated PPE near new protective clothing or street clothing. Potentially contaminated, reusable clothing must be stored (usually bagged) away from new PPE in a well-ventilated area with good airflow around each item until the garment is decontaminated or disposed.
- See Roux's Respiratory Protection Management Program for detailed respirator storage procedures.

### 3.3 Fitting, Donning, and Doffing PPE

Roux shall provide appropriately sized PPE for Roux employees. Improper fitting such as tight-fitting garments, can tear, especially at the knees, crotch, shoulders, back, and elbows due to movement. Protective clothing that is too large can easily snag and/or limit a user's dexterity. The Roux PM shall ensure site/project-specific PPE is kept in a sufficient stock of appropriately sized PPE (e.g., Level C ensembles). The OHSM, Equipment Manager, and/or their designee shall maintain a sufficient stock of appropriately sized PPE in central storage locations.

The Site Supervisor (SS)/Site Health and Safety Officer (SHSO) is responsible for ensuring that site-specific procedures for donning and doffing PPE are incorporated into site-specific HASPs. Donning and doffing procedures may differ depending on the type of PPE and clothing (e.g., Level D, Modified Level D, Level C) utilized. Figures 1-4, provide examples of procedures that can be used to don and doff Levels A through D and are provided as a reference. Site-specific conditions may warrant adjustments to such procedures. Consult the CHSD for additional information.

Site-specific donning and doffing procedures should include using an assistant where necessary. Donning and doffing certain types of PPE without assistance could risk failure or damage. Care must be taken to prevent the spread of contaminants on the PPE. Decontamination procedures should limit the potential for cross-contamination and may include gross decontamination of outer boots, gloves, and suits as necessary to prevent contaminants from tracking into the contamination reduction zone (CRZ) and clean zone(s).

### 3.4 Controlling Hazards Associated with PPE Use

PPE use can pose potential health and safety hazards, which range from minor discomfort to life-threatening height stroke. The magnitude of the potential hazard can vary and will depend on various factors, including an individual's fitness level, the type of PPE worn, the work demands, individual work practices, duration of work, and environmental conditions. The use of PPE can also lead to reduced efficiency and work performance.

Among hazards associated with PPE use, heat stress emerges as a prevalent and significant hazard. Particularly, the use of substantial and/or impermeable attire, such as heavy coveralls, Tyvek suits, splash suits, or Level A or B suits, alongside strenuous work, amplifies the risk of heat stress. Regular monitoring for indications of heat stress is imperative for workers operating in such conditions. Roux's Heat Illness Prevention Program lays out requirements for signs and symptoms as well as preventative measures to mitigate the potential for heat illness. In addition, cold environments can pose adverse risk, such examples include use of non-insulated steel-toe safety shoes in frigid conditions which has the potential to increase the risk of frostbite, while safety gloves worn for chemical protection may inadequately insulate hands from the cold.

It is the responsibility of Project Teams to integrate site-specific protocols to manage PPE hazards into such site-specific HASPs. These protocols can encompass considerations like work/rest ratios, crew rotations, and other accommodations for exceptionally demanding conditions.

The following measures are recommended for controlling hazards posed by PPE use:

- Prioritize through the hierarchy of controls with a focus on elimination, substitution, engineering, work practice and administrative control implementation rather than sole reliance on PPE. PPE shall be considered the last line of worker protection.
- Workers should maintain physical fitness to be able to withstand the potential physical strain of PPE use. A physically fit individual will endure less physiological strain, manifesting in a lower heart rate, reduced body temperature (indicating diminished retained body heat), more effective sweating, slightly lower oxygen consumption, and carbon dioxide production. Furthermore, acclimatization to extreme environmental conditions, such as high temperatures, can influence work performance and physical capacity.
- Understand the symptoms of PPE failure. Workers encountering any of these symptoms while donning PPE should promptly notify their onsite SHSO and/or SS:
    - Perception of odors when wearing a respirator
    - Skin irritation
    - Acute discomfort
    - Difficulty breathing
    - Unusual fatigue
    - Dizziness
    - Inability to see, hear, or speak clearly
    - Uncomfortable restriction of movement
    - Rapid pulse, nausea, or chest pain
- Implement a buddy system. Especially in situations where there is a need for increased level of protection (e.g., Level A-C) workers should work in pairs or teams, and are instructed to monitor the integrity of their team/partners gear and remain alert for symptoms of PPE failure.

### 3.5 PPE Selection

For site-specific work, the Project Manager, in consultation with the Health and Safety Department, has overall responsibility for coordinating the selection and distribution of PPE to field staff and for ensuring that the selected PPE is appropriate for the site-specific hazards. Generally speaking, Roux employees are not engaged in emergency response or perform work at uncontrolled hazardous waste sites; if this is the case, the CHSD shall be made aware of the work and consult with the Project Team on appropriate PPE selection. Most work falls under environmental sampling of low-concentration contaminants in water/soil/perimeter air. Therefore work is carried out in Level D PPE. Upgrades would be based on more information as it becomes available. The United States Environmental Protection Agency (EPA) has created Guidelines for PPE Ensemble Selection to assist workers in selecting PPE ensembles for specific activities and tasks and determining which ensembles and air monitoring equipment should be used to address specific chemicals. Guidelines to Ensembles for Specific Activities/Tasks Where Chemical Exposure is Possible is provided as a reference in Appendix A.

### 3.6 Protective Clothing Selection Criteria

#### 3.6.1 Chemicals Present

The most important factor in selecting PPE is the determination of what chemicals the employee may be exposed to. The number of chemicals on field investigations may range from a few to several hundred. The exact chemicals or group of chemicals present at the site (certain groups tend to require similar protection) can be determined by collecting and analyzing air, soil, water, or other site media samples. When data are lacking, research into the hazardous materials used or stored at the site can be used to infer potential chemical exposures on the site.

The most appropriate clothing shall be selected once the known or suspected chemicals have been identified and considered based on the type of work to be performed.

Protective garments are made of several different substances for protection against specific chemicals. There is no universal protective material. All will decompose, permeate, or otherwise fail to protect under given circumstances. Fortunately, most manufacturers make guides to using their products (i.e., Dupont's Tyvek™ Permeation Guide). These guides are usually for gloves and coveralls and typically provide information regarding chemical degradation rates (failure of the material to maintain structural integrity when in contact with the chemical) and may provide information on the permeation rate (whether or not the material allows the chemical to pass through). When permeation tables are available, they shall be used with degradation tables to determine the most appropriate protective material for the specific chemical hazard(s).

During most site work, chemicals are usually in mixed combinations, and the protective materials are not in continuous contact with pure chemicals for long periods; therefore, the selected material may be adequate for the particular chemical of most concern and the type of work being performed, yet not the "best" protecting material for all site chemicals and activities. Selection shall depend upon the most hazardous chemicals based on their hazards and concentrations. Sometimes, layering (i.e., using several layers of protective materials) affords the best protection.

#### 3.6.2 Concentration of the Chemical(s)

One of the major criteria for selecting protective material is the concentration of the chemical(s) in air, liquid, and solid state. Airborne and liquid chemical concentrations should be compared to the OSHA standards, American Conference of Governmental Industrial Hygienists (ACGIH) and National Institute for Occupational Safety and Health (NIOSH) guidelines to determine the level of skin or other absorptive

surface (e.g., eyes) protection needed. While these standards are not designed specifically for skin exposed directly to the liquid, they may provide skin designations indicative of chemicals known to have significant skin or dermal absorption effects. For example, airborne levels of PCBs on-site may be low because they are not very volatile, so the inhalation hazard may be minimal; however, PCB-containing liquid coming in direct contact with the skin may cause overexposure. Thus, PCBs have been assigned a skin designation in the OSHA and ACGIH exposure limit tables.

### 3.6.3 Physical State

The characteristics of a chemical may range from nontoxic to highly toxic, depending on its physical state. Inorganic lead in soil would not be considered toxic to site personnel unless it became airborne since it is generally not absorbed through the intact skin. However, organic lead in a liquid could be readily absorbed through the skin. Soil is frequently contaminated with hazardous materials. Concentrations will vary from a few parts per million to nearly one hundred percent by volume. The degree of hazard depends on the type of soil and concentration of the chemical. Generally speaking, “dry” soils that have not become airborne do not pose a hazard to site personnel if they take minimal precautions, such as wearing lightweight gloves.

### 3.6.4 Length of Exposure

The length of time a material is exposed to a chemical increases the probability of a breakthrough. Determinations of actual breakthrough times for short-term exposures indicate that several materials that would be considered inadequate under long-term exposures can be used. Different materials that would be considered inadequate under long-term exposures can be used. It should be kept in mind that during the manufacturer’s permeation testing, a pure (100% composition) liquid is usually placed in direct contact with the material, producing a worst-case situation.

### 3.6.5 Abrasion

When selecting protective clothing, the job the employee is engaged in must be taken into consideration. Persons moving drums or performing other manual tasks may require added protection for their hands, lower chest, and thighs. Using leather gloves and a heavy apron over other normal protective clothing will help prevent damage to the normal PPE and thus reduce worker exposures.

### 3.6.6 Dexterity

Although protection from skin and inhalation hazards is the primary concern when selecting PPE, the ability to perform the assigned task must be maintained. For example, personnel cannot be expected to perform work that requires fine dexterity if they must wear thick gloves. Therefore, the PPE selection process must consider the task being performed and provide PPE alternatives or techniques that maintain dexterity while still protecting the worker (e.g., wearing tight latex gloves over more bulky hand protection to increase dexterity).

### 3.6.7 Ability to Decontaminate

If disposable clothing cannot be used, the ability to decontaminate the materials selected must be taken into consideration. Once a chemical makes contact with the material, the PPE must be cleaned before reusing it. If the chemical has completely permeated the material, the clothing cannot be adequately decontaminated, and the material should be discarded.

### 3.6.8 Climactic Conditions

The human body works best with few restraints from clothing. Protective clothing adds a body burden by increasing weight, restricting movement, and preventing the body's natural cooling process. In severe situations, a modified work program must be used.

Some materials act differently when they are very hot and very cold. For example, PVC becomes almost brittle in frigid temperatures. If there are any questions about the stability of the protective materials under different conditions, the manufacturer should be contacted before using PPE in the field.

### 3.6.9 Work Load

Like climactic conditions, the type of work activity may affect work duration and personnel's ability to perform certain tasks. Similarly, the amount of protective materials a person wears will affect their ability to perform certain tasks. For example, a person in a total encapsulating suit, even at 72˚F, cannot work for more than a short period of time without requiring a break.

The work schedule should be adjusted to maintain the health of the employees. Modified work practices may include allowing more time for individual job tasks and implementing work/rest cycles per NIOSH and ACGIH thermal stress recommendations. Special consideration should be given to the selection of clothing that protects the worker and adds the least burden when personnel are required to perform strenuous tasks. Excessive bodily stress frequently represents the most significant hazard encountered during fieldwork.

### 3.6.10 Types of Protective Materials

1. Cellulose or Paper: suitable for nuisance dust and coarse fibers.
2. Natural and Synthetic Fibers
    a. Tyvek™: suitable small-sized hazardous particles, including lead, asbestos, and mold.
    b. Tychem™: is suitable for a variety of hazards, from light liquid splashes to heavy exposures to industrial chemicals and agents.
    c. Nomex™: is suitable for flame-resistance, radiation resistance, acids, alkalis, fluorocarbon refrigerants (CFCs), and non-polar solvents.
3. Elastomers
    a. Polyethylene: suitable for polar substances and solvents.
    b. Saran™ : suitable for flame-resistance, acids, alkalis, oils, and organic solvents.
    c. Polyvinyl Chloride (PVC): suitable for alcohols, oils, inorganic acids, alkalis, and salts.
    d. Neoprene: suitable for polar/non-polar substances, petroleum mixtures, and ≤ 200 F heat resistance.
    e. Butyl Rubber: suitable for polar substances and solvents.
    f. Viton: suitable for non-polar substances, petroleum mixtures, and ≤ 200 F heat resistance.
    g. Nitrile: suitable for polar/non-polar substances and petroleum mixtures.

## 3.7 Protection Levels

### 3.7.1 Level A Protection

Level A protection (a fully encapsulated suit) is used when skin hazards exist or when no known data positively rule out skin and other absorption hazards. Per the Roux Respiratory Protection Management Program, Roux employees are not authorized to enter an IDLH atmosphere; therefore, the program does

not allow supplied air respirators (SARs) or self-contained breathing apparatus (SCBA). At no time will Level A work be performed without the consent of the CHSD and OM.

The following conditions warrant the use of Level A protection:

- Hazardous substances have been identified and require the highest level of protection for skin, eyes, and the respiratory system;
- The atmosphere contains less than 19.5% oxygen or an atmosphere immediately dangerous to life and health (IDLH) that have not been ruled out;
- Site operations involve a high potential for splash, skin immersion, or exposure to suspected skin hazards;
- Direct-reading instruments indicate high levels of unidentified but potentially hazardous vapors or gases in the air (e.g., Total atmospheric readings on the Photoionization Detector (PID), Flame Ionization Detector (FID), and similar instruments indicate 500 to 1,000 ppm of unidentified substances); and
- Direct-reading instruments are not available to test the air and suspected highly toxic substances may be present.

It may be necessary to base the decision to use Level A protection on indirect evidence. Other conditions that may indicate the need for Level A protection include:

- Confined spaces;
- Sites containing known skin hazards;
- Sites with no established history to rule out skin and other absorption hazards;
- Site exhibiting signs of acute mammalian toxicity (e.g., dead animals, illnesses associated with past entry into the site by humans);
- Sites at which sealed drums of unknown materials must be opened;
- Visible indicators such as leaking containers or smoking chemical fires; and
- Extremely hazardous substances (e.g., cyanide compounds, concentrated pesticides, Department of Transportation Poison "A" materials, suspected carcinogens, and infectious substances) are known or suspected to be present, and skin contact is possible.

**Table 1: Recommended Level A Ensemble Components**

| Component | Type | Manufacturer / Recommended Material | Notes |
|---|---|---|---|
| Respirator | Positive-pressure full-facepiece SCBA or positive-pressure supplied-air respirator with escape SCBA[1] | NIOSH approved respirator | See Roux's Respiratory Protection Program. Roux employees are not approved to utilize Supplied Air (SAR) and/or Self-contained breathing apparatus (SCBA). |
| Suit | Totally encapsulated chemical- and vapor-protective suit | DuPont® Tychem™ or Responder CSM™ | Other material types may be substituted based on known contaminants. |
| Inner Gloves | Lightweight | Nitrile, Neoprene, PVC, or Viton® | Other material types may be substituted based on known contaminants. |
| Outer Gloves | An integral part of the suit | See Appendix A for additional information | Confirm chemical performance rating for suspected contaminants. Glove material may have different performance ratings than suit material. All fully-encapsulating suit materials must be compatible with substances involved. |
| Inner Boot | Integral Part of Suit | - | - |
| Outer Boot | HazMat boot[2] | – | Confirm chemical performance rating of outer boot for suspected contaminants. Provides slip/abrasion resistance. |
| Hard Hat | Standard[3] | - | - |

[1] Must be NIOSH approved.
[2] Must meet NFPA 1991/NFPA 1994 requirements depending on the site hazard assessment.
[3] Must comply with the applicable ANSI standard.

### 3.7.2 Level B Protection

Level B protection is utilized when the highest level of respiratory protection is needed, but hazardous material exposure to the few unprotected areas of the body is unlikely. At no time will Level B work be performed by Roux personnel without the consent of the CHSD and OM.

The following conditions suggest a need for Level B protection:

- The type and atmospheric concentration of toxic substances have been identified, and they require the highest level of respiratory protection;

- IDLH atmospheres where the substance or concentration in the air does not present a severe skin hazard;
- The type and concentrations of toxic substances do not meet the selection criteria permitting the use of air-purifying respirators and
- It is highly unlikely that the work being done will generate high concentrations of vapors, gases, particulates, or splashes of materials that will affect personnel's skin.

**Table 2: Recommended Level B Ensemble Components**

| Component | Type | Manufacturer / Recommended Material | Notes |
|---|---|---|---|
| Respirator | Positive-pressure full- facepiece SCBA or positive-pressure supplied-air respirator with escape SCBA[1] | NIOSH approved respirator | See Roux's Respiratory Protection Program. Roux employees are not approved to utilize Supplied Air (SAR) and/or Self-contained breathing apparatus (SCBA). |
| Suit | Chemical-resistant clothing (liquid-splash protective suit with integral boot and hood)[2] | DuPont® Tychem® CPF 3 | Other material types may be substituted based on known contaminants. |
| Inner Gloves | Light/medium weight | Nitrile, Neoprene, PVC, Viton®, or PE/EVAL | Other material types may be substituted based on known contaminants. |
| Outer Gloves | Medium/heavy weight | Butyl MIL-G12223, Nitrile, Neoprene, PVC, Viton®, PE/EVAL, or heavy weight Nitrile or Neoprene | Confirm chemical performance rating. Other material types may be substituted based on known contaminants. |
| Inner Boot | Safety work boot | – | Other boot types may be substituted based on known contaminants and physical hazards. Puncture resistant shanks may be required. |
| Outer Boot | Heavy weight Latex booties or outer boots[2] | Latex booties are used to protect suit for light duty applications. NFPA-rated outer boots may be required based on anticipated site conditions and tasks. | Other outer boot coverings may be substituted based on known contaminants/anticipated tasks. |
| Hard Hat | Standard[3] | - | - |

[1] Must be NIOSH approved.
[2] Must meet NFPA 1991/NFPA 1994 requirements depending on the site hazard assessment.
[3] Must comply with the applicable ANSI standard.

### 3.7.3 Level C Protection

Level C protection is utilized when skin and respiratory hazards are well defined, and the criteria for using negative pressure respirators have been fulfilled (i.e., known contaminants and contaminant concentrations, acceptable oxygen levels, approved filter/cartridge available, known cartridge service

life, etc.). Level C protection may require an emergency escape respirator during specific initial entry and site reconnaissance situations or when applicable after that.

**Table 3: Recommended Level C Ensemble Components**

| Component | Type | Manufacturer / Recommended Material | Notes |
|---|---|---|---|
| Respirator | Full-face air-purifying respirator with appropriate cartridge[1] | NIOSH approved respirator | See Roux's Respiratory Protection Program for information about the brand, model, acceptable configurations, and specific care instructions for the standard issue respirator. Half-faced respirators may be donned if hazard assessment warrants. |
| Suit | Puncture/tear resistant suit material with boot and hood[2] | DuPont® Tychem® CPF 2/CPF 3 or Saranex | Other material types may be substituted based on known contaminants, if considered more appropriate. |
| Inner Gloves | Light weight | Nitrile, Neoprene | Other material types like Neoprene, PVC, Viton®, or PE/EVAL may be substituted based on known contaminants, if considered more appropriate. |
| Outer Gloves | Medium/heavy weight | Butyl, Nitrile, or other work glove | Confirm chemical performance rating. Other material types may be substituted based on known contaminants, if considered more appropriate. |
| Inner Boot | Safety work boot | – | Other boot types may be substituted based on known contaminants and physical hazards. Puncture resistant shanks may be required. |
| Outer Boot | Heavy weight Latex booties or outer boots[2] | Latex booties are used to protect suit for light duty applications. NFPA rated outer boots may be required based on anticipated site conditions and tasks. | Other outer boot coverings may be substituted based on known contaminants. |
| Hard Hat | Standard[3] | - | - |
| Eye Protection | Safety glasses/goggles[3,4] | - | - |

1 Must be NIOSH approved.
2 Must meet NFPA 1991/NFPA 1994 requirements depending on the site hazard assessment.
3 Must comply with the applicable ANSI standard.
4 In the case a ½ face respirator is worn eye protection is required.

### 3.7.4 Level D Protection

Level D is the basic work uniform. Level D is worn when the atmosphere contains no known hazard. Work functions preclude splashes, immersion, potential for inhalation, or direct contact with hazard chemicals. For most Roux field jobs Level D is the standard minimum level of protection worn.

**Table 4: Recommended Level D Ensemble Components**

| Component | Type | Manufacturer / Recommended Material | Notes |
|---|---|---|---|
| Respirator | None | - | - |
| Suit | Coveralls, street clothes, or disposable Tyvek suit | – | Other types may be substituted as appropriate. |
| Inner Gloves | N/A | | |
| Outer Gloves | As appropriate for physical hazards and for comfort | – | - |
| Boot | Safety work boot | – | Roux's standard safety boot is 6", steel/composite safety toe, and electrical hazard (EH) rated. |
| Outer Boot Covering | As appropriate | – | – |
| Hard Hat | Standard[3] | - | - |
| Eye Protection | Safety glasses/goggles[3] | - | - |
| High Visibility Clothing | High Visibility Vest /Jacket[3] | - | ANSI Class 2 Safety Vests/Jackets are the standard. Class 3 Safety vests/jackets shall be worn in worksites with 50+ MPH traffic. |
| Hearing Protection | Ear muffs/plugs | - | Workers exposed to excessive noise shall don hearing protection. Refer to Roux's Hearing Conservation Program for more information. |

[1] Must be NIOSH approved.
[2] Must meet NFPA 1991/NFPA 1994 requirements depending on the site hazard assessment.
[3] Must comply with the applicable ANSI standard.

### 3.7.5 Level E Protection

Level E protection is used when radioactivity above 10 millirems per hour (mrems/hr) is detected at the site. Personal protective equipment for Level E includes:

- Coveralls;
- Air purifying respirator (i.e., P100 or equivalent);
- Time limits on exposure;
- Appropriate dermal protection for the type of radiation present, and

- Radiation dosimetry and ambient monitoring.

### 3.7.6 Additional Considerations

Fieldwork will contain a variety of situations due to chemicals in various concentrations and combinations. These situations may be partially ameliorated by following the work practices listed below:

1. Foot protection is needed on every site. If the ground to be worked on is contaminated with liquid, and it is necessary to walk in the chemicals, some sort of protective shoe coverings or "booties" can be worn over the boots. Shoe coverings should be designed with soles to help prevent slips. Using disposable shoe coverings when contacting liquid contamination can on and reduce overall decontamination requirements. If non-liquids are to be encountered, a Tyvek™ bootie could be used. The advantage of booties is questionable if the ground contains any sharp objects. Boots should be worn with either cotton or wool socks to help absorb perspiration.
2. If the site requires hard hats, chin straps should be used if a person is stooping over where his/her hat may fall off. Respirator straps should not be placed over the hard hats, as this will affect the respirator's fit.

   Some types of protective materials conduct heat and cold readily. In cold conditions, natural material clothing should be worn under protective clothing. Protective clothing should be removed before allowing a person "to get warm." Applying heat, such as with a space heater, to the outside of the protective clothing may drive contaminant permeation. In hot weather, under clothing will absorb sweat. It is recommended that workers use all cotton undergarments.
3. Body protection should be worn and taped to prevent anything from running into the top of the boot. Gloves should be worn and taped to prevent substances from entering the top of the glove. Duct tape is preferred, but masking tape can be used. When aprons are used, they should be taped across the back for added protection. However, this should be done so that the person has mobility.
4. Atmospheric conditions such as precipitation, temperature, wind direction, humidity, wind velocity, and pressure determine the behavior of contaminants in air or the potential for volatile materials to become airborne. These parameters should be considered in determining the need for and the level of protection.
5. A program must be established for periodic air monitoring during site operations. Without an air monitoring program, any changes to air quality would go undetected and might jeopardize on-site personnel. Monitoring can be done with various types of air pumps and filtering devices, followed by laboratory analysis of the filtration media, personnel dosimeters, and periodic walk-throughs by personnel carrying real-time direct-reading survey instruments.
6. For operations in the exclusion zone, different levels of protection may be selected, and various types of chemical-resistant clothing may be worn. This selection should be based on the job function, reason for being in the area, and the potential for skin contact with, or inhalation of, the chemicals present.
7. Escape masks must be readily available when levels of respiratory protection do not include an SCBA and the possibility of an IDLH atmosphere exists. The use and placement of escape masks should be made on a case-by-case basis. Escape masks should be strategically located at the site in areas with a higher potential for vapors, gases, or particulates.

## 3.8 Decontamination of PPE

Roux prefers the use of single-use PPE to minimize the requirement for significant decontamination. The following procedures shall be considered should a field project require the need for decontamination of PPE. Roux has also developed a Site Control and Decontamination Management Program.

### 3.8.1 Site-Specific Decontamination Plans

The Project Manager (or another authorized individual) is responsible for formulating the site-specific PPE decontamination plan as an integral part of the Health and Safety Plan (HASP). The SHSO/SS' responsibilities include ensuring the plan's implementation and adapting it as site conditions evolve. Refer to Roux's Site Control and Decontamination Management program for additional requirements.

The decontamination plan, often created in conjunction with PPE selection, must be established prior to workers entering potentially contaminated areas. The plan should address the following key aspects:

- Decontamination Hazard Evaluation and Risk Assessment: Assessing risks associated with decontamination processes.
- PPE for Decontamination Line Assistants: Specifying appropriate PPE for personnel involved in decontamination.
- Roles and Responsibilities During Decontamination: Defining tasks and duties during the decontamination process.
- Layout and Quantity of Decontamination Stations: Determining the number and arrangement of decontamination stations, along with the designated PPE decontamination procedures at each station.
- Decontamination Equipment: Identifying the necessary equipment for effective decontamination.
- Decontamination Methods: Describing the methods and protocols for decontaminating personnel and equipment.
- Preventing Contamination of Clean Areas: Outlining procedures to prevent contamination of uncontaminated zones.
- Minimizing Contact with Contaminants During CPC Removal: Providing guidelines to reduce wearer exposure during the removal of Chemical Protective Clothing (CPC).
- Disposal Procedures for Inadequately Decontaminated Clothing and Equipment: Ensuring proper disposal practices for items that have not been adequately decontaminated.

The initial decontamination plan, which is developed in conjunction with the initial site hazard assessment, should be based on worst-case assumptions about the extent and type of contamination.

### 3.8.2 Emergency Decontamination of PPE

The designated SHSO/SS (or another authorized individual) assumes responsibility for addressing emergency decontamination within the site-specific Health and Safety Plan (HASP). Emergency decontamination may become necessary due to events such as fires, explosions, sudden violent storms, or medical emergencies. The emergency decontamination plan must clearly delineate emergency egress routes, as well as procedures for both decontamination and egress.

Some key points to consider regarding emergency decontamination.

- Immediate Notification: The SHSO/SS (or their designated counterpart) must be promptly informed of any circumstances requiring emergency decontamination. Additionally, they are accountable for tracking all personnel during the process.
- Urgent Medical Treatment: If urgent medical attention is necessary, decontamination of PPE may be delayed until the victim's condition stabilizes.
- Balancing Priorities: When feasible, decontamination should occur without hindering essential first aid. However, if a worker is exposed to an extremely toxic or corrosive substance, immediate decontamination is crucial to prevent severe injury or fatality.

- Heat-Related Illness: In cases of heat-related illness, protective clothing must be promptly removed (refer to Roux's Heat Illness Prevention Program).

Emergency Facilities:

- For workers potentially exposed to corrosive materials, quick drenching or flushing facilities must be provided in accordance with 29 CFR 1910.151(c) and 1926.50(g).
- Similarly, if workers face other hazards (such as splash or dust) that could harm the eyes or body, readily accessible wash stations are essential. These stations should be properly located and maintained, including regular water changes, as per the manufacturer's instructions.

### 3.8.3 Protection of Decontamination Line Assistants

Should work require a decontamination corridor the following shall be used as guidance. The SHSO/SS (or other designated person) must for ensuring that decontamination line assistants receive appropriate PPE and addressing their protection within the site-specific HASP. Key zones—namely, the Exclusion Zone (EZ), Contamination Reduction Zone (CRZ), and Clean Support Zone—must be clearly marked both in the field and within the site-specific HASP. The following should be considered when preparing for protection of decontamination line assistants.

1. Task Hazard Analysis: A thorough analysis must be conducted for line assistants to determine the optimal PPE level required. Factors influencing this decision include:
    - Expected or Visible Contamination on Responders
    - Type of Contaminant and associated respiratory and skin hazards
    - Total Vapor/Gas Concentrations in the CRZ
    - Presence of Particulates and specific inorganic or organic vapors in the CRZ
    - Results of Wipe Tests (refer to Section 5.4)
    - Slope and Configuration of the CRZ
2. PPE Levels: Depending on the situation:
    - Line assistants may need to wear the same PPE level as workers in the EZ.
    - Alternatively, line assistants may be adequately protected by using PPE one level lower (e.g., wearing Level C protection while decontaminating workers who are wearing Level B)."

### 3.8.4 Procedures to Minimize PPE Contamination

Proper work practices and procedures should be implemented at sites to minimize the potential for contact with chemicals of concern while onsite. The following guidance should be considered when developing site-specific procedures:

- Opt for single-use Personal Protective Equipment (PPE) when feasible to minimize decontamination needs.
- Ensure complete sealing of PPE closures and interfaces, including pockets, zippers, and other potential openings.
- Position gloves and boots underneath the sleeves and pant legs of outerwear to reduce exposure.
- Place hoods outside the collar if they are not already attached.
- Utilize disposable outerwear and equipment when suitable to facilitate easy disposal and reduce decontamination efforts.
- Secure all junctions with tape to block contaminants from entering gloves, boots, jackets, and suits.

- Adopt work practices that limit contact with hazardous substances, such as avoiding unnecessary exposure.
- Employ remote techniques for sampling, handling, and opening containers, like using drum grapples and pneumatic impact wrenches.
- Store monitoring and sampling devices in bags designed with openings for sample ports and sensors.
- Cover contaminant sources with plastic sheeting or overpacks to shield PPE from contamination.

### 3.8.5 PPE Disposal

The PM, in coordination with the field team, shall ensure the safe disposal of all wastes generated from PPE use. PPE must be placed in appropriate containers, labeled, and prepared for disposal separately from other forms of waste. Refer to Roux's General Waste Corporate Management Program for further guidance.

# APPENDIX A
# EPA's Emergency Responder Health and Safety Manual

# APPENDIX E

Subsurface Utility Clearance Management Program

# SUBSURFACE UTILITY CLEARANCE MANAGEMENT PROGRAM

| | | |
|---|---|---|
| **CORPORATE HEALTH AND SAFETY MANAGER** | **:** | **Brian Hobbs, CIH, CSP** |
| **EFFECTIVE DATE** | **:** | **01/19** |
| **REVISION NUMBER** | **:** | **2** |

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## APPENDICES

Appendix A – Definitions

Appendix B – Example of Completed One Call

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## 1. PURPOSE

Roux Associates, Inc. and its affiliated companies, Roux Environmental Engineering and Geology, D.P.C, and Remedial Engineering (collectively, "Roux") has instituted the following program for completing proper utility mark-outs and for conducting subsurface clearance activities. This establishes a method to ensure, to the greatest extent possible, that utilities have been identified and contact and/or damage to underground utilities and other subsurface structures will be avoided.

## 2. SCOPE AND APPLICABILITY

The Subsurface Utility Clearance Management Program applies to all Roux employees, its contractors and subcontractors. Employees are expected to follow this program for all intrusive work involving Roux or other personnel (e.g., contractors/subcontractors) working for Roux unless the client's requirements are more stringent. Deviation from the program regardless of the specific work activity or work location must be pre-approved based on client's site knowledge, site experience and client's willingness for the use of this program. Any and all exceptions shall be documented and pre-approved by the Project Principal and the Office Manager.

## 3. PROCEDURES

### 3.1 Before Intrusive Activities

During the project kick-off meeting for intrusive activities the PM will review the Roux Subsurface Utility Clearance Checklist and Utility Verification (Appendix C) / Site Walkthrough Record (Appendix D) and the below bullet points with the project field team:

(Please note that these are intended as general reminders only and should not be solely relied upon.)

- Ensure the Mark-out / Stake-out Request Information Sheet (or one-call report) is complete and accurate for the site including address and cross streets and review for missing utilities. (Note: utility mark-out organizations do not have contracts with all utilities and it is often necessary to contact certain utilities separately such as the local water and sewer authorities).
- Have written confirmation prior to mobilizing to the site that the firm or Roux personnel performing the intrusive activity has correctly completed the mark-out notification process including requesting mark-outs, waiting for mark-outs to be applied to ground surfaces at the site, and receiving written confirmation of findings (via fax or email) from utility operators for all known or suspected utilities in the proposed area of intrusive activity, and provided utility owner written confirmation to Roux personnel for review and project files documentation.
- Do not begin any intrusive activity until all utilities mark-out has been completed (i.e., did all utilities mark-out the site?) and any unresolved mark-out issues are finalized. Perform a site walk to review the existing utilities and determine if said utilities have been located by the utility locators.

  (Note: The Tolerance Zone is defined as two feet plus half of the diameter or half of the greatest dimension (for elliptical sewers, duct banks and other non-cylindrical utilities) of a utility and two feet from the outside edge of any subsurface structure.)
- Install Pre-Clearance exploratory test holes (e.g., hand-dug test holes or other soft digging techniques) for the first 5-ft below land surface (BLS) at each location prior to conducting mechanized intrusive activities. The size of the pre-clearance exploratory test hole should be at a minimum twice the diameter of any downhole tool or boring device. (Note: Pre-Clearance exploratory test holes should be defined in the SOW/proposal provided to the client to prevent project delays and to allow adequate time for PM and PP to evaluate alternative approaches for the project. Alternative approaches will need to be pre-approved by the OM.

- For excavations, all utilities need to be marked and then exposed by hand following the protocols in this program. Pre-clearing for excavations may be performed by the "moat" technique (i.e., soft digging around the perimeter). In these cases, dig in small lifts (<12" for first 5 feet) using a dedicated spotter.) For Tolerance Zone work, unless otherwise agreed upon with the Utility Operator, work within the tolerance zone requires verification by means of hand-dug test holes performed to expose the utility. Once structures have been verified a minimum clearance of two feet must be maintained between the utility and any powered equipment.
- In addition, the following activities should be conducted:
  - Review the work scope to be performed with the site owner/tenant to determine if it may impact any utilities;
  - Attempt to procure any utility maps or historic drawings of subsurface conditions of the site;
  - **Determine the need for utility owner companies to be contacted or to have their representatives on site**;
  - Where mark-outs terminate at the property boundary, consider the use of private utility locating / GPR / geophysical-type services which may be helpful in locating utilities. Use of private utility locating firms, however, does not eliminate the legal requirement for the Excavator firm to submit a request for Public Utility Mark-outs. Also, the information provided by the service may be inaccurate and unable to locate subsurface utilities and structures in urban areas, landfills, urban fill areas and below reinforced slabs, etc. They should not be relied upon as the only means of performing utility clearance;
  - Documented description of the dig site which is included in the projects Health and Safety Plan (HASP) and one call report will be maintained in the field and distributed amongst Roux personnel its contractors and subcontractors; and
  - Documentation of the actual placement of mark outs in the field shall be collected using dated pictures, videos and/or sketches with distance from markings to fixed objects. All documentation shall be maintained within the project file.

### 3.2 During Intrusive Activities

The PM, field team lead or personnel performing oversight is to:

- Ensure the mark-out remains valid. (In certain states there are limits regarding the duration of time after the mark-out was applied to the ground surface work can be started or interrupted.) Additionally, the mark-outs must be maintained, documented, and in many cases refreshed periodically to be considered valid, this will be accomplished through calls to the one call center.
- Ensure intrusive activities are only performed within the safe boundaries of the mark-out as detailed in the One-Call Report.
- Halt all work if intrusive activities have resulted in discovery of an unmarked utility. Roux personnel shall notify the facility owner/operator and the one call center. All incidents such as this will be reported as per Roux Incident Investigation and Reporting Management Program.
- Halt all work if intrusive activities must take place outside of the safe boundaries of a mark-out and only proceed after new mark-outs are performed.
- Halt the intrusive activities and immediately consult with the PP if an unmarked utility is encountered.
- Completing any subsurface utility clearance incident reports that are necessary.

- If a utility cannot be found as marked Roux personnel shall notify the facility owner/operator directly or through the one call center. Following notification, the excavation may continue, unless otherwise specified in state law.
- Contractors/subcontractors must contact the one-call center to refresh the ticket when the excavation continues past the life of the ticket. Ticket life shall be dictated by state law however at a maximum ticket life shall not exceed 20 working days.

### 3.3 Stop Work Authority

Each Roux employee has Stop Work Authority which he or she will execute upon determination of any imminent safety hazard, emergency situation, or other potentially dangerous situation, such as hazardous weather conditions. This Stop Work Authority includes subsurface clearance issues such as the adequacy of a mark-out or identification during intrusive operations of an unexpected underground utility. Authorization to proceed with work will be issued by the PM/PP after such action is reviewed and resolved. The PM will initiate and execute all management notifications and contact with emergency facilities and personnel when this action is appropriate.

## Appendix A - Definitions

| | |
|---|---|
| ***Intrusive Work Activities*** | All activities such as digging or scraping the surface, including but not limited to, excavation, test pitting or trenching, soil vapor sampling or the installation of soil borings, soil vapor monitoring points and wells, or monitoring wells, and drilling within the basement slab of a recently demolished building. |
| ***Mark-out / Stake Out*** | The process of contracting with a competent and qualified company to confirm the presence or absence of underground utilities and structures. This process will clearly mark-out and delineate utilities that are identified so that intrusive work activities can be performed without causing disturbance or damage to the subsurface utilities and structures. After utility mark-outs are completed the soft digging will be completed prior to intrusive work. |
| ***Tolerance Zone*** | Defined as two feet on either side of the designated centerline of an identified utility, plus half of the diameter or half of the greatest dimension (for elliptical sewers, duct backs and other non-cylindrical utilities) of that utility and two feet from the outside edge of any subsurface structure. |
| ***Structure*** | For the purpose of this program a structure is defined as any underground feature that may a present potential source(s) of energy such as, but not limited to, utility vaults, bunkers, piping, electrical boxes, wires, conduits, culverts, utility lines, underground tanks and ducts. |
| ***Soft Digging*** | The safest way to remove material from unknown obstructions or services is by using tools such as a vactor or air knife, non-mechanical tools, or hand tools. The methods are clean and non-evasive and used for uncovering and exposing buried services, excavating and for providing a quick method of soil removal from sensitive areas. |
| ***Verification*** | Exploratory test-hole dug with hand tools within the Tolerance Zone to expose and verify the location, type, size, direction-of-run and depth of a utility or subsurface structure. Vacuum excavation (soft dig) methods can further facilitate exposure of a subsurface utility and accurately provide its location and identification prior to intrusive work approaching the Tolerance Zone. |

**Appendix B - Example of Completed One Call Report**

***Example Completed One-Call Report***

New York 811

Send To: C_EMAIL Seq No: 744

Ticket No: 133451007 ROUTINE

Start Date: 12/16/13 Time: 7:00 AM Lead Time: 20

State: NY County: QUEENS Place: QUEENS
Dig Street: 46TH AVE Address:
Nearest Intersecting Street: VERNON BLVD
Second Intersecting Street: 11TH ST

Type of Work: SOIL BORINGS
Type of Equipment: GEOPROBE
Work Being Done For: ROUX

In Street: X On Sidewalk: X Private Property: Other:
On Property Location if Private: Front: Rear: Side:

Location of Work: MARK THE ENTIRE NORTH SIDE OF THE STREET AND SIDEWALK OF: 46TH AVE BETWEEN VERNON BLVD AND 11TH STREET

Remarks:

Nad: Lat: Lon: Zone:
ExCoord NW Lat: 40.7475399 Lon: -73.9534811 SE Lat: 40.7457406 Lon: -73.9493680

Company : ZEBRA ENVIROMENTAL Best Time: 6AM-5PM
Contact Name: DAVID VINES Phone: (516)596-6300
Field Contact: DAVID VINES Phone: (516)596-6300
Caller Address: 30 N PROSPECT AVE Fax Phone: (516)596-4422
LYNBROOK, NY 11563
Email Address: david@zebraenv.com

Additional Operators Notified:
ATTNY01 AT&T CORPORATION (903)753-3145
CEQ CONSOLIDATED EDISON CO. OF N.Y (800)778-9140
MCINY01 MCI (800)289-3427
PANYNJ01 PORT AUTHORITY OF NY & NJ (201)595-4841
VZQ VERIZON COMMUNICATIONS (516)297-1602

Link to Map for C_EMAIL: http://ny.itic.occinc.com/XGMZ-DF2-L23-YAY

Original Call Date: 12/11/13 Time: 1:15 PM Op: webusr
IMPORTANT NOTE: YOU MUST CONTACT ANY OTHER UTILITIES DIRECTLY

## Appendix C - Roux Subsurface Utility Clearance Checklist

***Roux Subsurface Utility Clearance Checklist*** ***Date of Revision – 12/3/14***

**Work site set-up and work execution**

| ACTIVITY | Yes | No | N/A | COMMENTS INCLUDING JUSTIFICATION IF RESPONSE IS NO OR NOT APPLICABLE |
|---|---|---|---|---|
| Daily site safety meeting conducted, SPSAs performed, JSAs reviewed, appropriate work permits obtained. | | | | |
| HASP is available and reviewed by site workers / visitors. | | | | |
| Subsurface Utility Clearance Procedure has been reviewed with all site workers. | | | | |
| Work area secured; traffic control established as needed. Emergency shut-off switch located. Fire extinguishers / other safety equipment available as needed. | | | | |
| Utility mark-outs (public / private) clear and visible. Provide Excavator's Stake-Out Reference Number / Request Date / Time. | | | | |
| Tolerance zone work identified. | | | | |
| Work execution plan reviewed and adhered to (ground disturbance methods, clearance depths, any special utility protection requirements, or any other execution requirements; especially for Tolerance Zone work). | | | | |
| Verbal endorsement received from Roux PM for any required field deviations to work execution plan. | | | | |

Key reminders for execution:

The Subsurface Utility Clearance Protocol should be referenced to determine all requirements while executing subsurface work. The bullet points below are intended as general reminders only and should not be solely relied upon.

- Tolerance zone is defined as two feet plus half of the diameter or half of the greatest dimension (for elliptical sewers, duct banks and other non-cylindrical utilities) of a utility and two feet from the outside of any subsurface structure.
- Install Pre-Clearance exploratory test holes (e.g., hand-dug test holes or vacuum excavation) must be performed for the first five feet below land surface (BLS) at each location prior to conducting mechanized intrusive activities. The size of the pre-clearance exploratory test hole should be at a minimum twice the diameter of any downhole tool or boring device. (Note: Pre-clearance exploratory test holes should be defined in the SOW/proposal provided to the client to prevent project delays and to allow adequate time for PM and PP to evaluate alternative approaches for the project. Alternate approaches will need to be pre-approved by the OM.
- For excavations, all utilities need to be marked and then exposed by hand following the protocols in this program. Pre-clearing for excavations may be performed by the "moat" technique (i.e., soft

digging around the perimeter). In these cases, dig in small lifts (<12" for first five feet) using a dedicated spotter.) For Tolerance Zone work, unless otherwise agreed upon with the Utility Operator, work within the tolerance zone requires verification by means of hand-dug test holes to expose the utility. Once structures have been verified a minimum clearance of two feet must be maintained between the utility and any powered equipment.

**Appendix D - Utility Verification/Site Walkthrough Record**

**Employee Name:** _____

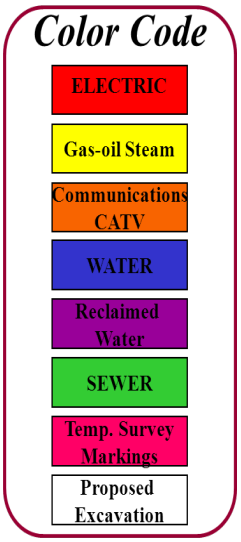
**Date:** _____

**Instructions:** For each utility suspected at the job site, indicate location on the job site, approximate burial depth, and means of detecting the utility. Leave blank if that utility is not believed to be present.

| Utility | Description of Utility Location Identified Onsite | Approx. Depth (bls) | Method / Instrumentation used to determine Utility Location | Utility Owner Response (Date/Time) | Mark Out Indicates (Clear / Conflict) |
|---|---|---|---|---|---|
| **Electrical Lines** | | | | | |
| **Gas Lines** | | | | | |
| **Pipelines** | | | | | |
| **Steam Lines** | | | | | |
| **Water Lines** | | | | | |
| **Sanitary and Stormwater Sewer lines** | | | | | |
| **Pressured Air-Lines** | | | | | |
| **Tank Vent Lines** | | | | | |
| **Fiber Optic Lines** | | | | | |
| **Underground Storage Tanks** | | | | | |
| **Phone Lines/ Other** | | | | | |

* bls - below land surface

Site Sketch Showing Utilities:




Other Comments / Findings:

Completed by: _____

Signature: _____ Date: _____

# APPENDIX F

## Heavy Equipment Exclusion Zone Management Program

# HEAVY EQUIPMENT EXCLUSION ZONE MANAGEMENT PROGRAM

| | | |
|---|---|---|
| **CORPORATE HEALTH AND SAFETY MANAGER** | : | **Brian Hobbs, CIH, CSP** |
| **EFFECTIVE DATE** | : | **01/2019** |
| **REVISION NUMBER** | : | **1** |

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## 1. PURPOSE

The purpose of the Exclusion Zone Management Program is to establish the minimum clearance distance that must be maintained between workers and heavy equipment while equipment is in operation (i.e., engaged or moving). The intent is to have no personnel or equipment entering the Exclusion Zone while the equipment is in operation or moving to ensure that Roux and Subcontractor employees are not unnecessarily exposed to the hazards of the equipment.

## 2. SCOPE AND APPLICABILITY

This Management Program applies to all Roux Associates, Inc. and its affiliated companies, Roux Environmental Engineering and Geology, D.P.C, and Remedial Engineering (collectively, "Roux") employees and their subcontractors who are performing field work and are potentially exposed to heavy equipment. For the purpose of this program, heavy equipment includes, but is not necessarily limited to: excavation equipment, drill rigs, vacuum trucks, forklifts, lull telehandlers, man lifts, bobcats, delivery trucks, etc.

## 3. PROCEDURES

As specified in the following sections of this Program, an Exclusion Zones must be established and maintained during activities involving the movement/operation of heavy equipment. The Exclusion Zone requirements apply to all personnel on the site but are primarily focused on those personnel who are required to be working in the vicinity of the equipment. The exclusion zone is in effect when heavy equipment is moving or engaged (ex. movement of an arm or bucket of an excavator, rotation of an auger, lifting of a load with a forklift, raising/lowering of a man lift, etc.).

1. The Exclusion Zone must meet the following minimum requirements:
   - A minimum distance of 10 feet from all heavy equipment and loads being moved by the equipment;
   - Greater than the swing/reach radius of any moving part on the heavy equipment (i.e., for large equipment this may mean an exclusion zone distance larger than 20 feet);
   - Greater than the tip-over distance of the heavy equipment; and
   - Greater than the radius of blind spots.

   The size of the Exclusion Zone will need to be determined on a task-specific basis considering the size of the heavy equipment in use and the task being performed. Prior to all heavy equipment operations, the Exclusion Zone(s) distance must be specifically identified in the Job Safety Analysis (JSA).
2. The spotter (or another individual) should be assigned responsibility for enforcing the Exclusion Zone. The spotter should be positioned immediately outside of the Exclusion Zone within a clear line of sight of the equipment operator. The spotter must signal the operator to stop work if anyone or anything has the potential to enter or compromise the Exclusion Zone. The operator should stop work if the spotter is not within his/her line of sight. If multiple pieces of equipment are being used, each piece of equipment must have its own Exclusion Zone and spotter. For large excavation and demolition projects the spotter should be in constant radio contact (not cell phone) with the machine driver.
3. If an individual must enter the Exclusion Zone, the designated Spotter must signal the Equipment Operator to stop the equipment. Once the equipment is no longer moving (ex. movement of an arm of an excavator is STOPPED, lifting of a load with a forklift STOPPED, raising/lowering of a man lift is

STOPPED, etc.), the operator must DISENGAGE THE CONTROLS and STOP and SIGNAL BY "SHOWING HIS HANDS". This signal will indicate that it is safe for the personnel to enter the limits of the Exclusion Zone to perform the required activity. The equipment must remain completely stopped/disengaged until all personnel have exited the limits of the Exclusion Zone and the designated Spotter has signaled by "SHOWING HIS HANDS" to the Equipment Operator that it is safe to resume operations.

4. When entering the limits of the Exclusion Zone, personnel must at a minimum:
   - Establish eye contact with the operator and approach the heavy equipment in a manner that is in direct line of sight to the Equipment Operator;
   - Never walk under any suspended loads or raised booms/arms of the heavy equipment; and
   - Identify a travel path that is free of Slip/Trip/Fall hazards.
5. The Exclusion Zone should be delineated using cones with orange snow fence or solid poles between the cones, barrels, tape or other measures. For work in rights-of-way rigid barriers, such as Jersey barriers or temporary chain link fence should be used. For certain types of wide-spread or moving/mobile equipment operations, such delineation may not be practicable around pieces of equipment or individual work areas. In such instances, it is expected that the entire operation will be within a larger secure work area or that additional means will be utilized to ensure security of the work zone.

All subcontractors who provide heavy equipment operations to field projects must implement a program that meets or exceeds the expectations described above as well as any additional requirements that may be required on a client or site-specific basis.

### 3.1 Exceptions

It is recognized that certain heavy equipment activities may require personnel to work within the limits of the Exclusion Zone as specified in this program. Such activities may include certain excavation clearance tasks, drill crew activities or construction tasks. However, any such activity must be pre-planned with emphasis on limiting the amount and potential exposure of any activity required within the zone. The critical safety steps to mitigate the hazards associated with working within the Exclusion Zone must be defined in the JSA and potentially other project-specific plans (i.e., critical lift plans, etc.), and approved by the Roux Project Principal and client representative, if required, prior to implementation.

## 4. TRAINING

Many Roux projects have different requirements that are client-specific or site-specific in nature. It is the responsibility of the Project Principal (or Project Manager if delegated this responsibility by the Project Principal) to ensure that the workers assigned to his/her projects are provided orientation and training with respect to these client and/or site-specific requirements.

# APPENDIX D

Community Air Monitoring Plan

# Community Air Monitoring Plan (CAMP)

2030 Fulton Street
Brooklyn, New York

November 7, 2025

Prepared for:

**Steps Family LLC**
**c/o Riseboro Community Partnership**
217 Wyckoff Avenue
Brooklyn, New York 11237

Prepared by:

**Roux Environmental Engineering and Geology, D.P.C.**
209 Shafter Street
Islandia, New York 11749

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# Table

1. Action Limit Summary for VOCs and Particulates

# Appendix

A. Action Limit Report

# 1. Introduction

Roux Environmental Engineering and Geology, D.P.C. (Roux ) on behalf of Steps Family LLC (Steps Family) has developed a project-specific Community Air Monitoring Plan (CAMP) to implement real time monitoring at 2030 Fulton Street, Fulton Street, Brooklyn, New York (Site) during Site development and remedial activities. The monitoring program will be implemented at all times during soil disturbance activities. Intrusive activities at the Site will include remedial investigation activities (e.g., soil borings, groundwater sampling, soil vapor sampling) and future remediation/redevelopment activities (e.g., drilling of foundation piles, excavation, soil disposal, installation of Site cover materials, as required). The CAMP is designed to provide protection for the downwind community and on-Site workers not directly involved with the subject work activities from potential airborne releases as a direct result of soil disturbance activities. This plan was prepared in accordance with the New York State Department of Environmental Conservation (NYSDEC) Division of Environmental Remediation DER-10, Appendix 1A (New York State Department of Health Generic Community Air Monitoring Plan).

The specifics of the CAMP are presented in the following five (5) sections:

- 2.0 VOC Monitoring Approach
- 3.0 Particulate Monitoring Approach
- 4.0 Meteorological Monitoring Approach
- 5.0 Available Suppression Techniques
- 6.0 Reporting

# 2. VOC Monitoring Approach

Volatile organic compounds (VOC)s are present in soil and soil vapor at the Site; therefore, VOCs will be monitored during all intrusive activities. Total VOC concentrations in air will be monitored continuously at one (1) location downwind of all soil disturbance activities. In addition, one (1) upwind monitoring station will be set up upgradient of the wind direction at the Site perimeter, to record the background concentration of VOCs in ambient air at the Site that are unrelated to soil disturbance activities (i.e., stemming from off-Site). The difference in VOC concentration between the upwind and downwind monitoring stations (net concentration) will be used to determine the VOC concentration produced by soil disturbance activities at the Site. The VOC monitoring equipment will be located at temporary monitoring stations that will be established daily, based upon on-Site logistics, weather conditions, and individual work areas. The monitoring work will be conducted using MiniRAE™ 3000 (or equivalent) portable VOC monitors, or similar type monitors. The equipment will be calibrated at least once daily before use, using isobutylene as the calibration gas. Each monitoring station is equipped with an audible alarm to indicate exceedance of the action levels (as defined below and summarized in Table 1).

The equipment is capable of calculating 15-minute running average concentrations, which will be compared to the levels specified below.

- If the net concentration of total VOCs at the Site exceeds 5 parts per million (ppm) for the 15-minute average, work activities must be temporarily halted and monitoring continued. If the net VOC concentration readily decreases (per instantaneous readings) below 5 ppm work activities can resume with continued monitoring.
- If the net concentration of total VOCs at the downwind perimeter of the Site persists at concentrations in excess of 5 ppm but less than 25 ppm, work activities must be halted, the source of VOCs identified, suppression techniques employed to abate emissions, and monitoring continued. After these steps, work activities can resume if the net VOC concentration at the Site is below 5 ppm for the 15-minute average. If net concentrations are in excess of 25 ppm, the contributing soil disturbance activities will be halted, the source further evaluated, and the activity modified until monitoring indicates that net VOC concentrations at the Site perimeter are below 5 ppm. Once net VOC concentrations are below 5 ppm at the Site perimeter, work will resume with continued monitoring.

All 15-minute running average readings will be recorded and made available for the New York City Office of Environmental Remediation (NYC OER), NYSDEC and the New York State Department of Health (NYSDOH) personnel (if requested) to review. Instantaneous readings and visual observations, if any, used for decision purposes (i.e., implementing VOCs suppression, halting work, or continuing work) will be recorded. If an exceedance of the action level occurs, an Action Limit Report (ALR) will be completed, identifying the monitoring device location, the measured VOC level, the activity causing the exceedance, meteorological conditions, and the corrective actions taken. A blank ALR form is provided in Appendix A. All documentation will be kept on file at the Site.

# 3. Particulate Monitoring Approach

Particulate concentrations will be monitored continuously at temporary particulate monitoring stations set up at upwind and downwind locations during all soil disturbance activities. The particulate monitoring will be performed using real-time monitoring equipment capable of measuring particulate matter less than 10 micrometers in size (PM-10) and capable of averaging readings over a period of 15 minutes (or less) for comparison to the airborne particulate action levels (as defined below and summarized in Table 1). The monitoring equipment used will be the DustTrak II Aerosol 8530 Monitors™ or equivalent. One (1) upwind and one (1) downwind monitor will be deployed each day, equipped with an omni-directional sampling inlet and a PM-10 sample head. The upwind monitoring station will be used to record the background concentration of particulates in ambient air at the Site, which are unrelated to soil disturbance activities at the Site (i.e., stemming from off-Site). The net concentration will be used to determine the particulate concentration produced by soil disturbance activities at the Site. The data logging averaging period will be set to 15-minutes with time and date stamp recording. Alarm averaging will be set at 90 micrograms per cubic meter (µg/m3) per 15-minute period. This setting will allow proactive evaluation of Site conditions prior to reaching Action Levels of 100 µg/m3 above net concentrations. The equipment will be outfitted with an audible alarm to indicate exceedance of the action level. In addition, fugitive dust migration will be visually assessed during all work activities. The monitoring will be used to compare values to the following:

- If the net PM-10 particulate concentration is 100 µg/$m^3$ for the 15-minute period or if airborne dust is observed leaving the Site, then dust suppression techniques must be employed. Work may continue with dust suppression techniques provided that the net PM-10 particulate concentrations do not exceed 150 µg/$m^3$ and provided that no visible dust is migrating from the Site.
- If, after implementation of dust suppression techniques, the net PM-10 particulate concentrations are greater than 150 µg/$m^3$, work must be stopped, a re-evaluation of activities initiated, and dust suppression techniques modified. Work can resume provided that dust suppression measures and other controls are successful in reducing the net PM-10 particulate concentration to within 150 µg/$m^3$ and in preventing visible dust migration.

All 15-minute average readings will be recorded and be available for NYC OER, NYSDEC, and NYSDOH personnel to review. Instantaneous readings and visual observations, if any, used for decision purposes (i.e., implementing dust suppression, halting work, or continuing work) will be recorded. If an exceedance of the action level occurs, an ALR will be completed, identifying the monitoring device location, the measured particulate concentration, the activity causing the exceedance, meteorological conditions, and the corrective actions taken, as provided in Appendix A. All documentation will be kept on file at the Site.

# 4. Meteorological Monitoring Approach

Meteorological data consisting of wind speed, wind direction, temperature, barometric pressure, and relative humidity will be collected. At a minimum, a full set of meteorological parameters will be measured and recorded at the start of each workday, noon of each workday, and the end of each workday. Wind direction readings will be utilized to position the VOC and particulate monitoring equipment in appropriate upwind and downwind locations. A Davis Corporation wireless instrument station or equivalent will be used to measure and log the meteorological monitoring data.

# 5. Available Suppression Techniques

Odor Control

Due to the nature of the project, there is a potential for generation of nuisance odors and the need for odor control. If nuisance odors are identified, work will be halted and the source of odors will be identified and corrected. Work will not resume until all nuisance odors have been abated. NYC OER, NYSDEC and NYSDOH will be notified of all odor events and of all other complaints about the project.

All necessary means will be employed to prevent off-Site nuisances. At a minimum, procedures will include: (a) limiting the area of open excavations; (b) shrouding open excavations with tarps and other covers; and (c) using odor control solutions/foams to cover exposed odorous soils. If odors develop and cannot be otherwise controlled, additional means to eliminate odor nuisances will include: (d) use of chemical odorants in spray or misting systems; and, (e) use of staff to monitor odors in surrounding neighborhoods.

Dust Control

Due to the nature of the project, dust control will be necessary. Dust suppression will be achieved through the use of water for wetting excavation areas. Water will be available on-Site at suitable supply and pressure for use in dust control. Roadways and sidewalks adjacent to the Site entrances/exits will be swept as needed.

# 6. Reporting

All recorded monitoring data will be downloaded, and field logged periodically, including ALRs (if any) and Daily CAMP Monitoring Location Plans. All records will be maintained on-Site and available for NYC OER/NYSDOH review. A summary of CAMP findings, including excursions, will be provided in Weekly Reports. If an ALR is generated due to VOC or particulate exceedances, the NYC OER, NYSDEC, and NYSDOH will be notified within 24 hours of the exceedance.

# TABLES

1. Action Limit Summary for VOCs and Particulates

## Table 1. Action Limit Summary for VOCs and Particulates, 2030 Fulton Street, Brooklyn, NY

| Contaminant | Downwind Action Levels* | Action/Response |
|---|---|---|
| Volatile Organic Compounds (VOCs) (Monitoring Via Photoionization Detector and Odor Observation) | < 5 ppm | 1. Resume work with continuing monitoring. |
| | 5 ppm < level < 25 ppm | 1. Work activities must be temporarily halted, source vapors must be identified, suppression techniques employed to abate emissions and monitoring continued.<br>2. After these steps, if VOC levels (200 feet downwind of the exclusion zone or half the distance to the nearest potential receptor or structure, whichever is less) is below 5 ppm over background, resume work. |
| | > 25 ppm | 1. Identified contributing ground intrusive activities must be halted and vapor suppression techniques must be evaluated and modified until monitoring indicates VOC levels below the action level.<br>2. After these steps, if VOC levels (half the distance to the nearest potential receptor or structure) are below 5 ppm over background, resume work. |
| Particulates (Monitoring Via Particulate Meter and Observation) | < 100 ug/$m^3$ | 1. If dust is observed leaving the work area, then dust control techniques must be implemented or additional controls used. |
| | 100 ug/m3 < level < 150 ug/$m^3$ | 1. Employ dust suppression techniques.<br>2. Work may continue with dust suppression techniques provided that downwind PM-10 particulate concentration do not exceed 150 ug/$m^3$ above the upwind level and provided that no visible dust is migrating from the work area. |
| | > 150 ug/$m^3$ | 1. STOP work<br>2. Re-evaluate activities, modify dust suppression techniques. Work can resume provided that dust suppression measures and other controls are successful in reducing the downwind PM-10 particulate concentration to within 150 ug/$m^3$ of the upwind level and in preventing visible dust migration. |

* The net concentration of VOCs or particulates above background concentrations. Background concentrations are recorded at the upwind monitoring station. The net concentration is the difference between the upwind monitoring station and the downwind monitoring station concentration. Particulate readings are based on the respirable (PM-10) fraction.

# APPENDIX A

Action Limit Report

# ACTION LIMIT REPORT

Project Location: **2030 Fulton Street, Brooklyn, NY**

Date: _____ Time: _____

Name: _____

Contaminant: PM-10: _____ VOC: _____

Wind Speed: _____ Wind Direction: _____

Temperature: _____ Barometric Pressure: _____

DOWNWIND DATA

Monitor ID #: _____ Location: _____ Level Reported: _____

UPWIND DATA

Monitor ID#: _____ Location: _____ Level Reported: _____

BACKGROUND CORRECTED (NET) CONCENTRATION

Monitor ID#: _____ Location: _____ Level Reported: _____

ACTIVITY DESCRIPTION

CORRECTIVE ACTION TAKEN