Remedial Investigation Work Plan (RIWP)

11-24 Wyckoff Avenue
Queens, New York 11385

April 10, 2026

Prepared for:

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Certification

I, Noelle Clarke, certify that I am currently a NYS registered professional engineer and that this Remedial Investigation Work Plan for the 11-24 Wyckoff Ave Site was prepared in accordance with all applicable statutes and regulations and in substantial conformance with the DER-10 Technical Guidance for Site Investigation and Remediation, dated May 2010 (DER-10).

Noelle Clarke, P.E.	April 10, 2026	
NYS Professional Engineer #072491	Date	Signature

1. Introduction

Roux Environmental Engineering and Geology, D.P.C. (Roux), on behalf of 1124 Wyckoff LLC (Volunteer), has prepared this Remedial Investigation Work Plan (RIWP) for the property located at 11-24 Wyckoff Avenue, Ridgewood, Queens, New York (Site), as shown on Figure 1. The Site is approximately 0.34 acres and is identified as Block 3542 and Lot 50 on the Queens Tax Map (Figure 2). The Volunteer is the current owner of the Site.

The Site is presently developed with a one story industrial/manufacturing building, which is "U" shaped and has an asphalt paved parking area located in the center. The building contains two partial basements that are situated in the southwestern and southeastern corners of the building. The existing building is currently used as the design studio, manufacturing facility, warehouse and office for the Volunteer's New York City-based fashion company. The proposed redevelopment plan includes renovating/updating the existing building and is anticipated to include a roof-top addition of between 1,500 and 2,000 square feet and required modifications to the foundation in that area. Industrial use, as defined in 6 New York Codes, Rules, and Regulations (NYCRR) Part 375-6 Environmental Remediation Programs, is proposed for the Site.

This RIWP was originally submitted by the Volunteer concurrently with the New York State Department of Environmental Conservation (NYSDEC) Brownfield Cleanup Program (BCP) Application and has been updated in accordance with the NYSDEC comment letter dated December 4, 2025. On February 2, 2024, NYSDEC sent the Volunteer a Site Characterization Work Plan - Addendum (SC Work Plan Addendum) dated August 16, 2023 prepared by HRP Associates, Inc. and associated Soil Vapor & Groundwater Sampling letter/Property Access Voluntary Consent (Right of Entry) Form (NYSDEC Letter) prepared by NYSDEC on February 2, 2024. The NYSDEC Letter and SC Work Plan Addendum indicated that the Site is under consideration by NYSDEC for potential listing (P-Site) on the New York State Registry of Inactive Hazardous Waste Disposal Sites. The NYSDEC Work Plan detailed an environmental investigation including soil, groundwater, and soil vapor samples both on-Site and off-Site.

The Volunteer decided to pursue entry into the BCP and on August 8, 2024, NYSDEC, the Volunteer , the Volunteer's attorney, and Roux participated in a BCP Pre-application Meeting to discuss next steps at the Site. The Volunteer applied as a volunteer for the BCP and will conduct a remedial investigation (in lieu of HRP's SC) under the BCP and with approval of this RIWP. The Site was accepted into the BCP and the Brownfield Cleanup Agreement (BCA) was executed on August 11, 2025.

This RIWP has been prepared in accordance with NYSDEC procedures set forth in the document titled DER-10 Technical Guidance for Site Investigation and Remediation, dated May 2010 (DER-10), and complies with all applicable Federal, State, and local laws, regulations, and requirements. The purpose of the RI is to identify the nature and extent of the contamination at the Site to determine the appropriate remediation for the Site. The planned RI includes an investigation of soil, groundwater, and soil vapor; to further characterize environmental media at the Site; to qualitatively assess the potential exposure of receptors to Site contaminants; and to develop any other additional data necessary to support the development of a Remedial Action Work Plan (RAWP). The remainder of this RIWP is organized as follows:

Section 2: Site Background

Section 3: Objectives, Rationale, and Scope of Work

Section 4: Health and Safety Plan

Section 5: Quality Assurance/Quality Control Protocols
Section 6: Reporting and Project Schedule

2. Site 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 the previous environmental work conducted at the Site.

2.1 Site Description and Setting

A Site Location Map and Tax Map are included as Figures 1 and 2, respectively. A summary of the acreage and the property information is provided below.

Property Location	
Property Name:	11-24 Wyckoff Avenue
Property Address:	11-24 Wyckoff Avenue
Property Town, County, State:	Ridgewood, Queens New York
Property Tax Identification:	Block 3542 and Lot 50
Property Topographic Quadrangle:	Queens, New York
Nearest Intersection:	Wyckoff Avenue and Cody Avenue directly northeast, Wyckoff Avenue and Cooper Avenue to the southeast, and Wyckoff Avenue and Decatur Street to the northwest.
Area Description:	The Site is in a mixed-use area in the Ridgewood neighborhood of Queens, New York. To the north is a three-Story CubeSmart Self Storage building, single family residential homes, and the L-train Subway (below grade), to the south is a one-story industrial/manufacturing building, to the east is a lot used for parking and storage, and to the west are single family residential homes. A portion of the L-train Subway runs beneath the northeast portion of the building.

Property Information	
Property Size:	0.34-acres
Site Shape:	Rectangular (with U-shaped building)
Property Use:	The Site is used as a warehouse/manufacturing/office building for a fashion company. The building has an active sub-slab depressurization system (SSDS) with sub-slab monitoring points installed throughout the building, three in-line fans located on the roof, and risers to the roof with vacuum gauges/alarms installed in closed utility boxes/chases.
Number and Size of Buildings (Year Built):	One one-story U-shaped building, approximately 13,000 square feet (1902).
Basement/ Slab-on-Grade:	The building contains two partial basements that are situated in the southwestern and southeastern corners of the building. The remainder of the building is slab-on-grade.

2.1.1 Previous and Current Site Operations

Based on previous environmental reports and documentation, including historic Sanborn Fire Insurance Maps, the Site was developed prior to 1902 with multiple sheds in the western portion, a stable in the northern portion, and portions of a dwelling in the southeastern portion of the Site. In the 1914 Sanborn Map, the Site was developed with three auto houses in the western portion, a garage in the northern portion and portions of the same dwelling in the southeastern portion. In the 1936 Sanborn Map, the Site appeared similar to the previous map, except that the dwelling in the eastern portion is noted as auto laundry. The Site appeared generally the same in 1950 as it did on the 1936 Sanborn Map, except the auto laundry appears offsite and adjoining to the southeast. The following 1980 Sanborn Map depicts the Site as a knitting mill that remains until the most recent 2005 Sanborn Map. The existing building is currently used as the design studio, manufacturing facility, warehouse and office for the Volunteer's New York City-based fashion company. The proposed redevelopment plan includes renovating/updating the existing building and is anticipated to include a roof-top addition of between 1,500 and 2,000 square feet and required modifications to the foundation in that area.

A map showing an aerial view of the existing conditions is provided as Figure 3.

2.1.2 Neighboring Properties

The Site is bounded by the following properties, as summarized in the table below.

Adjacent Property Direction	Property Information
North	Three-story CubeSmart Self Storage, single family residential homes, and the L-train subway (below grade). A portion of the L-train subway passes beneath the northeast portion of the building.
South	One-story industrial/manufacturing building
East	Storage/parking
West	Single family residential homes

2.1.3 Topography/Hydrogeology

As noted in Roux's March 2023 Phase I ESA, the grade of the Site is generally flat and the average elevation of the Site is approximately 78 feet above mean sea level (ft amsl), as shown in the USGS 7.5 Minute Series Topographic Map included with the Phase I ESA. In the Phase II ESA completed by WCD Group (WCD) in December 2018, groundwater was observed at the Site at approximately 62 feet below land surface (ft bls). Groundwater flow direction is presumed to be generally towards the south in the direction of Jamaica Bay and the Atlantic Ocean. It is noted that the L-train Subway, subsurface conduits, and nearby dewatering projects may influence local groundwater flow direction.

Based on information presented in the Phase II ESA completed by WCD in December 2018, Site soil consists of historic fill (well graded sands with gravel and minimal building debris) to about 6 to 8 ft bls overlying native sand with larger gravel and cobble.

2.1.4 Wetland Areas and Surface Water Bodies

The Site is not located in state or nationally regulated wetlands and there are no surface water bodies present. The Site does not appear in the 100-year or 500-year flood zones according to online Federal Emergency Management Act (FEMA) maps.

2.2 Results of Previous Environmental Investigations

The following previous environmental reports were reviewed as part of this RIWP, and are included as Appendix A.

- Phase I Environmental Site Assessment (Phase I ESA) prepared by WCD dated July 16, 2018;
- Phase II ESA prepared by WCD dated September 5, 2018 and Phase II ESA prepared by WCD dated December 19, 2018;
- Tank Closure Report prepared by Gallagher Bassett Technical Services (GBTS) dated March 16, 2020;
- Sub-Slab Depressurization System Completion Report prepared by GBTS dated June 10, 2022;
- Phase I ESA prepared by Roux dated March 31, 2023;
- SSDS Monitoring and Soil Vapor Intrusion Investigation Letter, prepared by Roux, dated May 30, 2023; and
- Site Characterization Work Plan – Addendum dated August 16, 2023, prepared by HRP Associates, Inc. and associated Soil Vapor & Groundwater Sampling letter/Property Access Voluntary Consent (Right of Entry) Form prepared by New York State Department of Environmental Conservation (NYSDEC) dated February 2, 2024.

Phase I ESA: prepared by WCD dated July 16, 2018

The WCD July 2018 Phase I ESA identified two Recognized Environmental Conditions (RECs) in connection with the Site which included the following:

1. Potential releases from the active 2,000-gallon underground storage tank (UST); and
2. Former industrial and commercial uses of the Site and the adjoining properties.

There were no Controlled Recognized Environmental Conditions (CRECs) or Historical RECs (HRECs) identified in the WCD Phase I ESA. Based upon these findings, WCD recommended the performance of a Phase II ESA. The Phase I ESA did identify the potential for asbestos-containing material (ACM) and lead-based paint (LBP); however, it is noted that these building material hazards are outside the scope of a Phase I ESA.

Phase II ESA, prepared by WCD, dated September 5, 2018 and Phase II ESA ,prepared by WCD dated, December 19, 2018

Following completion of the Phase I ESA and identification of the RECs, a Phase II ESA was conducted at the Site by WCD in September 2018. As part of the Phase II, WCD advanced five soil borings and installed four temporary soil vapor implants. It is noted that groundwater was not investigated as part of the September Phase II ESA. The results of the sampling did not identify any soil contamination except for some Polycyclic Aromatic Hydrocarbon (PAH) detections below regulatory standards.

Soil vapor was identified to be impacted with chlorinated solvents including 1,1,1-trichloroethane (1,1,1-TCA), tetrachloroethylene (PCE), and trichloroethylene (TCE) at moderately elevated concentrations. The maximum detection of PCE was noted to be 2,700 µg/m3. Soil vapor was also identified to have detections of petroleum related compounds; however, these compounds were detected at concentrations much lower than those of the chlorinated solvents. Based upon the results of the Phase II, WCD identified that the UST did not appear to have leaked and no further investigation was recommended to that end. WCD recommended that the UST be removed or closed-in-place and appropriately registered with the NYSDEC.

A second Phase II was completed by WCD in December 2018 that expanded upon the scope of their first Phase II conducted in September 2018. This Phase II consisted of the installation and sampling of thirteen soil borings, four temporary soil vapor implants and a single permanent groundwater well. The results of this investigation identified both petroleum related and chlorinated solvents in the groundwater sample; with PCE identified at a concentration of 150 µg/L, which exceeds the NYSDEC standard of 5 µg/L. Trace concentrations of PCE and 1,1,1-TCA were also identified in the soil samples collected from approximately 5-feet below grade. The Phase II concluded that the compounds identified in soil were below NYSDEC Unrestricted Use Soil Cleanup Objectives (UUSCOs); however, their presence suggests onsite usage of these compounds and there is still potential a source is located in the soil that has not yet been identified.

Tank Closure Report prepared by Gallagher Bassett Technical Services (GBTS) dated March 16, 2020

A tank closure report completed by GBTS in March 2020 indicated that the UST at the Site was accessed from the surface, pumped and pressure washed clean, filled with a concrete slurry and closed-in-place. A total of eight endpoint soil samples were collected from around and underneath the tank through holes cut in the tank shell prior to abandonment. These samples did not show any concentrations of volatile organic compounds (VOCs) that would suggest a release and the report concluded that the abandoned UST is no longer considered an environmental concern for the Site. The sampling around the tank did identify concentrations of semi-volatile organic compounds (SVOCs), specifically PAHs, above regulatory standards; however, the report concluded that these compounds were likely associated with poor quality historic fill material placed at the Site for development purposes and not associated with historical Site uses.

Sub-Slab Depressurization System Completion Report prepared by GBTS dated June 10, 2022

Due to the elevated concentrations in soil vapor identified at the Site, WCD recommended the installation of a vapor barrier/SSDS to control the potential for soil vapor intrusion into the indoor air space at the Site. According to the SSDS Completion Report, GBTS broke up the entire ground floor slab to facilitate installation of both a vapor barrier system (VBS) and SSDS. The SSDS consists of three loops; Loops 1 and 2 serve the eastern and western building wings, respectively. Loop 3 services both of the basements as well as the slab-on-grade area between each wing. Permanent sub-slab monitoring points were installed to measure pressure beneath the slab. According to the report, pressure readings indicated that the SSDS was functioning normally with the exception of a single monitoring point (SSMP-5 – south central area) reading exactly -0.004 inches water column (IWC), which is considered the minimum value for demonstrating sub-slab depressurization.

Phase I ESA prepared by Roux dated March 31, 2023

Roux was retained by the Volunteer to complete a Phase I ESA for the Site in 2023. Based on the information gathered during the Phase I ESA process, Roux identified the following RECs:

- Presence of Chlorinated Solvents in Soil, Groundwater, and Soil Vapor and Historical Site Usage: Based on the compounds detected in soil, groundwater, and soil vapor in the Phase II ESAs completed by WCD, there is potential that chlorinated solvents were used at the Site historically as part of the knitting mill, textile, and auto laundry operations. There is still potential that there is a

source of chlorinated solvents in the soil that has not been identified. There is an active SSDS in place and operating at the Site, for mitigation of potential soil vapor intrusion. Therefore the identified contamination in soil, groundwater, and soil vapor and the operation of the SSDS is considered a REC.

- Surrounding Industrial and Manufacturing Usage: The Site was historically adjoined to the east with a gasoline filling station that was identified to have buried gasoline tanks. An additional gasoline filling station appeared to be located approximately 115-feet to the northwest in the 1936-1950 Sanborn Maps at 1102 Wyckoff Avenue. Additional industrial/ manufacturing uses were identified in close proximity to the Site to the west with potential to adversely impact upon the Site and include: auto repair/ duco paint spraying, brass casting, paper tube and cardboard manufacturing, wood box manufacturing, and soap manufacturing. These uses were generally identified before 1980 and the area to the west was noted as unspecified manufacturing after 1980 until the most recent 2006 Sanborn Map. The surrounding industrial/manufacturing uses have the potential to adversely impact upon the environmental quality of the Site and the surrounding area.

Roux did not identify any HRECs or CRECs in connection with the Site.

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

- Presence of Historic Fill Material: The Site was identified to have poor quality fill material with elevated concentrations of SVOCs characterized as PAHs from the samples collected in the vicinity and underneath the closed-in-pace UST. The presence of the fill material and SVOCs does not appear to be related to historical Site usage but historical developments and demolitions that may have imported fill materials from unknown origins. The presence of this material should be considered during any future soil disposal and redevelopment scenarios.
- Presence of an Abandoned 2,000-gallon UST: The Site currently has a closed-in-place UST in the southern portion of the building. The UST was investigated in a tank closure report prepared by GBTS in March 2020 and samples collected from underneath and around the tank did not identify any releases from the UST. The tank itself and interior contents (concrete) are still considered a BER as any future redevelopment that disturbs the tank area may require its removal and subsequent disposal in accordance with local, state and federal laws.

SSDS Monitoring and Soil Vapor Intrusion Investigation Letter, prepared by Roux, dated May 30, 2023

Roux was retained by the Volunteer to complete monitoring of the existing SSDS and to conduct a soil vapor intrusion investigation. Roux collected vacuum measurements from the existing soil vapor monitoring points and inspected SSDS piping to confirm that the SSDS was operating properly. Additionally, six soil vapor samples, six indoor air samples, and one outdoor air sample were collected. Results showed that petroleum related VOCs including benzene, toluene, ethylbenzene, and xylenes were detected at relatively low concentrations in all samples collected. Additionally, chlorinated VOCs (CVOCs) were also detected at relatively low concentrations in the soil vapor and indoor air samples. CVOCs detected in indoor air included carbon tetrachloride and PCE. CVOCs detected in soil vapor included TCE, PCE, and 1,1,1-TCA. Based on the available data and comparison to the guidance values in the New York State Department of Health (NYSDOH) Final Guidance for Evaluating Soil Vapor Intrusion in the State of New York, October 2006, Revised May 2017 and February 2024 (NYSDOH SVI Guidance), it was determined that the SSDS was working properly and appears to be adequately addressing the potential for soil vapor intrusion (SVI) into the building. Roux recommended continuing to run the SSDS and to conduct SSDS inspections/vacuum readings, as well as conducting annual soil vapor and indoor air sampling.

Site Characterization Work Plan – Addendum, dated August 16, 2023 prepared by HRP Associates, Inc. and associated Soil Vapor & Groundwater Sampling letter/Property Access Voluntary Consent (Right of Entry) Form prepared by NYSDEC dated February 2, 2024prepared by HRP Associates, Inc. (HRP) for NYSDEC dated August 16, 2023

On May 16, 2022, HRP was authorized to complete NYSDEC Work Assignment (WA) No. 35 (D009808-35) for Site Characterizations (SC) at P-Sites located in Region 2 (New York, New York). HRP prepared the Site Characterization Work Plan - Addendum dated August 16, 2023 for the Site (SC Work Plan Addendum). The SC Work Plan Addendum identifies the Site as P-Site No. 241255 and proposes the installation of 16 shallow and deep soil borings to characterize soils and fill material beneath the slab of the building and in the loading dock area and to determine if an onsite source of CVOC impacts exists. HRP also proposed five groundwater monitoring wells and five soil vapor points to be installed in the off-Site areas surrounding the Site to delineate CVOC impacts, determine if impacts may be related to an off-site source, and identify potential impacts to offsite receptors. The work plan noted that a portion of the L-train subway line runs beneath the northeastern portion of the Site and that the New York City Transit Authority (NYCTA) /Metropolitan Transportation Authority (MTA) would need to review the proposed scope of work and determine if it had the potential to impact their facilities. The NYSDEC letter requested that the application voluntarily provide NYSDEC and it's subcontractors access to perform the above-referenced SC work.

On August 8, 2024, NYSDEC, the Volunteer, the Volunteer's attorney, and Roux participated in a BCP Preapplication Meeting to discuss next steps at the Site. TThe Volunteer applied as a volunteer for the BCP and will conduct a remedial investigation (in lieu of HRP's SC) under the BCP and with approval of this RIWP. The Site was accepted into the BCP and the Brownfield Cleanup Agreement (BCA) was executed on August 11, 2025.

3. Objectives, Rationale, and Scope of Work

The RI scope of work described in the following sections has been developed to satisfy NYSDEC BCP requirements to delineate the nature and extent of contamination and allow for effective evaluation of multiple BCP cleanup track alternatives. Data collected during the RI will determine the basis for future Remedial Actions for the Site.

Standards, Criteria, and Guidance (SCGs) for soil at BCP Sites are the numerical soil cleanup objectives (SCOs) presented in 6 NYCRR Part 375-6.8(a) and (b). The SCOs are categorized into unrestricted and restricted use (residential, restricted-residential, commercial, or industrial) criteria, as well as criteria for protection of groundwater and ecological resources (which can also be satisfied by application of the unrestricted use criteria). 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. Based on the anticipated future use of the Site as a manufacturing/industrial building, the applicable standards are ISCOs and protection of groundwater SCOs (PGWSCOs), for evaluation of a Track 4 remedy. An evaluation of the Site data relative to the NYSDEC unrestricted use SCOs (UUSCOs) for a Track 1 Unrestricted Use remedy will also be presented in the Remedial Investigation Report (RIR) and the RAWP, as required by NYSDEC DER-10,

Although the groundwater beneath the Site is not used as a drinking water source, based upon the evaluation of the current groundwater data discussed herein, the NYSDEC AWQSGVs – TOGS 1.1.1. will be considered.

SVI investigation data will be evaluated in accordance with the NYSDOH SVI guidance.

3.1 Objectives

Based on the analytical results from the previously completed Phase II ESAs the Site, the primary contaminants of concern (COCs) in soil are SVOCs, the primary COCs in groundwater are VOCs, and the primary COCs in soil vapor are CVOCs. The objectives of this RIWP are to:

- Delineate the nature and extent of impacts to soil across the Site;
- Delineate the nature and extent of impacts to groundwater;
- Evaluate the current potential for SVI; and
- Develop a Conceptual Site Model for the Site based on the RI data;
- Complete an on-Site and off-Site qualitative human health exposure assessment (EA) by evaluating the existing potential exposure pathways for Site-related contaminants based on the reasonably anticipated future land use of the Site and surrounding areas.

The data collected as part of previous investigations and the scope of work to be completed during the RI will evaluate soil and groundwater quality, and the potential for SVI onsite and at the property boundaries (to the extent practical considering the L-train subway beneath the Site) to provide the basis for remedial action selection and to determine the potential for offsite impacts. Environmental data collected 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 RAWP.

3.2 Remedial Investigation Scope

The proposed scope of work was developed in an effort to sample areas of the Site that were not previously investigated during previous investigations so that, together with the previously collected soil data, the entire Site will be sufficiently characterized to support the development of the Site-wide RAWP.

To accomplish this, the RI will focus on the following:

- The performance of Site reconnaissance to confirm proposed sampling locations;
- The performance of a geophysical survey to identify any underground anomalies and clear boring locations of potential subsurface obstructions;
- The advancement of 13 soil borings and collection of 37 soil samples for laboratory analysis;
- Installation of four groundwater monitoring wells and collection of four groundwater samples from the newly installed permanent monitoring wells;
- Installation of five soil vapor points and collection of five soil vapor samples and five indoor air samples to evaluate the current potential for SVI in the building;
- The collection of survey data of the newly installed soil borings and monitoring and the performance of one synoptic round of groundwater level measurements for developing a groundwater elevation contour map; and
- The performance of a qualitative exposure assessment to identify exposure pathways and evaluate contaminant fate and transport.

The overall scope of each component of the RI is discussed in the following subsections. The proposed Remedial Investigation Sampling Locations are shown on Figure 6 of this RIWP and additional information including intervals to be sampled and sample rationale is provided in the Quality Assurance Project Plan/Field Sampling Plan (QAPP/FSP; Appendix B). Detailed field sampling procedures are also provided in the QAPP/FSP.

3.2.1 Site Reconnaissance and Geophysical Survey and NYCTA/MTA Filing

Roux has reviewed the previously completed environmental reports and has identified potential COCs, described in Section 3.1, which will be targeted with soil, groundwater, and SVI samples during the RI. Roux performed Site reconnaissance on August 23, 2024, to assess potential access restrictions at the proposed sampling locations which will be targeted during the RI. Roux will also perform Site reconnaissance prior to the implementation of the subsurface investigation. An inspection of the existing Site conditions will be conducted to determine final sampling locations based on actual field conditions. The current layout of the proposed sampling locations, as shown in Figure 6, was developed to provide coverage across the entire Site footprint, excluding the northeast portion of the Site due to the L-train subway below. Based on the Site reconnaissance and geophysical survey, the layout of proposed sampling locations may change.

Due to the L-train subway running beneath the northeastern portion of the Site, the NYCTA/MTA must review and provide a determination of no impact for subsurface activity prior to implementation of the proposed drilling. Roux submitted NYCTA/MTA-required drawings for the Site including: a Site Plan showing the NYCTA/MTA infrastructure and proposed soil boring locations within 200 linear feet of the infrastructure; a Cross-Section showing the angles of influence of the proposed soil borings to the NYCTA/MTA infrastructure; and NYCTA/MTA general requirement notes. MTA reviewed the drawings and required that borings and/or monitoring wells be located a minimum of 10 feet from the underground easement. This resulted in relocation

of a few borings that were closer to the easement, as shown on Figure 6. Roux received a NYCTA/MTA letter of no impact on October 30, 2025.

3.2.2 Soil Investigation

A total of 38 soil samples (eight of which will only be sampled for Per- and Polyfluoroalkyl Substances [PFAS]) are proposed to be collected for laboratory analysis from the 13 soil borings that will be advanced at the locations shown in Figure 6 as part of the RI. All soil boring locations will be pre-cleared to 5 ft bls using non-intrusive methods, such as vacuum clearance and hand tools, prior to advancement of soil borings to verify the absence of utilities and/or other subsurface features (i.e., obstructions and/or refusal). Following utility clearance and the geophysical survey, the soil borings will be advanced using a Geoprobe™ direct push drill rig. It should be noted that the depths of indoor soil borings RSB-1, RSB-2, RSB-3, and RSB-12 may be limited due to access constraints of the drill rig, particularly by the ceiling heights. The ceiling height inside the building significantly limits the options for drilling equipment (e.g., use of a sonic rig is not possible due to the mast height). Based on the August 23, 2024 Site reconnaissance, a compact Geoprobe drill rig (e.g., Model 6610DT) designed for limited access areas was considered the most feasible option to complete most of the borings within the building footprint (excluding those in the southern portion of the Site, which will be performed using a Geoprobe 420 portable drill rig). The Geoprobe 6610DT rig has direct-push and hollow stem auger capabilities. If field conditions are favorable, the compact Geoprobe drill rig has the ability to advance soil borings RSB-1, RSB-2, RSB-3, and RSB-12 to the target depth of approximately 70 ft bls and install the wells described in Section 3.2.3. However, unfavorable field conditions, such as drilling in urban fill and in consolidated formations (e.g., silt and clay), can limit the practical achievable depth. If unfavorable conditions for the Geoprobe are encountered inside the building, consideration will be given to switching to a sonic drill rig for the exterior boring/well in the courtyard.

Below is a table summarizing the soil borings samples rationale.

Sample Location ID	Target Depth	Region of Site	Rationale	Sample Depths	Analyses*
RSB-1, RSB-2, RSB-3, and RSB-12	70 ft bls	Delineating upgradient and downgradient soil	Surface soil samples will be collected to evaluate exposure pathways. Other samples will provide coverage across the Site and evaluate the potential presence of soil contamination	0-2 ft bls, most impacted (if encountered, if not from middle of boring), 60-62 ft bls (2 ft above groundwater)	Target Compound List (TCL)/NYSDEC Part 375 VOCs, SVOCs (including 1,4-Dioxane), pesticides/herbicides, polychlorinated biphenyls (PCBs), Target Analyte List (TAL)/NYSDEC Part 375 metals, PFAS (three samples only)

Sample Location ID	Target Depth	Region of Site	Rationale	Sample Depths	Analyses*
RSB-4 through RSB-11, and RSB-13	10 ft bls	Delineating upgradient and downgradient soil	Surface soil samples will be collected to evaluate exposure pathways. Other samples will provide coverage across the Site and evaluate the potential presence of soil contamination	0-2 ft bls, most impacted (if encountered, if not 8-10 ft bls)	TCL/NYSDEC Part 375 VOCs, SVOCs (including 1,4-Dioxane), pesticides/herbicides, PCBs, TAL/NYSDEC Part 375 metals, PFAS (five samples only)

*1,4-Dioxane and PFAS are considered Emerging Contaminants (ECs). PFAS are proposed for 25 percent (eight total) samples

Soil will be collected from the sample intervals and for the parameters summarized in the table above. During advancement of borings, the soil will be inspected for evidence of contamination (visual and olfactory) and field screened continuously for organic vapors using a PID with a 10.6 eV lamp. Soil lithology will be recorded according to the Unified Soils Classification System (USCS).

It is estimated that 38 soil samples will be collected for laboratory analysis. Two blind duplicate soil samples, matrix spike (MS) soil samples, matrix spike duplicate (MSD) soil samples, field blanks, and trip blanks will be collected for QA/QC purposes. Additional details regarding sampling methods are provided in the QAPP/FSP (Appendix B).

Soil cuttings will be returned to boring locations to the extent possible. Soil generated as part of the soil investigation, if any, will be drummed, labeled, sampled, and stored onsite pending disposal.

3.2.3 Groundwater Investigation

To characterize onsite groundwater flow and groundwater quality, four of the soil borings (RSB-1, RSB-2, RSB-3, and RSB-12) will be converted into new groundwater monitoring wells (RMW-1, RMW-2, RMW-3, and RMW-4, respectively) for gauging, sampling and analysis. The proposed RI monitoring well locations are provided in Figure 6.

The monitoring wells will be installed for future groundwater sampling and analysis to delineate groundwater contamination and to provide a better understanding of onsite groundwater flow. The monitoring wells will be installed using a Geoprobe™ with augers and constructed with 10 ft of two-inch diameter, 0.02-inch slot poly vinyl chloride (PVC) screen to bridge the water table. The soil boring locations that will be converted into monitoring wells following sample collection, will be advanced to approximately 70 ft bls. The QAPP/FSP in Appendix B contains monitoring well design and construction details.

The new monitoring wells will be developed following installation to establish good hydraulic connection between the well and the surrounding formation aquifer. No sooner than one week following installation and development, a comprehensive water-level gauging event will occur prior to groundwater sampling to characterize groundwater flow onsite. Groundwater in each well will be measured using an electronic oil-water interface probe and a groundwater contour map will be developed using the gauging and survey data (as described in Section 3.2.5). Field parameters (e.g., pH, dissolved oxygen, oxidation-reduction potential,

turbidity, etc.) will be collected using a water quality meter during monitoring well purging, prior to sampling. Field parameters will be recorded on monitoring well sampling data forms. Following purging, a groundwater sample will be collected from each newly installed monitoring well.

Four groundwater samples will be collected for laboratory analysis from the monitoring wells. Additionally, one blind duplicate groundwater sample, MS groundwater sample, MSD groundwater sample, field blank, and trip blank will be collected for QA/QC purposes. Additional details regarding sampling methods are provided in the QAPP/FSP (Appendix B).

Water generated as part of the well development and purging effort prior to sample collection will be drummed, labeled, sampled, and stored onsite pending disposal.

3.2.4 Soil Vapor Intrusion Investigation

Five new permanent sub-slab vapor points will be installed immediately below the slab, two within the current basement footprint and three within the first floor footprint where there is no basement. All locations are shown on Figure 6. All five new vapor points will be sampled to evaluate sub-slab soil vapor conditions at the Site. A helium tracer gas test will be performed on each vapor point prior to sampling in accordance with the procedures outlined in the NYSDOH SVI Guidance.

Five indoor air samples will be collected from the breathing zone, next to each of the sub-slab soil vapor samples. To the extent practicable, indoor air samples will be placed in well-ventilated and open area typical of the normal working environment.

The outdoor ambient air sample (AMB-1) will be collected concurrently with the sub-slab soil vapor and indoor air samples. The outdoor ambient air sample will serve to better define the background atmospheric conditions within the area of the Site. This canister will be placed in a location chosen to provide representative background results based on conditions at the time of sampling. The ambient air will be sampled concurrently with the indoor air samples and by utilizing the same sample collection methods and equipment.

In order to evaluate the extent of contamination within the soil vapor, the active SSDS will be shut down a minimum of two weeks (14 days) prior to the soil vapor and indoor air sampling implementation. The system will be turned back on immediately following collection of the soil vapor and air samples.

3.2.5 Investigation Laboratory Analysis and Data Usability Summary Report

Soil and groundwater samples collected from the soil investigation described in detail below will be analyzed for the full TCL VOCs and SVOCs (including 1,4-Dioxane) + 30 (10 VOCs and 20 SVOCs) highest concentration tentatively identified compounds (TICs), TAL metals (including hexavalent chromium and total cyanide), TCL pesticides, TCL herbicides, and TCL PCBs. Approximately 25 percent (eight total) of the soil samples and all three groundwater samples will be analyzed for Per- and Polyfluoroalkyl Substances (PFAS). PFAS include the 40 compounds listed in accordance with the Sampling, Analysis, and Assessment of PFAS under NYSDEC's Part 375 Remedial Programs guidance document dated April 2023 (NYSDEC April 2023 PFAS Guidance). To delineate and characterize groundwater quality beneath the Site, groundwater samples will be analyzed for total and dissolved TAL metals. All SVI investigation samples will be analyzed using USEPA Method TO-15 for VOCs.

All samples will be analyzed at a NYSDOH Environmental Laboratory Approval Program-certified (ELAP) laboratory. Additional details regarding laboratory analyses are included in the QAPP/FSP (Appendix B). All analytical data for the RI will be received with standard 5-day turn-around-time.

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

3.2.6 Surveying

All newly installed monitoring wells sampled as part of this RI will be surveyed by a New York Licensed Surveyor to obtain horizontal and vertical coordinates and grade elevations. Measuring point elevations from the newly installed monitoring wells will also be surveyed to enable groundwater flow contouring. Horizontal coordinates will be based upon New York State Plane Coordinate System, Long Island Zone, North American Datum of 1983 (NAD 83) in US Survey Feet. Vertical elevations will be measured for top-of-casing (measuring point) and grade elevations referenced to North American Vertical Datum of 1988 (NAVD88).

3.2.7 Investigation Derived Waste

Following soil sample collection, boreholes that were not converted to wells will be backfilled with soil cuttings (unless gross contamination is observed, in which case it will be backfilled with clean sand), and the surface will be restored with either concrete or asphalt depending on the location. Excess soil cuttings and soil cuttings that are grossly contaminated will be placed into sealed and labeled Department of Transportation (DOT) approved 55-gallon drums, pending characterization for off-Site disposal at a permitted facility. Well development water and purge water generated during sampling will be placed into sealed and labeled DOT approved 55-gallon drums, pending characterization for off-Site disposal at a permitted facility.

3.2.8 Community Air Monitoring Program

Roux will implement the Community Air Monitoring Plan (CAMP) during any outdoor intrusive subsurface activities to conduct monitoring and protection for potential offsite receptors. The CAMP scope of work will include the use of one downwind air monitoring station and one upwind air monitoring station each equipped with one PID and one particulate meter. Upwind concentrations will be measured at the start of each workday and periodically throughout the day to establish background concentrations. Daily reporting of any exceedances will be provided to the NYSDEC and NYSDOH. The results of the CAMP data will be provided to the NYSDEC and NYSDOH on a weekly basis. CAMP data results will be provided in the final RIR along with locations of monitoring stations, monitoring equipment, procedures, and contaminant action levels. The NYSDOH Generic CAMP is included as Appendix C.

A Special Requirements CAMP is not anticipated as ground intrusive and soil handing activities inside the building will be conducted when the building is unoccupied (e.g, either outside of normal business hours [nights/weekends] or when the building is closed for typical operations). In addition, ground intrusive and soil handing activities outside the building will not be conducted within 20 feet of the building when occupied. If the building is occupied during implementation of the RI, then Special Requirements CAMP will be conducted.

3.3 Quality Assurance/ Quality Control (QA/QC) Protocols

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

3.4 Health and Safety

A Health and Safety Plan (HASP) has been prepared for the Site and is provided in Appendix D.

3.5 RI Implementation Sequence

Since the Site is an active design studio and retail manufacturing building with full time employees, the RI will be implemented in two parts to minimize the exposure to personnel. Part 1 will include the installation of the groundwater monitoring well (RMW-1) located in the courtyard (outside), as well the associated soil sample collection at this boring. RMW-1 will be developed following installation but will not be sampled at this time. RMW-1 will be redeveloped and sampled during Part 2. Part 1 is proposed to commence in Spring 2026 with limited Site employees onsite. Part 2 will include the remaining drilling and soil, groundwater, and soil vapor/indoor air sampling, which is all indoors. Part 2 is proposed for December 2026, when the Site employees are out-of-office for two weeks (or longer if needed) so that the drilling can occur while the building is unoccupied.

4. Reporting and Project Schedule

Roux and the Volunteer will commence implementation of this RIWP following the receipt of NYSDEC's written approval. Roux received a NYCTA/MTA letter of no impact on October 30, 2025. Roux will notify NYSDEC no less than seven days in advance of implementation of the RIWP, unless NYSDEC agrees to a shorter period in writing. Roux will also provide NYSDEC notice as to any change in the approved project schedule.

Daily Reports

Daily report submittals to be provided to the NYSDEC and NYSDOH during intrusive activities during the RI field implementation by 12pm the following day and will include the following:

- A summary of onsite work completed, and summary tables of the CAMP monitoring data will be submitted to NYSDEC on a daily basis;
- NYSDEC and NYSDOH will be notified immediately (within 24 hours) of any CAMP exceedances and corrective actions taken;
- Discovery of any previously unknown contaminated media during RI activities will be promptly communicated to NYSDEC's project manager; and
- A Site map will be provided to identify locations discussed in progress reports provided to NYSDEC.

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 RIWP 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 10th of the month following the end of monthly reporting period. Monthly reports will commence upon approval of the RIWP:

- 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.

RIR

Following the completion of both parts of the RI, a RIR will be prepared to summarize the results and findings of the RI. The RIR will include all data developed during the RI and will meet the technical requirements of DER-10. The RIR will summarize the results of the RI and the actions taken to implement the RIWP, include a detailed synopsis of all RIWP work performed, figures depicting the layout of the Site with sampling locations, figures presenting the analytical results and analyses performed, soil boring logs, monitoring well installation logs, tables presenting all laboratory data results, SVI evaluation, laboratory data packages, a DUSR, and sampling field forms.

RAWP

Following submission of the RIR, a RAWP will be prepared. The RAWP will present, develop, and evaluate potential remedial alternatives and identify a selected remedial approach for the Site. The RAWP will be prepared in accordance with the NYSDEC requirements for the BCP and will meet the technical requirements of DER-10.

Estimated Project Schedule	
Submit BCP Application and RIWP	September 2024
Implement RI Part 1	May 2026
Implement RI Part 2	December 2026
Submit RIR	April 2027
Submit RAWP	May 2027
Anticipated Issuance of Certificate of Completion	March 2028

TABLES

1. VOCs in Soils (WCD Phase II ESA Table)
1A. VOCs in Soils (WCD Phase II ESA Table)
2. PAHS in Soils (WCD Phase II ESA Table)
3. VOCs in Groundwater (WCD Phase II ESA Table)
4. VOCs in Soil Vapor (WCD Phase II ESA Table)
5. Petroleum Compounds in Soil (GBTS Tank Closure Table)
6. Summary of Volatile Organic Compounds in Soil Vapor (Roux SVI Investigation Letter)

Table 1: VOCs in Soils

WCD File: PQ18052.21

All data in mg/Kg (ppm)	**Sample ID**	**SB-01 13-15**		**SB-02 13-15**		**SB-03 5-7**		**SB-04 5-7**	
U= Not Detected ≥ indicated value	Sample Date	(2018-08-21)		(2018-08-21)		(2018-08-23)		(2018-08-23)	
Data above SCOs shown in **Bold**	Dilution Factor	1		1		1		1	
VOCs, 8260	**UUSCO**	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*
1,1,1,2-Tetrachloroethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,1,1-Trichloroethane	0.68	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,1,2,2-Tetrachloroethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,1,2-Trichloro-1,2,2-trifluoroethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,1,2-Trichloroethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,1-Dichloroethane	0.27	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,1-Dichloroethylene (1,1-DCE)	0.33	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,2,3-Trichlorobenzene	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,2,3-Trichloropropane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,2,4-Trichlorobenzene	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,2,4-Trimethylbenzene	3.6	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,2-Dibromo-3-chloropropane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,2-Dibromoethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,2-Dichlorobenzene	1.1	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,2-Dichloroethane	0.02	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,2-Dichloropropane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,3,5-Trimethylbenzene	8.4	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,3-Dichlorobenzene	2.4	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,4-Dichlorobenzene	1.8	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
1,4-Dioxane	0.1	0.05	*U*	0.049	*U*	0.065	*U*	0.057	*U*
2-Butanone (MEK)	0.12	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
2-Hexanone	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
4-Methyl-2-pentanone	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Acetone	0.05	0.005	*U*	0.0049	*U*	0.0065	*U*	0.0057	*U*
Acrolein	NA	0.005	*U*	0.0049	*U*	0.0065	*U*	0.0057	*U*
Acrylonitrile	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Benzene	0.06	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Bromochloromethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Bromodichloromethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Bromoform	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Bromomethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Carbon disulfide	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Carbon tetrachloride	0.76	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Chlorobenzene	1.1	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Chloroethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Chloroform	0.37	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Chloromethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
cis-1,2-Dichloroethylene (cis-DCE)	0.25	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
cis-1,3-Dichloropropylene	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Cyclohexane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Dibromochloromethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Dibromomethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Dichlorodifluoromethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Ethyl Benzene	1	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Hexachlorobutadiene	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Isopropylbenzene	2.3	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Methyl acetate	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Methyl tert-butyl ether (MTBE)	0.93	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Methylcyclohexane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Methylene chloride	0.05	0.005	*U*	0.0049	*U*	0.0065	*U*	0.0057	*U*
n-Butylbenzene	12	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
n-Propylbenzene	3.9	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
o-Xylene	0.26	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
p- & m- Xylenes	0.26	0.005	*U*	0.0049	*U*	0.0065	*U*	0.0057	*U*
p-Isopropyltoluene	10	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
sec-Butylbenzene	11	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Styrene	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
tert-Butyl alcohol (TBA)	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
tert-Butylbenzene	5.9	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Tetrachloroethylene (PCE)	1.3	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Toluene	0.7	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
trans-1,2-Dichloroethylene (trans-DCE)	0.19	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
trans-1,3-Dichloropropylene	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Trichloroethylene (TCE)	0.47	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Trichlorofluoromethane	NA	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Vinyl chloride (VC)	0.02	0.0025	*U*	0.0024	*U*	0.0033	*U*	0.0029	*U*
Xylenes, Total	0.26	0.0076	*U*	0.0073	*U*	0.0098	*U*	0.0086	*U*

Analyte Detected

Analyte Above UUSCO

Notes: SCOs based on NYSDEC Part 375-6.8 and CP-51 NA = not available

Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Table 1: VOCs in Soils

WCD File: PQ18052.21

All data in mg/Kg (ppm)	**Sample ID**	**SB-05 9.5-11.5**	
U= Not Detected ≥ indicated value	Sample Date	(2018-08-23)	
Data above SCOs shown in **Bold**	Dilution Factor	1	
VOCs, 8260	**UUSCO**	*Result*	*Qualifier*
1,1,1,2-Tetrachloroethane	NA	0.0026	*U*
1,1,1-Trichloroethane	0.68	0.0026	*U*
1,1,2,2-Tetrachloroethane	NA	0.0026	*U*
1,1,2-Trichloro-1,2,2-trifluoroethane	NA	0.0026	*U*
1,1,2-Trichloroethane	NA	0.0026	*U*
1,1-Dichloroethane	0.27	0.0026	*U*
1,1-Dichloroethylene (1,1-DCE)	0.33	0.0026	*U*
1,2,3-Trichlorobenzene	NA	0.0026	*U*
1,2,3-Trichloropropane	NA	0.0026	*U*
1,2,4-Trichlorobenzene	NA	0.0026	*U*
1,2,4-Trimethylbenzene	3.6	0.0026	*U*
1,2-Dibromo-3-chloropropane	NA	0.0026	*U*
1,2-Dibromoethane	NA	0.0026	*U*
1,2-Dichlorobenzene	1.1	0.0026	*U*
1,2-Dichloroethane	0.02	0.0026	*U*
1,2-Dichloropropane	NA	0.0026	*U*
1,3,5-Trimethylbenzene	8.4	0.0026	*U*
1,3-Dichlorobenzene	2.4	0.0026	*U*
1,4-Dichlorobenzene	1.8	0.0026	*U*
1,4-Dioxane	0.1	0.053	*U*
2-Butanone (MEK)	0.12	0.0026	*U*
2-Hexanone	NA	0.0026	*U*
4-Methyl-2-pentanone	NA	0.0026	*U*
Acetone	0.05	0.0053	*U*
Acrolein	NA	0.0053	*U*
Acrylonitrile	NA	0.0026	*U*
Benzene	0.06	0.0026	*U*
Bromochloromethane	NA	0.0026	*U*
Bromodichloromethane	NA	0.0026	*U*
Bromoform	NA	0.0026	*U*
Bromomethane	NA	0.0026	*U*
Carbon disulfide	NA	0.0026	*U*
Carbon tetrachloride	0.76	0.0026	*U*
Chlorobenzene	1.1	0.0026	*U*
Chloroethane	NA	0.0026	*U*
Chloroform	0.37	0.0026	*U*
Chloromethane	NA	0.0026	*U*
cis-1,2-Dichloroethylene (cis-DCE)	0.25	0.0026	*U*
cis-1,3-Dichloropropylene	NA	0.0026	*U*
Cyclohexane	NA	0.0026	*U*
Dibromochloromethane	NA	0.0026	*U*
Dibromomethane	NA	0.0026	*U*
Dichlorodifluoromethane	NA	0.0026	*U*
Ethyl Benzene	1	0.0026	*U*
Hexachlorobutadiene	NA	0.0026	*U*
Isopropylbenzene	2.3	0.0026	*U*
Methyl acetate	NA	0.0026	*U*
Methyl tert-butyl ether (MTBE)	0.93	0.0026	*U*
Methylcyclohexane	NA	0.0026	*U*
Methylene chloride	0.05	0.0053	*U*
n-Butylbenzene	12	0.0026	*U*
n-Propylbenzene	3.9	0.0026	*U*
o-Xylene	0.26	0.0026	*U*
p- & m- Xylenes	0.26	0.0053	*U*
p-Isopropyltoluene	10	0.0026	*U*
sec-Butylbenzene	11	0.0026	*U*
Styrene	NA	0.0026	*U*
tert-Butyl alcohol (TBA)	NA	0.0026	*U*
tert-Butylbenzene	5.9	0.0026	*U*
Tetrachloroethylene (PCE)	1.3	0.0026	*U*
Toluene	0.7	0.0026	*U*
trans-1,2-Dichloroethylene (trans-DCE)	0.19	0.0026	*U*
trans-1,3-Dichloropropylene	NA	0.0026	*U*
Trichloroethylene (TCE)	0.47	0.0026	*U*
Trichlorofluoromethane	NA	0.0026	*U*
Vinyl chloride (VC)	0.02	0.0026	*U*
Xylenes, Total	0.26	0.0079	*U*

Analyte Detected

Analyte Above UUSCO

Notes: SCOs based on NYSDEC Part 375-6.8 and CP-51 NA = not available

Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Table 1A: VOCs in Soils

WCD File: PQ18052.21

All data in mg/Kg (ppm)	**Sample ID**	**HB-01 2-3**		**HB-02 2-3**		**HB-03 3-4**	
U= Not Detected ≥ indicated value	Sample Date	(2018-10-22)		(2018-10-22)		(2018-10-22)	
Data above SCOs shown in **Bold**	Dilution Factor	1		1		1	
VOCs, 8260	**UUSCO**	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*
1,1,1,2-Tetrachloroethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,1,1-Trichloroethane	0.68	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,1,2,2-Tetrachloroethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,1,2-Trichloro-1,2,2-trifluoroethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,1,2-Trichloroethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,1-Dichloroethane	0.27	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,1-Dichloroethylene (1,1-DCE)	0.33	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2,3-Trichlorobenzene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2,3-Trichloropropane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2,4-Trichlorobenzene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2,4-Trimethylbenzene	3.6	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2-Dibromo-3-chloropropane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2-Dibromoethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2-Dichlorobenzene	1.1	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2-Dichloroethane	0.02	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2-Dichloropropane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,3-Dichlorobenzene	2.4	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,3-Dichloropropane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,4-Dichlorobenzene	1.8	0.0026	*U*	0.0029	*U*	0.0024	*U*
2,2-Dichloropropane	0.1	0.0026	*U*	0.0029	*U*	0.0024	*U*
2-Chlorotoluene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
4-Chlorotoluene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Bromobenzene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Bromochloromethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Bromodichloromethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Bromoform	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Bromomethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Carbon tetrachloride	0.76	0.0026	*U*	0.0029	*U*	0.0024	*U*
Chlorobenzene	1.1	0.0026	*U*	0.0029	*U*	0.0024	*U*
Chloroethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Chloroform	0.37	0.0026	*U*	0.0029	*U*	0.0024	*U*
Chloromethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
cis-1,2-Dichloroethylene (cis-DCE)	0.25	0.0026	*U*	0.0029	*U*	0.0024	*U*
cis-1,3-Dichloropropylene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Dibromochloromethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Dibromomethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Dichlorodifluoromethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Hexachlorobutadiene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Methylene chloride	0.05	0.0053	*U*	0.0057	*U*	0.0048	*U*
Tetrachloroethylene (PCE)	1.3	0.0026	*U*	0.0029	*U*	0.0024	*U*
trans-1,2-Dichloroethylene (trans-DCE)	0.19	0.0026	*U*	0.0029	*U*	0.0024	*U*
trans-1,3-Dichloropropylene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Trichloroethylene (TCE)	0.47	0.0026	*U*	0.0029	*U*	0.0024	*U*
Trichlorofluoromethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Vinyl chloride (VC)	0.02	0.0026	*U*	0.0029	*U*	0.0024	*U*

Analyte Detected

Analyte Above UUSCO

Notes: SCOs based on NYSDEC Part 375-6.8 and CP-51 NA = not available

Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Table 1A: VOCs in Soils

WCD File: PQ18052.21

All data in mg/Kg (ppm)	**Sample ID**	**HB-04 3-4**		**HB-05 5-6**		**HB-06 4-5**	
U= Not Detected ≥ indicated value	Sample Date	(2018-10-22)		(2018-10-22)		(2018-10-22)	
Data above SCOs shown in **Bold**	Dilution Factor	1		1		1	
VOCs, 8260	**UUSCO**	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*
1,1,1,2-Tetrachloroethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,1,1-Trichloroethane	0.68	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,1,2,2-Tetrachloroethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,1,2-Trichloro-1,2,2-trifluoroethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,1,2-Trichloroethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,1-Dichloroethane	0.27	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,1-Dichloroethylene (1,1-DCE)	0.33	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2,3-Trichlorobenzene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2,3-Trichloropropane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2,4-Trichlorobenzene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2,4-Trimethylbenzene	3.6	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2-Dibromo-3-chloropropane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2-Dibromoethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2-Dichlorobenzene	1.1	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2-Dichloroethane	0.02	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2-Dichloropropane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,3-Dichlorobenzene	2.4	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,3-Dichloropropane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,4-Dichlorobenzene	1.8	0.0023	*U*	0.0025	*U*	0.0021	*U*
2,2-Dichloropropane	0.1	0.0023	*U*	0.0025	*U*	0.0021	*U*
2-Chlorotoluene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
4-Chlorotoluene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Bromobenzene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Bromochloromethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Bromodichloromethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Bromoform	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Bromomethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Carbon tetrachloride	0.76	0.0023	*U*	0.0025	*U*	0.0021	*U*
Chlorobenzene	1.1	0.0023	*U*	0.0025	*U*	0.0021	*U*
Chloroethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Chloroform	0.37	0.0023	*U*	0.0025	*U*	0.0021	*U*
Chloromethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
cis-1,2-Dichloroethylene (cis-DCE)	0.25	0.0023	*U*	0.0025	*U*	0.0021	*U*
cis-1,3-Dichloropropylene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Dibromochloromethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Dibromomethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Dichlorodifluoromethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Hexachlorobutadiene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Methylene chloride	0.05	0.0047	*U*	0.005	*U*	0.0042	*U*
Tetrachloroethylene (PCE)	1.3	0.0023	*U*	0.0025	*U*	0.0021	*U*
trans-1,2-Dichloroethylene (trans-DCE)	0.19	0.0023	*U*	0.0025	*U*	0.0021	*U*
trans-1,3-Dichloropropylene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Trichloroethylene (TCE)	0.47	0.0023	*U*	0.0025	*U*	0.0021	*U*
Trichlorofluoromethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Vinyl chloride (VC)	0.02	0.0023	*U*	0.0025	*U*	0.0021	*U*

Analyte Detected

Analyte Above UUSCO

Notes: SCOs based on NYSDEC Part 375-6.8 and CP-51 NA = not available

Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Table 1A: VOCs in Soils

WCD File: PQ18052.21

All data in mg/Kg (ppm)	**Sample ID**	**SB-06 4-5**		**SB-07 4-5**		**SB-08 4-5**	
U= Not Detected ≥ indicated value	Sample Date	(2018-10-22)		(2018-10-22)		(2018-10-22)	
Data above SCOs shown in **Bold**	Dilution Factor	1		1		1	
VOCs, 8260	**UUSCO**	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*
1,1,1,2-Tetrachloroethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,1,1-Trichloroethane	0.68	0.0028	*U*	0.0064		0.0027	*U*
1,1,2,2-Tetrachloroethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,1,2-Trichloro-1,2,2-trifluoroethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,1,2-Trichloroethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,1-Dichloroethane	0.27	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,1-Dichloroethylene (1,1-DCE)	0.33	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2,3-Trichlorobenzene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2,3-Trichloropropane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2,4-Trichlorobenzene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2,4-Trimethylbenzene	3.6	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2-Dibromo-3-chloropropane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2-Dibromoethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2-Dichlorobenzene	1.1	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2-Dichloroethane	0.02	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2-Dichloropropane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,3-Dichlorobenzene	2.4	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,3-Dichloropropane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,4-Dichlorobenzene	1.8	0.0028	*U*	0.0028	*U*	0.0027	*U*
2,2-Dichloropropane	0.1	0.0028	*U*	0.0028	*U*	0.0027	*U*
2-Chlorotoluene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
4-Chlorotoluene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Bromobenzene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Bromochloromethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Bromodichloromethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Bromoform	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Bromomethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Carbon tetrachloride	0.76	0.0028	*U*	0.0028	*U*	0.0027	*U*
Chlorobenzene	1.1	0.0028	*U*	0.0028	*U*	0.0027	*U*
Chloroethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Chloroform	0.37	0.0028	*U*	0.007		0.0027	*U*
Chloromethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
cis-1,2-Dichloroethylene (cis-DCE)	0.25	0.0028	*U*	0.0028	*U*	0.0027	*U*
cis-1,3-Dichloropropylene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Dibromochloromethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Dibromomethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Dichlorodifluoromethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Hexachlorobutadiene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Methylene chloride	0.05	0.0055	*U*	0.0056	*U*	0.0053	*U*
Tetrachloroethylene (PCE)	1.3	0.0041	*J*	0.0035	*J*	0.0027	*U*
trans-1,2-Dichloroethylene (trans-DCE)	0.19	0.0028	*U*	0.0028	*U*	0.0027	*U*
trans-1,3-Dichloropropylene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Trichloroethylene (TCE)	0.47	0.0028	*U*	0.0028	*U*	0.0027	*U*
Trichlorofluoromethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Vinyl chloride (VC)	0.02	0.0028	*U*	0.0028	*U*	0.0027	*U*

Analyte Detected

Analyte Above UUSCO

Notes: SCOs based on NYSDEC Part 375-6.8 and CP-51 NA = not available

Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Table 1A: VOCs in Soils

WCD File: PQ18052.21

All data in mg/Kg (ppm)	**Sample ID**	**SB-09 4-5**	
U= Not Detected ≥ indicated value	Sample Date	(2018-10-22)	
Data above SCOs shown in **Bold**	Dilution Factor	1	
VOCs, 8260	**UUSCO**	*Result*	*Qualifier*
1,1,1,2-Tetrachloroethane	NA	0.0027	*U*
1,1,1-Trichloroethane	0.68	0.0027	*U*
1,1,2,2-Tetrachloroethane	NA	0.0027	*U*
1,1,2-Trichloro-1,2,2-trifluoroethane	NA	0.0027	*U*
1,1,2-Trichloroethane	NA	0.0027	*U*
1,1-Dichloroethane	0.27	0.0027	*U*
1,1-Dichloroethylene (1,1-DCE)	0.33	0.0027	*U*
1,2,3-Trichlorobenzene	NA	0.0027	*U*
1,2,3-Trichloropropane	NA	0.0027	*U*
1,2,4-Trichlorobenzene	NA	0.0027	*U*
1,2,4-Trimethylbenzene	3.6	0.0027	*U*
1,2-Dibromo-3-chloropropane	NA	0.0027	*U*
1,2-Dibromoethane	NA	0.0027	*U*
1,2-Dichlorobenzene	1.1	0.0027	*U*
1,2-Dichloroethane	0.02	0.0027	*U*
1,2-Dichloropropane	NA	0.0027	*U*
1,3-Dichlorobenzene	2.4	0.0027	*U*
1,3-Dichloropropane	NA	0.0027	*U*
1,4-Dichlorobenzene	1.8	0.0027	*U*
2,2-Dichloropropane	0.1	0.0027	*U*
2-Chlorotoluene	NA	0.0027	*U*
4-Chlorotoluene	NA	0.0027	*U*
Bromobenzene	NA	0.0027	*U*
Bromochloromethane	NA	0.0027	*U*
Bromodichloromethane	NA	0.0027	*U*
Bromoform	NA	0.0027	*U*
Bromomethane	NA	0.0027	*U*
Carbon tetrachloride	0.76	0.0027	*U*
Chlorobenzene	1.1	0.0027	*U*
Chloroethane	NA	0.0027	*U*
Chloroform	0.37	0.0027	*U*
Chloromethane	NA	0.0027	*U*
cis-1,2-Dichloroethylene (cis-DCE)	0.25	0.0027	*U*
cis-1,3-Dichloropropylene	NA	0.0027	*U*
Dibromochloromethane	NA	0.0027	*U*
Dibromomethane	NA	0.0027	*U*
Dichlorodifluoromethane	NA	0.0027	*U*
Hexachlorobutadiene	NA	0.0027	*U*
Methylene chloride	0.05	0.0053	*U*
Tetrachloroethylene (PCE)	1.3	0.0027	*U*
trans-1,2-Dichloroethylene (trans-DCE)	0.19	0.0027	*U*
trans-1,3-Dichloropropylene	NA	0.0027	*U*
Trichloroethylene (TCE)	0.47	0.0027	*U*
Trichlorofluoromethane	NA	0.0027	*U*
Vinyl chloride (VC)	0.02	0.0027	*U*

Analyte Detected

Analyte Above UUSCO

Notes: SCOs based on NYSDEC Part 375-6.8 and CP-51 NA = not available
Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Table 1A: VOCs in Soils

WCD File: PQ18052.21

All data in mg/Kg (ppm)	**Sample ID**	**HB-01 2-3**		**HB-02 2-3**		**HB-03 3-4**	
U= Not Detected ≥ indicated value	Sample Date	(2018-10-22)		(2018-10-22)		(2018-10-22)	
Data above SCOs shown in **Bold**	Dilution Factor	1		1		1	
VOCs, 8260	**UUSCO**	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*
1,1,1,2-Tetrachloroethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,1,1-Trichloroethane	0.68	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,1,2,2-Tetrachloroethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,1,2-Trichloro-1,2,2-trifluoroethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,1,2-Trichloroethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,1-Dichloroethane	0.27	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,1-Dichloroethylene (1,1-DCE)	0.33	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2,3-Trichlorobenzene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2,3-Trichloropropane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2,4-Trichlorobenzene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2,4-Trimethylbenzene	3.6	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2-Dibromo-3-chloropropane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2-Dibromoethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2-Dichlorobenzene	1.1	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2-Dichloroethane	0.02	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,2-Dichloropropane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,3-Dichlorobenzene	2.4	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,3-Dichloropropane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
1,4-Dichlorobenzene	1.8	0.0026	*U*	0.0029	*U*	0.0024	*U*
2,2-Dichloropropane	0.1	0.0026	*U*	0.0029	*U*	0.0024	*U*
2-Chlorotoluene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
4-Chlorotoluene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Bromobenzene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Bromochloromethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Bromodichloromethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Bromoform	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Bromomethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Carbon tetrachloride	0.76	0.0026	*U*	0.0029	*U*	0.0024	*U*
Chlorobenzene	1.1	0.0026	*U*	0.0029	*U*	0.0024	*U*
Chloroethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Chloroform	0.37	0.0026	*U*	0.0029	*U*	0.0024	*U*
Chloromethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
cis-1,2-Dichloroethylene (cis-DCE)	0.25	0.0026	*U*	0.0029	*U*	0.0024	*U*
cis-1,3-Dichloropropylene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Dibromochloromethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Dibromomethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Dichlorodifluoromethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Hexachlorobutadiene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Methylene chloride	0.05	0.0053	*U*	0.0057	*U*	0.0048	*U*
Tetrachloroethylene (PCE)	1.3	0.0026	*U*	0.0029	*U*	0.0024	*U*
trans-1,2-Dichloroethylene (trans-DCE)	0.19	0.0026	*U*	0.0029	*U*	0.0024	*U*
trans-1,3-Dichloropropylene	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Trichloroethylene (TCE)	0.47	0.0026	*U*	0.0029	*U*	0.0024	*U*
Trichlorofluoromethane	NA	0.0026	*U*	0.0029	*U*	0.0024	*U*
Vinyl chloride (VC)	0.02	0.0026	*U*	0.0029	*U*	0.0024	*U*

Analyte Detected

Analyte Above UUSCO

Notes: SCOs based on NYSDEC Part 375-6.8 and CP-51 NA = not available

Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Table 1A: VOCs in Soils

WCD File: PQ18052.21

All data in mg/Kg (ppm)	**Sample ID**	**HB-04 3-4**		**HB-05 5-6**		**HB-06 4-5**	
U= Not Detected ≥ indicated value	Sample Date	(2018-10-22)		(2018-10-22)		(2018-10-22)	
Data above SCOs shown in **Bold**	Dilution Factor	1		1		1	
VOCs, 8260	**UUSCO**	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*
1,1,1,2-Tetrachloroethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,1,1-Trichloroethane	0.68	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,1,2,2-Tetrachloroethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,1,2-Trichloro-1,2,2-trifluoroethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,1,2-Trichloroethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,1-Dichloroethane	0.27	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,1-Dichloroethylene (1,1-DCE)	0.33	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2,3-Trichlorobenzene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2,3-Trichloropropane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2,4-Trichlorobenzene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2,4-Trimethylbenzene	3.6	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2-Dibromo-3-chloropropane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2-Dibromoethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2-Dichlorobenzene	1.1	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2-Dichloroethane	0.02	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,2-Dichloropropane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,3-Dichlorobenzene	2.4	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,3-Dichloropropane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
1,4-Dichlorobenzene	1.8	0.0023	*U*	0.0025	*U*	0.0021	*U*
2,2-Dichloropropane	0.1	0.0023	*U*	0.0025	*U*	0.0021	*U*
2-Chlorotoluene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
4-Chlorotoluene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Bromobenzene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Bromochloromethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Bromodichloromethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Bromoform	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Bromomethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Carbon tetrachloride	0.76	0.0023	*U*	0.0025	*U*	0.0021	*U*
Chlorobenzene	1.1	0.0023	*U*	0.0025	*U*	0.0021	*U*
Chloroethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Chloroform	0.37	0.0023	*U*	0.0025	*U*	0.0021	*U*
Chloromethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
cis-1,2-Dichloroethylene (cis-DCE)	0.25	0.0023	*U*	0.0025	*U*	0.0021	*U*
cis-1,3-Dichloropropylene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Dibromochloromethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Dibromomethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Dichlorodifluoromethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Hexachlorobutadiene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Methylene chloride	0.05	0.0047	*U*	0.005	*U*	0.0042	*U*
Tetrachloroethylene (PCE)	1.3	0.0023	*U*	0.0025	*U*	0.0021	*U*
trans-1,2-Dichloroethylene (trans-DCE)	0.19	0.0023	*U*	0.0025	*U*	0.0021	*U*
trans-1,3-Dichloropropylene	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Trichloroethylene (TCE)	0.47	0.0023	*U*	0.0025	*U*	0.0021	*U*
Trichlorofluoromethane	NA	0.0023	*U*	0.0025	*U*	0.0021	*U*
Vinyl chloride (VC)	0.02	0.0023	*U*	0.0025	*U*	0.0021	*U*

Analyte Detected

Analyte Above UUSCO

Notes: SCOs based on NYSDEC Part 375-6.8 and CP-51 NA = not available

Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Table 1A: VOCs in Soils

WCD File: PQ18052.21

All data in mg/Kg (ppm)	**Sample ID**	**SB-06 4-5**		**SB-07 4-5**		**SB-08 4-5**	
U= Not Detected ≥ indicated value	Sample Date	(2018-10-22)		(2018-10-22)		(2018-10-22)	
Data above SCOs shown in **Bold**	Dilution Factor	1		1		1	
VOCs, 8260	**UUSCO**	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*
1,1,1,2-Tetrachloroethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,1,1-Trichloroethane	0.68	0.0028	*U*	0.0064		0.0027	*U*
1,1,2,2-Tetrachloroethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,1,2-Trichloro-1,2,2-trifluoroethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,1,2-Trichloroethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,1-Dichloroethane	0.27	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,1-Dichloroethylene (1,1-DCE)	0.33	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2,3-Trichlorobenzene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2,3-Trichloropropane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2,4-Trichlorobenzene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2,4-Trimethylbenzene	3.6	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2-Dibromo-3-chloropropane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2-Dibromoethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2-Dichlorobenzene	1.1	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2-Dichloroethane	0.02	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,2-Dichloropropane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,3-Dichlorobenzene	2.4	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,3-Dichloropropane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
1,4-Dichlorobenzene	1.8	0.0028	*U*	0.0028	*U*	0.0027	*U*
2,2-Dichloropropane	0.1	0.0028	*U*	0.0028	*U*	0.0027	*U*
2-Chlorotoluene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
4-Chlorotoluene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Bromobenzene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Bromochloromethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Bromodichloromethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Bromoform	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Bromomethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Carbon tetrachloride	0.76	0.0028	*U*	0.0028	*U*	0.0027	*U*
Chlorobenzene	1.1	0.0028	*U*	0.0028	*U*	0.0027	*U*
Chloroethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Chloroform	0.37	0.0028	*U*	0.007		0.0027	*U*
Chloromethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
cis-1,2-Dichloroethylene (cis-DCE)	0.25	0.0028	*U*	0.0028	*U*	0.0027	*U*
cis-1,3-Dichloropropylene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Dibromochloromethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Dibromomethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Dichlorodifluoromethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Hexachlorobutadiene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Methylene chloride	0.05	0.0055	*U*	0.0056	*U*	0.0053	*U*
Tetrachloroethylene (PCE)	1.3	0.0041	*J*	0.0035	*J*	0.0027	*U*
trans-1,2-Dichloroethylene (trans-DCE)	0.19	0.0028	*U*	0.0028	*U*	0.0027	*U*
trans-1,3-Dichloropropylene	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Trichloroethylene (TCE)	0.47	0.0028	*U*	0.0028	*U*	0.0027	*U*
Trichlorofluoromethane	NA	0.0028	*U*	0.0028	*U*	0.0027	*U*
Vinyl chloride (VC)	0.02	0.0028	*U*	0.0028	*U*	0.0027	*U*

Analyte Detected

Analyte Above UUSCO

Notes: SCOs based on NYSDEC Part 375-6.8 and CP-51 NA = not available

Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Table 1A: VOCs in Soils

WCD File: PQ18052.21

All data in mg/Kg (ppm)	**Sample ID**	**SB-09 4-5**	
U= Not Detected ≥ indicated value	Sample Date	(2018-10-22)	
Data above SCOs shown in **Bold**	Dilution Factor	1	
VOCs, 8260	**UUSCO**	*Result*	*Qualifier*
1,1,1,2-Tetrachloroethane	NA	0.0027	*U*
1,1,1-Trichloroethane	0.68	0.0027	*U*
1,1,2,2-Tetrachloroethane	NA	0.0027	*U*
1,1,2-Trichloro-1,2,2-trifluoroethane	NA	0.0027	*U*
1,1,2-Trichloroethane	NA	0.0027	*U*
1,1-Dichloroethane	0.27	0.0027	*U*
1,1-Dichloroethylene (1,1-DCE)	0.33	0.0027	*U*
1,2,3-Trichlorobenzene	NA	0.0027	*U*
1,2,3-Trichloropropane	NA	0.0027	*U*
1,2,4-Trichlorobenzene	NA	0.0027	*U*
1,2,4-Trimethylbenzene	3.6	0.0027	*U*
1,2-Dibromo-3-chloropropane	NA	0.0027	*U*
1,2-Dibromoethane	NA	0.0027	*U*
1,2-Dichlorobenzene	1.1	0.0027	*U*
1,2-Dichloroethane	0.02	0.0027	*U*
1,2-Dichloropropane	NA	0.0027	*U*
1,3-Dichlorobenzene	2.4	0.0027	*U*
1,3-Dichloropropane	NA	0.0027	*U*
1,4-Dichlorobenzene	1.8	0.0027	*U*
2,2-Dichloropropane	0.1	0.0027	*U*
2-Chlorotoluene	NA	0.0027	*U*
4-Chlorotoluene	NA	0.0027	*U*
Bromobenzene	NA	0.0027	*U*
Bromochloromethane	NA	0.0027	*U*
Bromodichloromethane	NA	0.0027	*U*
Bromoform	NA	0.0027	*U*
Bromomethane	NA	0.0027	*U*
Carbon tetrachloride	0.76	0.0027	*U*
Chlorobenzene	1.1	0.0027	*U*
Chloroethane	NA	0.0027	*U*
Chloroform	0.37	0.0027	*U*
Chloromethane	NA	0.0027	*U*
cis-1,2-Dichloroethylene (cis-DCE)	0.25	0.0027	*U*
cis-1,3-Dichloropropylene	NA	0.0027	*U*
Dibromochloromethane	NA	0.0027	*U*
Dibromomethane	NA	0.0027	*U*
Dichlorodifluoromethane	NA	0.0027	*U*
Hexachlorobutadiene	NA	0.0027	*U*
Methylene chloride	0.05	0.0053	*U*
Tetrachloroethylene (PCE)	1.3	0.0027	*U*
trans-1,2-Dichloroethylene (trans-DCE)	0.19	0.0027	*U*
trans-1,3-Dichloropropylene	NA	0.0027	*U*
Trichloroethylene (TCE)	0.47	0.0027	*U*
Trichlorofluoromethane	NA	0.0027	*U*
Vinyl chloride (VC)	0.02	0.0027	*U*

Analyte Detected

Analyte Above UUSCO

Notes: SCOs based on NYSDEC Part 375-6.8 and CP-51 NA = not available
Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Table 2: PAHs in Soils

WCD File: PQ18052.20

All data in mg/Kg (ppm)	**Sample ID**	**SB-01 13-15**		**SB-02 13-15**		**SB-03 5-7**		**SB-04 5-7**		**SB-05 9.5-11.5**	
U= Not Detected ≥ indicated value	Sample Date	(2018-08-21)		(2018-08-21)		(2018-08-23)		(2018-08-23)		(2018-08-23)	
Data above SCOs shown in **Bold**	Dilution Factor	2		2		2		2		2	
SVOCs, 8270	**UUSCO**	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*
2-Methylnaphthalene	NA	0.0432	*U*	0.0431	*U*	0.0501	*U*	0.0458	*U*	0.0432	*U*
Acenaphthene	20	0.0432	*U*	0.0431	*U*	0.0501	*U*	0.0458	*U*	0.0432	*U*
Acenaphthylene	100	0.0432	*U*	0.0431	*U*	0.0501	*U*	0.0458	*U*	0.0432	*U*
Anthracene	100	0.0432	*U*	0.0431	*U*	0.0501	*U*	0.0461	*JD*	0.0432	*U*
Benzo(a)anthracene	1	0.0432	*U*	0.0431	*U*	0.0856	*JD*	0.284	*D*	0.0432	*U*
Benzo(a)pyrene	1	0.0432	*U*	0.0431	*U*	0.122	*D*	0.316	*D*	0.0432	*U*
Benzo(b)fluoranthene	1	0.0432	*U*	0.0431	*U*	0.12	*D*	0.306	*D*	0.0432	*U*
Benzo(g,h,i)perylene	100	0.0432	*U*	0.0431	*U*	0.0776	*JD*	0.193	*D*	0.0432	*U*
Benzo(k)fluoranthene	0.8	0.0432	*U*	0.0431	*U*	0.114	*D*	0.274	*D*	0.0432	*U*
Chrysene	1	0.0432	*U*	0.0431	*U*	0.136	*D*	0.279	*D*	0.0432	*U*
Dibenzo(a,h)anthracene	0.33	0.0432	*U*	0.0431	*U*	0.0501	*U*	0.057	*JD*	0.0432	*U*
Fluoranthene	100	0.0432	*U*	0.0431	*U*	0.314	*D*	0.455	*D*	0.0432	*U*
Fluorene	30	0.0432	*U*	0.0431	*U*	0.0501	*U*	0.0458	*U*	0.0432	*U*
Indeno(1,2,3-cd)pyrene	0.5	0.0432	*U*	0.0431	*U*	0.0656	*JD*	0.2	*D*	0.0432	*U*
Naphthalene	12	0.0432	*U*	0.0431	*U*	0.0501	*U*	0.0458	*U*	0.0432	*U*
Phenanthrene	100	0.0432	*U*	0.0431	*U*	0.192	*D*	0.224	*D*	0.0432	*U*
Pyrene	100	0.0432	*U*	0.0431	*U*	0.228	*D*	0.393	*D*	0.0432	*U*

Analyte Detected

Analyte Above UUSCO

Notes: SCOs based on NYSDEC Part 375-6.8 and CP-51 NA = not available

Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Table 3: VOCs in Groundwater

WCD File: PQ18052.21

All data in µg/L (parts per billion, ppb)	**Sample ID**	**MW-01**	
U= Not Detected ≥ indicated value	Sample Date	(2018-11-05)	
Data above AWQS shown in **Bold**	Dilution Factor	1	
VOCs, 8260	**AWQS**	*Result*	*Qualifier*
1,1,1,2-tetrachloroethane	5	0.21	*J*
1,1,1-trichloroethane	5	0.31	*J*
1,1,2,2-tetrachloroethane	5	0.2	*U*
1,1,2-trichloro-1,2,2-trifluoroethane	5	0.2	*U*
1,1,2-trichloroethane	1	0.2	*U*
1,1-dichloroethane	5	0.2	*U*
1,1-dichloroethylene (1,1-DCE)	5	0.2	*U*
1,2,3-trichlorobenzene	5	0.2	*U*
1,2,3-trichloropropane	0.04	0.2	*U*
1,2,4-trichlorobenzene	5	0.2	*U*
1,2,4-trimethylbenzene	5	0.21	*J*
1,2-dibromo-3-chloropropane	0.04	0.2	*U*
1,2-dibromoethane	5	0.2	*U*
1,2-dichlorobenzene	3	0.2	*U*
1,2-dichloroethane	0.6	0.2	*U*
1,2-dichloropropane	1	0.2	*U*
1,3,5-trimethylbenzene	5	0.2	*U*
1,3-dichlorobenzene	3	0.2	*U*
1,4-dichlorobenzene	3	0.2	*U*
1,4-dioxane	NA	40	*U*
2-butanone (MEK)	50	0.2	*U*
2-hexanone (MBK)	50	0.2	*U*
4-methyl-2-pentanone	NA	0.2	*U*
acetone	50	1	*U*
acrolein	5	0.2	*U*
acrylonitrile	5	0.2	*U*
benzene	1	0.2	*U*
bromochloromethane	5	0.2	*U*
bromodichloromethane	50	0.2	*U*
bromoform	50	0.2	*U*
bromomethane	5	0.2	*U*
carbon disulfide	NA	0.2	*U*
carbon tetrachloride	5	0.2	*U*
chlorobenzene	5	0.2	*U*
chloroethane	5	0.2	*U*
chloroform	7	1.8	
chloromethane	5	0.37	*J*
cis-1,2-dichloroethylene (cis-DCE)	5	4.9	
cis-1,3-dichloropropylene	0.4	0.2	*U*
cyclohexane	NA	0.2	*U*
dibromochloromethane	5	0.2	*U*
dibromomethane	5	0.2	*U*
dichlorodifluoromethane	5	0.2	*U*
ethyl benzene	5	0.2	*U*
hexachlorobutadiene	0.5	0.2	*U*
isopropylbenzene	5	0.2	*U*
methyl acetate	NA	0.2	*U*
methyl tert-butyl ether (MTBE)	10	0.2	*U*
methylcyclohexane	NA	0.2	*U*
methylene chloride	5	1	*U*
n-butylbenzene	5	0.2	*U*
n-propylbenzene	5	0.2	*U*
o-xylene (included in total xylenes)	5	0.29	*J*
p- & m- xylenes (included in total xylenes)	5	0.65	*J*
p-isopropyltoluene	5	0.2	*U*
sec-butylbenzene	5	0.2	*U*
styrene	5	0.2	*U*
tert-butyl alcohol (TBA)	NA	0.5	*U*
tert-butylbenzene	5	0.2	*U*
tetrachloroethylene (PCE)	5	**150**	*E*
toluene	5	**11**	
trans-1,2-dichloroethylene (trans-DCE)	5	0.2	*U*
trans-1,3-dichloropropylene	0.4	0.2	*U*
trans-1,4-dichloro-2-butene	NA	0.2	*U*
trichloroethylene (TCE)	5	**5.3**	
trichlorofluoromethane	5	0.2	*U*
vinyl chloride (VC)	2	0.2	*U*
xylenes, total	5	0.94	*J*

Detected concentrations

Concentrations above AWQS

Notes: AWQS based on NYSDEC TOGS 1.1.1 (Class GA) NA = not available

Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Table 4: VOCs in Soil Vapor

WCD File: PQ18052.21

Sample ID	SV-01		SV-02		SV-03		SV-04	
All data in µg/m³ Sample Date	(2018-08-21)		(2018-08-21)		(2018-08-21)		(2018-08-21)	
U= Not Detected ≥ value Dilution Factor	1.383		14.06		13.58		27.34	
VOCs, TO-15	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*
1,1,1,2-Tetrachloroethane	0.95	*U*	0.97	*U*	0.93	*U*	0.94	*U*
1,1,1-Trichloroethane	4.7	*D*	0.77	*U*	27	*D*	620	*D*
1,1,2,2-Tetrachloroethane	0.95	*U*	0.97	*U*	0.93	*U*	0.94	*U*
1,1,2-Trichloro-1,2,2-trifluoroethane	1.1	*U*	1.1	*U*	1	*U*	1	*U*
1,1,2-Trichloroethane	0.76	*U*	0.77	*U*	0.74	*U*	0.75	*U*
1,1-Dichloroethane	0.56	*U*	0.57	*U*	0.55	*U*	7.8	*D*
1,1-Dichloroethene	0.55	*U*	0.56	*U*	0.54	*U*	0.54	*U*
1,2,4-Trichlorobenzene	1	*U*	1	*U*	1	*U*	1	*U*
1,2,4-Trimethylbenzene	29	*D*	110	*D*	20	*D*	17	*D*
1,2-Dibromoethane	1.1	*U*	1.1	*U*	1	*U*	1.1	*U*
1,2-Dichlorobenzene	0.83	*U*	0.85	*U*	0.82	*U*	0.82	*U*
1,2-Dichloroethane	0.56	*U*	0.57	*U*	0.55	*U*	0.55	*U*
1,2-Dichloropropane	0.64	*U*	0.65	*U*	0.63	*U*	0.63	*U*
1,2-Dichlorotetrafluoroethane	0.97	*U*	0.98	*U*	0.95	*U*	0.96	*U*
1,3,5-Trimethylbenzene	8.9	*D*	28	*D*	4.9	*D*	3.7	*D*
1,3-Butadiene	2	*D*	0.93	*U*	0.9	*U*	0.91	*U*
1,3-Dichlorobenzene	0.83	*U*	0.85	*U*	0.82	*U*	0.82	*U*
1,3-Dichloropropane	0.64	*U*	0.65	*U*	0.63	*U*	0.63	*U*
1,4-Dichlorobenzene	0.83	*U*	0.85	*U*	0.82	*U*	0.82	*U*
1,4-Dioxane	1	*U*	1	*U*	0.98	*U*	0.99	*U*
2-Butanone	28	*D*	73	*D*	6.1	*D*	3.7	*D*
2-Hexanone	3	*D*	8.8	*D*	1.1	*U*	1.1	*U*
3-Chloropropene	2.2	*U*	2.2	*U*	2.1	*U*	2.1	*U*
4-Methyl-2-pentanone	5.5	*D*	9.6	*D*	0.56	*U*	0.56	*U*
Acetone	160	*D*	370	*D*	32	*D*	18	*D*
Acrylonitrile	0.3	*U*	0.31	*U*	0.29	*U*	0.3	*U*
Benzene	11	*D*	6.7	*D*	9.2	*D*	6.7	*D*
Benzyl chloride	0.72	*U*	0.73	*U*	0.7	*U*	0.71	*U*
Bromodichloromethane	0.93	*U*	0.94	*U*	0.91	*U*	0.92	*U*
Bromoform	1.4	*U*	1.5	*U*	1.4	*U*	1.4	*U*
Bromomethane	0.54	*U*	0.55	*U*	0.53	*U*	0.53	*U*
Carbon disulfide	3.5	*D*	0.44	*U*	0.42	*U*	0.43	*U*
Carbon tetrachloride	0.22	*U*	0.22	*U*	0.21	*U*	0.22	*U*
Chlorobenzene	0.64	*U*	0.65	*U*	0.63	*U*	0.63	*U*
Chloroethane	0.37	*U*	0.37	*U*	0.36	*U*	0.36	*U*
Chloroform	0.68	*U*	0.69	*U*	35	*D*	86	*D*
Chloromethane	3.1	*D*	10	*D*	0.28	*U*	0.28	*U*
cis-1,2-Dichloroethene	0.55	*U*	0.56	*U*	0.54	*U*	0.54	*U*
cis-1,3-Dichloropropene	0.63	*U*	0.64	*U*	0.62	*U*	0.62	*U*
Cyclohexane	14	*D*	51	*D*	6.9	*D*	0.47	*U*
Dibromochloromethane	1.2	*U*	1.2	*U*	1.2	*U*	1.2	*U*
Dichlorodifluoromethane	0.69	*U*	0.7	*U*	0.67	*U*	0.68	*U*
Ethyl Acetate	3.9	*D*	5.6	*D*	0.98	*U*	0.99	*U*
Ethylbenzene	30	*D*	25	*D*	24	*D*	19	*D*
Hexachlorobutadiene	1.5	*U*	1.5	*U*	1.4	*U*	1.5	*U*
Isopropanol	4.7	*D*	17	*D*	3.1	*D*	3.9	*D*
Methyl Methacrylate	0.57	*U*	0.58	*U*	0.56	*U*	0.56	*U*
Methyl tert butyl ether	0.5	*U*	0.51	*U*	0.49	*U*	0.49	*U*
Methylene chloride	4.8	*D*	4.8	*D*	0.94	*U*	0.95	*U*
Naphthalene	9.4	*D*	9.8	*D*	8.9	*D*	7.3	*D*
n-Heptane	13	*D*	40	*D*	12	*D*	12	*D*
n-Hexane	6.6	*D*	6	*D*	4.5	*D*	6.6	*D*
o-Xylene	31	*D*	35	*D*	22	*D*	18	*D*
p/m-Xylene	90	*D*	91	*D*	67	*D*	53	*D*
p-Ethyltoluene	31	*D*	100	*D*	19	*D*	16	*D*
Propylene	11	*D*	11	*D*	2.9	*D*	2.4	*D*
Styrene	4.5	*D*	6.5	*D*	3.6	*D*	0.58	*U*
Tetrachloroethene	8.4	*D*	7.4	*D*	2,700	*D*	1,700	*D*
Tetrahydrofuran	20	*D*	30	*D*	5	*D*	4.4	*D*
Toluene	110	*D*	74	*D*	90	*D*	68	*D*
trans-1,2-Dichloroethene	0.55	*U*	0.56	*U*	0.54	*U*	0.54	*U*
trans-1,3-Dichloropropene	0.63	*U*	0.64	*U*	0.62	*U*	0.62	*U*
Trichloroethene	0.19	*U*	0.19	*U*	10	*D*	5.3	*D*
Trichlorofluoromethane	0.78	*U*	0.79	*U*	4.9	*D*	0.77	*U*
Vinyl acetate	0.49	*U*	0.5	*U*	0.48	*U*	0.48	*U*
Vinyl bromide	0.61	*U*	0.62	*U*	0.59	*U*	0.6	*U*
Vinyl chloride	0.35	*U*	0.36	*U*	0.35	*U*	0.35	*U*

Detected concentrations
Relatively elevated concentrations

Notes: NA = not available
Result Qualifiers: J = approximate E = estimated B = detected in blank

Table 5: Petroleum Compounds in Soils

NYSDEC PBS No. 2-612992

File: PQ18052

GALLAGHER BASSETT | TECHNICAL SERVICES

All data in mg/Kg (ppm)			**Sample ID**	**UST-01 N**		**UST-01 S**		**UST-01 NW**		**UST-01 NE**	
U= Not Detected ≥ indicated value			Sample Date	6/11/2019		6/11/2019		6/11/2019		6/11/2019	
Data above SCOs shown in **Bold**			Dilution Factor	1		1		1		1	
VOCs, 8260	**UUSCO**	**RRUSCO**		*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*
1,2,4-Trimethylbenzene	3.6	52		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
1,3,5-Trimethylbenzene	8.4	52		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
Benzene	0.06	4.8		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
Ethyl Benzene	1	41		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
Isopropylbenzene	2.3	100		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
Methyl tert-butyl ether (MTBE)	0.93	100		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
Naphthalene	12	100		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
n-Butylbenzene	12	100		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
n-Propylbenzene	3.9	100		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
o-Xylene	0.26	100		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
p- & m- Xylenes	0.26	100		0.0059	*U*	0.01	*U*	0.0062	*U*	0.0054	*U*
p-Isopropyltoluene	10	NA		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
sec-Butylbenzene	11	100		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
tert-Butylbenzene	5.9	100		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
Toluene	0.7	100		0.003	*U*	0.0052	*U*	0.0031	*U*	0.0027	*U*
Xylenes, Total	0.26	100		0.0089	*U*	0.016	*U*	0.0093	*U*	0.0081	*U*

			Sample ID	**UST-01 N**		**UST-01 S**		**UST-01 NW**		**UST-01 NE**	
			Sample Date	6/11/2019		6/11/2019		6/11/2019		6/11/2019	
			Dilution Factor	20		2		2		5	
SVOCs, 8270	**UUSCO**	**RRUSCO**		*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*
Acenaphthene	20	100		0.9	*D*	0.17	*D*	0.11	*D*	0.17	*D*
Acenaphthylene	100	100		1	*D*	0.1	*D*	0.081	*JD*	0.2	*D*
Anthracene	100	100		2.8	*D*	0.49	*D*	0.32	*D*	0.68	*D*
Benzo(a)anthracene	1	1		**10**	*D*	**1.6**	*D*	0.87	*D*	**1.9**	*D*
Benzo(a)pyrene	1	1		**8.4**	*D*	**1.5**	*D*	0.77	*D*	**1.7**	*D*
Benzo(b)fluoranthene	1	1		**7.7**	*D*	**1.3**	*D*	0.73	*D*	**1.6**	*D*
Benzo(g,h,i)perylene	100	100		5	*D*	0.9	*D*	0.48	*D*	1.1	*D*
Benzo(k)fluoranthene	0.8	3.9		**7.6**	*D*	1.3	*D*	0.63	*D*	1.5	*D*
Chrysene	1	3.9		**10**	*D*	1.6	*D*	0.85	*D*	1.8	*D*
Dibenzo(a,h)anthracene	0.33	0.33		**1.9**	*D*	0.3	*D*	0.18	*D*	**0.4**	*D*
Fluoranthene	100	100		20	*D*	3.8	*D*	2.1	*D*	5.1	*D*
Fluorene	30	100		1.3	*D*	0.17	*D*	0.11	*D*	0.23	*D*
Indeno(1,2,3-cd)pyrene	0.5	0.5		**6**	*D*	**1**	*D*	**0.55**	*D*	**1.3**	*D*
Naphthalene	12	100		0.53	*D*	0.065	*JD*	0.065	*JD*	0.05	*JD*
Phenanthrene	100	100		14	*D*	2.3	*D*	1.6	*D*	2.9	*DE*
pyrene	100	100		18	*D*	3.3	*D*	1.8	*D*	4.2	*D*

Analyte Detected

Analyte Above UUSCO

Analyte Above RRUSCO

Notes: SCOs based on NYSDEC Part 375-6.8 and CP-51 NA = not available

Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Table 5: Petroleum Compounds in Soils

NYSDEC PBS No. 2-612992

File: PQ18052

GALLAGHER BASSETT | TECHNICAL SERVICES

All data in mg/Kg (ppm)			**Sample ID**	**UST-01 SW**		**UST-01 SE**		**UST-01 BN**		**UST-01 BS**	
U= Not Detected ≥ indicated value			Sample Date	6/11/2019		6/11/2019		6/11/2019		6/11/2019	
Data above SCOs shown in **Bold**			Dilution Factor	1		1		1		1	
VOCs, 8260	**UUSCO**	**RRUSCO**		*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*
1,2,4-Trimethylbenzene	3.6	52		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
1,3,5-Trimethylbenzene	8.4	52		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
Benzene	0.06	4.8		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
Ethyl Benzene	1	41		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
Isopropylbenzene	2.3	100		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
Methyl tert-butyl ether (MTBE)	0.93	100		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
Naphthalene	12	100		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
n-Butylbenzene	12	100		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
n-Propylbenzene	3.9	100		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
o-Xylene	0.26	100		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
p- & m- Xylenes	0.26	100		0.0084	*U*	0.0058	*U*	0.0061	*U*	0.006	*U*
p-Isopropyltoluene	10	NA		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
sec-Butylbenzene	11	100		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
tert-Butylbenzene	5.9	100		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
Toluene	0.7	100		0.0042	*U*	0.0029	*U*	0.0031	*U*	0.003	*U*
Xylenes, Total	0.26	100		0.013	*U*	0.0087	*U*	0.0092	*U*	0.009	*U*

			Sample ID	**UST-01 SW**		**UST-01 SE**		**UST-01 BN**		**UST-01 BS**	
			Sample Date	6/11/2019		6/11/2019		6/11/2019		6/11/2019	
			Dilution Factor	2		5		2		2	
SVOCs, 8270	**UUSCO**	**RRUSCO**		*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*	*Result*	*Qualifier*
Acenaphthene	20	100		0.045	*U*	0.2	*D*	0.045	*U*	0.049	*U*
Acenaphthylene	100	100		0.045	*U*	0.12	*D*	0.045	*U*	0.049	*U*
Anthracene	100	100		0.045	*U*	0.6	*D*	0.045	*U*	0.049	*U*
Benzo(a)anthracene	1	1		0.045	*U*	**1.8**	*D*	0.045	*U*	0.049	*U*
Benzo(a)pyrene	1	1		0.045	*U*	**1.7**	*D*	0.045	*U*	0.049	*U*
Benzo(b)fluoranthene	1	1		0.045	*U*	**1.6**	*D*	0.045	*U*	0.049	*U*
Benzo(g,h,i)perylene	100	100		0.045	*U*	1	*D*	0.045	*U*	0.13	*D*
Benzo(k)fluoranthene	0.8	3.9		0.045	*U*	1.3	*D*	0.045	*U*	0.049	*U*
Chrysene	1	3.9		0.045	*U*	1.7	*D*	0.045	*U*	0.049	*U*
Dibenzo(a,h)anthracene	0.33	0.33		0.045	*U*	**0.41**	*D*	0.045	*U*	0.049	*U*
Fluoranthene	100	100		0.045	*U*	4.5	*D*	0.045	*U*	0.049	*U*
Fluorene	30	100		0.045	*U*	0.21	*D*	0.045	*U*	0.049	*U*
Indeno(1,2,3-cd)pyrene	0.5	0.5		0.045	*U*	**1.2**	*D*	0.045	*U*	0.13	*D*
Naphthalene	12	100		0.045	*JD*	0.079	*JD*	0.045	*U*	0.049	*U*
Phenanthrene	100	100		0.045	*U*	2.5	*D*	0.045	*U*	0.049	*U*
pyrene	100	100		0.045	*U*	3.8	*D*	0.045	*U*	0.049	*U*

Analyte Detected

Analyte Above UUSCO

Analyte Above RRUSCO

Notes: SCOs based on NYSDEC Part 375-6.8 and CP-51 NA = not available

Result Qualifiers: J = approximate E = estimated B = detected in blank D = diluted

Notes Utilized Throughout Tables
Soil Vapor/Ambient Air
J - Estimated value
U - The analyte was analyzed for, but was not detected above the level of the associated reported quantitation limit
ug/m3 - Micrograms per cubic meter
Bold data indicates that parameter was detected

Table 1. Summary of Volatile Organic Compounds in Soil Vapor, 11-24 Wyckoff Avenue, Queens, New York

Sample Designation:		OA-1	SSMP-1	SSMP-1_IA	SSMP-2	SSMP-2_IA	SSMP-3	SSMP-3_IA	SSMP-4	SSMP-4_IA
Sample Date:		05/03/2023	05/03/2023	05/03/2023	05/03/2023	05/03/2023	05/03/2023	05/03/2023	05/03/2023	05/03/2023
Parameter	Units									
1,1,1-Trichloroethane (TCA)	UG/M3	0.109 U	1.09 U	0.109 U	1.09 U	0.109 U	1.09 U	0.109 U	1.09 U	0.109 U
1,1,2,2-Tetrachloroethane	UG/M3	1.37 U	1.37 U	1.37 U	1.37 U	1.37 U	1.37 U	1.37 U	1.37 U	1.37 U
1,1,2-Trichloro-1,2,2-Trifluoroethane	UG/M3	1.53 U	1.53 U	1.53 U	1.53 U	1.53 U	1.53 U	1.53 U	1.53 U	1.53 U
1,1,2-Trichloroethane	UG/M3	1.09 U	1.09 U	1.09 U	1.09 U	1.09 U	1.09 U	1.09 U	1.09 U	1.09 U
1,1-Dichloroethane	UG/M3	0.809 U	0.809 U	0.809 U	0.809 U	0.809 U	0.809 U	0.809 U	0.809 U	0.809 U
1,1-Dichloroethene	UG/M3	0.079 U	0.793 U	0.079 U	0.793 U	0.079 U	0.793 U	0.079 U	0.793 U	0.079 U
1,2,4-Trichlorobenzene	UG/M3	1.48 U	1.48 U	1.48 U	1.48 U	1.48 U	1.48 U	1.48 U	1.48 U	1.48 U
1,2,4-Trimethylbenzene	UG/M3	0.983 U	**1.4**	**1.31**	0.983 U	**1.95**	0.983 U	**1.74**	0.983 U	**1.96**
1,2-Dibromoethane (Ethylene Dibromide)	UG/M3	1.54 U	1.54 U	1.54 U	1.54 U	1.54 U	1.54 U	1.54 U	1.54 U	1.54 U
1,2-Dichlorobenzene	UG/M3	1.2 U	1.2 U	1.2 U	1.2 U	1.2 U	1.2 U	1.2 U	1.2 U	1.2 U
1,2-Dichloroethane	UG/M3	0.809 U	0.809 U	0.809 U	0.809 U	0.809 U	0.809 U	0.809 U	0.809 U	0.809 U
1,2-Dichloropropane	UG/M3	0.924 U	0.924 U	0.924 U	0.924 U	0.924 U	0.924 U	0.924 U	0.924 U	0.924 U
1,2-Dichlorotetrafluoroethane	UG/M3	1.4 U	1.4 U	1.4 U	1.4 U	1.4 U	1.4 U	1.4 U	1.4 U	1.4 U
1,3,5-Trimethylbenzene (Mesitylene)	UG/M3	0.983 U	0.983 U	0.983 U	0.983 U	0.983 U	0.983 U	0.983 U	0.983 U	0.983 U
1,3-Butadiene	UG/M3	0.442 U	0.442 U	0.442 U	0.442 U	0.442 U	0.442 U	0.442 U	0.442 U	0.442 U
1,3-Dichlorobenzene	UG/M3	1.2 U	1.2 U	1.2 U	1.2 U	1.2 U	1.2 U	1.2 U	**1.51**	1.2 U
1,4-Dichlorobenzene	UG/M3	1.2 U	1.2 U	1.2 U	1.2 U	1.2 U	1.2 U	1.2 U	1.2 U	1.2 U
1,4-Dioxane (P-Dioxane)	UG/M3	0.721 U	0.721 U	0.721 U	0.721 U	0.721 U	0.721 U	0.721 U	0.721 U	0.721 U
2,2,4-Trimethylpentane	UG/M3	0.934 U	**0.943**	0.934 U	0.934 U	0.934 U	0.934 U	0.934 U	0.934 U	0.934 U
2-Hexanone	UG/M3	0.82 U	0.82 U	0.82 U	0.82 U	0.82 U	0.82 U	0.82 U	0.82 U	0.82 U
4-Ethyltoluene	UG/M3	0.983 U	0.983 U	0.983 U	0.983 U	0.983 U	0.983 U	0.983 U	0.983 U	0.983 U
Acetone	UG/M3	**10.1**	**12.3**	**41.8**	**41.8**	**58.7**	**8.72**	**53.7**	**41.8**	**56.1**
Allyl Chloride (3-Chloropropene)	UG/M3	0.626 U	0.626 U	0.626 U	0.626 U	0.626 U	0.626 U	0.626 U	0.626 U	0.626 U
Benzene	UG/M3	**0.776**	**0.671**	0.639 U	**0.722**	0.639 U	0.639 U	0.639 U	0.639 U	**0.716**
Benzyl Chloride	UG/M3	1.04 U	1.04 U	1.04 U	1.04 U	1.04 U	1.04 U	1.04 U	1.04 U	1.04 U
Bromodichloromethane	UG/M3	1.34 U	1.34 U	1.34 U	1.34 U	1.34 U	1.34 U	1.34 U	1.34 U	1.34 U
Bromoform	UG/M3	2.07 U	2.07 U	2.07 U	2.07 U	2.07 U	2.07 U	2.07 U	2.07 U	2.07 U
Bromomethane	UG/M3	0.777 U	0.777 U	0.777 U	0.777 U	0.777 U	0.777 U	0.777 U	0.777 U	0.777 U
Carbon Disulfide	UG/M3	0.623 U	0.623 U	0.623 U	**0.722**	0.623 U	**1.76**	0.623 U	0.623 U	0.623 U
Carbon Tetrachloride	UG/M3	**0.459**	1.26 U	**0.484**	1.26 U	**0.465**	1.26 U	**0.478**	1.26 U	**0.465**
Chlorobenzene	UG/M3	0.921 U	0.921 U	0.921 U	0.921 U	0.921 U	0.921 U	0.921 U	0.921 U	0.921 U
Chloroethane	UG/M3	0.528 U	0.528 U	0.528 U	0.528 U	0.528 U	0.528 U	0.528 U	0.528 U	0.528 U
Chloroform	UG/M3	0.977 U	0.977 U	0.977 U	0.977 U	0.977 U	0.977 U	0.977 U	0.977 U	0.977 U
Chloromethane	UG/M3	**1.08**	**1.26**	**1.1**	**1.05**	**1.16**	0.413 U	**1.08**	**1.06**	**1.15**
Cis-1,2-Dichloroethylene	UG/M3	**0.083**	0.793 U	0.079 U	0.793 U	0.079 U	0.793 U	0.079 U	0.793 U	0.079 U
Cis-1,3-Dichloropropene	UG/M3	0.908 U	0.908 U	0.908 U	0.908 U	0.908 U	0.908 U	0.908 U	0.908 U	0.908 U
Cyclohexane	UG/M3	0.688 U	0.688 U	0.688 U	0.688 U	0.688 U	0.688 U	0.688 U	0.688 U	0.688 U
Dibromochloromethane	UG/M3	1.7 U	1.7 U	1.7 U	1.7 U	1.7 U	1.7 U	1.7 U	1.7 U	1.7 U

Table 1. Summary of Volatile Organic Compounds in Soil Vapor, 11-24 Wyckoff Avenue, Queens, New York

Sample Designation:		OA-1	SSMP-1	SSMP-1_IA	SSMP-2	SSMP-2_IA	SSMP-3	SSMP-3_IA	SSMP-4	SSMP-4_IA
Sample Date:		05/03/2023	05/03/2023	05/03/2023	05/03/2023	05/03/2023	05/03/2023	05/03/2023	05/03/2023	05/03/2023
Parameter	Units									
Dichlorodifluoromethane	UG/M3	**2.33**	**2.35**	**2.37**	**2.33**	**2.39**	**2.32**	**2.28**	**2.31**	**2.31**
Ethanol	UG/M3	**14.9**	**25.8**	**13.6**	**39.8**	**15.8**	**71.8**	**31.1**	**44.1**	**54.3**
Ethyl Acetate	UG/M3	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U	1.8 U
Ethylbenzene	UG/M3	0.869 U	0.869 U	0.869 U	0.869 U	0.869 U	0.869 U	0.869 U	0.869 U	0.869 U
Hexachlorobutadiene	UG/M3	2.13 U	2.13 U	2.13 U	2.13 U	2.13 U	2.13 U	2.13 U	2.13 U	2.13 U
Isopropanol	UG/M3	**1.59**	**1.88**	**1.72**	**2.7**	**2.19**	**4.15**	**3.12**	**3.91**	**4.84**
m,p-Xylene	UG/M3	1.74 U	**2.52**	1.74 U	**2.24**	**1.75**	**2.68**	1.74 U	**2.51**	**2.15**
Methyl Ethyl Ketone (2-Butanone)	UG/M3	1.47 U	**3.51**	1.47 U	**2.17**	1.47 U	**1.63**	1.47 U	**1.49**	1.47 U
Methyl Isobutyl Ketone (4-Methyl-2-Pentanone)	UG/M3	2.05 U	2.05 U	2.05 U	**2.41**	2.05 U	**2.86**	2.05 U	2.05 U	2.05 U
Methylene Chloride	UG/M3	**1.81**	1.74 U	1.74 U	1.74 U	1.74 U	1.74 U	1.74 U	1.74 U	1.74 U
N-Heptane	UG/M3	0.82 U	0.82 U	0.82 U	0.82 U	0.82 U	0.82 U	0.82 U	0.82 U	0.82 U
N-Hexane	UG/M3	**1.33**	**0.839**	**0.807**	**0.733**	**0.899**	0.705 U	**1.13**	**0.708**	**1.74**
O-Xylene (1,2-Dimethylbenzene)	UG/M3	0.869 U	**1.17**	0.869 U	**0.921**	0.869 U	**1.08**	0.869 U	**1.16**	0.869 U
Styrene	UG/M3	0.852 U	0.852 U	0.852 U	0.852 U	0.852 U	0.852 U	0.852 U	0.852 U	0.852 U
Tert-Butyl Alcohol	UG/M3	1.52 U	1.52 U	1.52 U	**1.93**	1.52 U	1.52 U	1.52 U	1.52 U	1.52 U
Tert-Butyl Methyl Ether	UG/M3	0.721 U	0.721 U	0.721 U	0.721 U	0.721 U	0.721 U	0.721 U	0.721 U	0.721 U
Tetrachloroethylene (PCE)	UG/M3	**1.53**	1.36 U	**0.21**	1.36 U	**0.197**	1.36 U	**0.251**	1.36 U	**0.373**
Tetrahydrofuran	UG/M3	1.47 U	1.47 U	1.47 U	1.47 U	1.47 U	1.47 U	1.47 U	1.47 U	1.47 U
Toluene	UG/M3	**2.28**	**2.79**	**1.9**	**2.86**	**2.11**	**3.35**	**2.47**	**2.69**	**2.95**
Trans-1,2-Dichloroethene	UG/M3	0.793 U	0.793 U	0.793 U	0.793 U	0.793 U	0.793 U	0.793 U	0.793 U	0.793 U
Trans-1,3-Dichloropropene	UG/M3	0.908 U	0.908 U	0.908 U	0.908 U	0.908 U	0.908 U	0.908 U	0.908 U	0.908 U
Trichloroethylene (TCE)	UG/M3	**1.08**	1.07 U	0.107 U	1.07 U	0.107 U	1.07 U	0.107 U	1.07 U	0.107 U
Trichlorofluoromethane	UG/M3	**1.14**	**1.19**	**1.2**	**1.21**	**1.23**	**1.17**	**1.12**	**1.14**	**1.16**
Vinyl Bromide	UG/M3	0.874 U	0.874 U	0.874 U	0.874 U	0.874 U	0.874 U	0.874 U	0.874 U	0.874 U
Vinyl Chloride	UG/M3	0.051 U	0.511 U	0.051 U	0.511 U	0.051 U	0.511 U	0.051 U	0.511 U	0.051 U

Table 1. Summary of Volatile Organic Compounds in Soil Vapor, 11-24 Wyckoff Avenue, Queens, New York

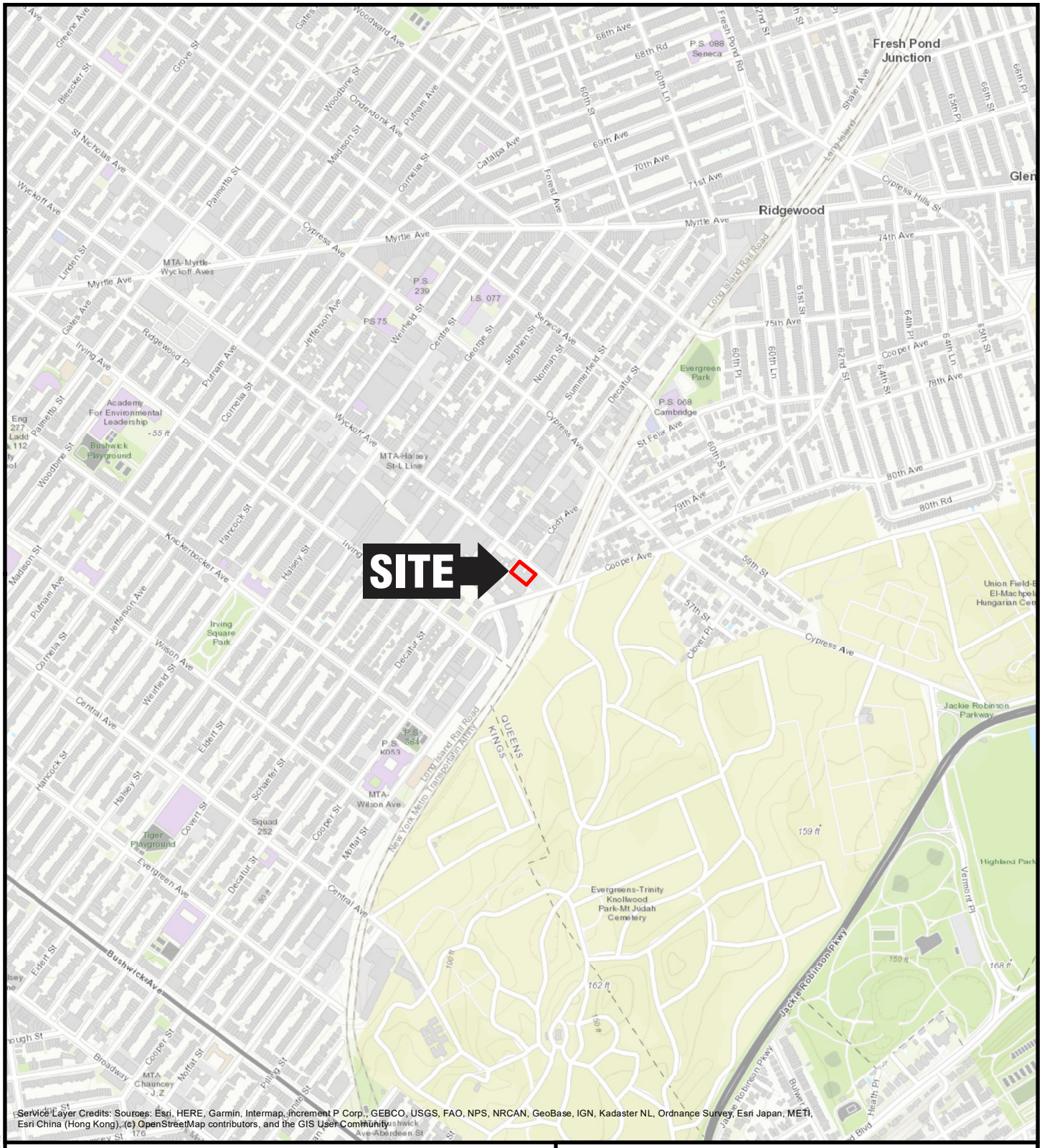
Sample Designation:		SSMP-7	SSMP-7_IA	SSMP-8	SSMP-8_IA
Sample Date:		05/03/2023	05/03/2023	05/03/2023	05/03/2023
Parameter	Units				
1,1,1-Trichloroethane (TCA)	UG/M3	**4.3**	0.24 U	**3.4**	0.109 U
1,1,2,2-Tetrachloroethane	UG/M3	1.37 U	0.302 U	1.37 U	1.37 U
1,1,2-Trichloro-1,2,2-Trifluoroethane	UG/M3	1.53 U	0.843 U	1.53 U	1.53 U
1,1,2-Trichloroethane	UG/M3	1.09 U	0.24 U	1.09 U	1.09 U
1,1-Dichloroethane	UG/M3	0.809 U	0.178 U	0.809 U	0.809 U
1,1-Dichloroethene	UG/M3	0.793 U	0.174 U	0.793 U	0.079 U
1,2,4-Trichlorobenzene	UG/M3	1.48 U	0.817 U	1.48 U	1.48 U
1,2,4-Trimethylbenzene	UG/M3	0.983 U	**2.12**	**3.97**	**2.32**
1,2-Dibromoethane (Ethylene Dibromide)	UG/M3	1.54 U	0.338 U	1.54 U	1.54 U
1,2-Dichlorobenzene	UG/M3	1.2 U	0.265 U	1.2 U	1.2 U
1,2-Dichloroethane	UG/M3	0.809 U	0.178 U	0.809 U	0.809 U
1,2-Dichloropropane	UG/M3	0.924 U	0.203 U	0.924 U	0.924 U
1,2-Dichlorotetrafluoroethane	UG/M3	1.4 U	0.769 U	1.4 U	1.4 U
1,3,5-Trimethylbenzene (Mesitylene)	UG/M3	0.983 U	**0.605**	**1.05**	0.983 U
1,3-Butadiene	UG/M3	0.442 U	0.097 U	0.442 U	0.442 U
1,3-Dichlorobenzene	UG/M3	**2.45**	0.265 U	1.2 U	1.2 U
1,4-Dichlorobenzene	UG/M3	1.2 U	0.265 U	1.2 U	1.2 U
1,4-Dioxane (P-Dioxane)	UG/M3	0.721 U	0.793 U	0.721 U	0.721 U
2,2,4-Trimethylpentane	UG/M3	0.934 U	2.06 U	0.934 U	0.934 U
2-Hexanone	UG/M3	**0.861**	1.8 U	0.82 U	0.82 U
4-Ethyltoluene	UG/M3	0.983 U	**0.551**	0.983 U	0.983 U
Acetone	UG/M3	**12.9**	**45.8**	**7.79**	**54.2**
Allyl Chloride (3-Chloropropene)	UG/M3	0.626 U	1.38 U	0.626 U	0.626 U
Benzene	UG/M3	0.639 U	**0.738**	0.639 U	**0.693**
Benzyl Chloride	UG/M3	1.04 U	1.14 U	1.04 U	1.04 U
Bromodichloromethane	UG/M3	1.34 U	0.295 U	1.34 U	1.34 U
Bromoform	UG/M3	2.07 U	0.455 U	2.07 U	2.07 U
Bromomethane	UG/M3	0.777 U	0.171 U	0.777 U	0.777 U
Carbon Disulfide	UG/M3	0.623 U	1.37 U	0.623 U	0.623 U
Carbon Tetrachloride	UG/M3	1.26 U	**0.54**	1.26 U	**0.453**
Chlorobenzene	UG/M3	0.921 U	1.01 U	0.921 U	0.921 U
Chloroethane	UG/M3	0.528 U	0.581 U	0.528 U	0.528 U
Chloroform	UG/M3	0.977 U	0.215 U	**1.62**	0.977 U
Chloromethane	UG/M3	0.413 U	**1.1**	0.413 U	**1.12**
Cis-1,2-Dichloroethylene	UG/M3	0.793 U	0.174 U	0.793 U	0.079 U
Cis-1,3-Dichloropropene	UG/M3	0.908 U	0.2 U	0.908 U	0.908 U
Cyclohexane	UG/M3	0.688 U	1.51 U	0.688 U	0.688 U
Dibromochloromethane	UG/M3	1.7 U	0.375 U	1.7 U	1.7 U

Table 1. Summary of Volatile Organic Compounds in Soil Vapor, 11-24 Wyckoff Avenue, Queens, New York

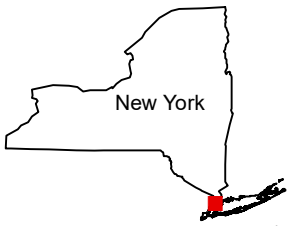
Sample Designation:		SSMP-7	SSMP-7_IA	SSMP-8	SSMP-8_IA
Sample Date:		05/03/2023	05/03/2023	05/03/2023	05/03/2023
Parameter	Units				
Dichlorodifluoromethane	UG/M3	**1.91**	**2.33**	0.989 U	**2.35**
Ethanol	UG/M3	**98.9**	**31.8**	**43.3**	**28.6**
Ethyl Acetate	UG/M3	1.8 U	3.96 U	1.8 U	1.8 U
Ethylbenzene	UG/M3	**1.02**	**0.556**	**1.52**	0.869 U
Hexachlorobutadiene	UG/M3	2.13 U	1.17 U	2.13 U	2.13 U
Isopropanol	UG/M3	**10.3**	**2.8**	**3.12**	**2.85**
m,p-Xylene	UG/M3	**3.4**	**1.82**	**5.43**	1.74 U
Methyl Ethyl Ketone (2-Butanone)	UG/M3	**2.34**	3.24 U	1.47 U	1.47 U
Methyl Isobutyl Ketone (4-Methyl-2-Pentanone)	UG/M3	**2.08**	4.51 U	2.05 U	2.05 U
Methylene Chloride	UG/M3	1.74 U	3.82 U	1.74 U	1.74 U
N-Heptane	UG/M3	0.82 U	1.8 U	**0.869**	0.82 U
N-Hexane	UG/M3	0.705 U	1.55 U	**1.07**	**1**
O-Xylene (1,2-Dimethylbenzene)	UG/M3	**1.58**	**0.747**	**2.54**	0.869 U
Styrene	UG/M3	0.852 U	0.187 U	0.852 U	0.852 U
Tert-Butyl Alcohol	UG/M3	**2.51**	3.33 U	1.52 U	1.52 U
Tert-Butyl Methyl Ether	UG/M3	0.721 U	1.59 U	0.721 U	0.721 U
Tetrachloroethylene (PCE)	UG/M3	**1.57**	0.298 U	**18.6**	**0.244**
Tetrahydrofuran	UG/M3	1.47 U	3.24 U	1.47 U	1.47 U
Toluene	UG/M3	**3.06**	**2.25**	**6.82**	**2.26**
Trans-1,2-Dichloroethene	UG/M3	0.793 U	0.174 U	0.793 U	0.793 U
Trans-1,3-Dichloropropene	UG/M3	0.908 U	0.2 U	0.908 U	0.908 U
Trichloroethylene (TCE)	UG/M3	**17**	0.236 U	1.07 U	0.107 U
Trichlorofluoromethane	UG/M3	**1.21**	**1.17**	**1.84**	**1.17**
Vinyl Bromide	UG/M3	0.874 U	1.92 U	0.874 U	0.874 U
Vinyl Chloride	UG/M3	0.511 U	0.112 U	0.511 U	0.051 U

FIGURES

1. Site Location Map
2. Tax Map
3. Existing Site Plan
4. Historical Soil and Groundwater Exceedances and Soil Vapor Detections
5. Sub-Slab Depressurization System Layout and Detections in Soil Vapor and Indoor Air
6. Proposed Sample Locations



QUADRANGLE LOCATION



N

1,000 0 1,000'

Title:

SITE LOCATION MAP

11-24 WYCKOFF AVENUE, QUEENS, NEW YORK

Prepared for:

1124 WYCKOFF LLC

ROUX

Compiled by: S.S.	Date: 04/03/23
Prepared by: M.S.R.	Scale: AS SHOWN
Project Mgr: S.S.	Project: 4167.0001Y002
File: 4167.0001Y102.1.mxd	

FIGURE 1

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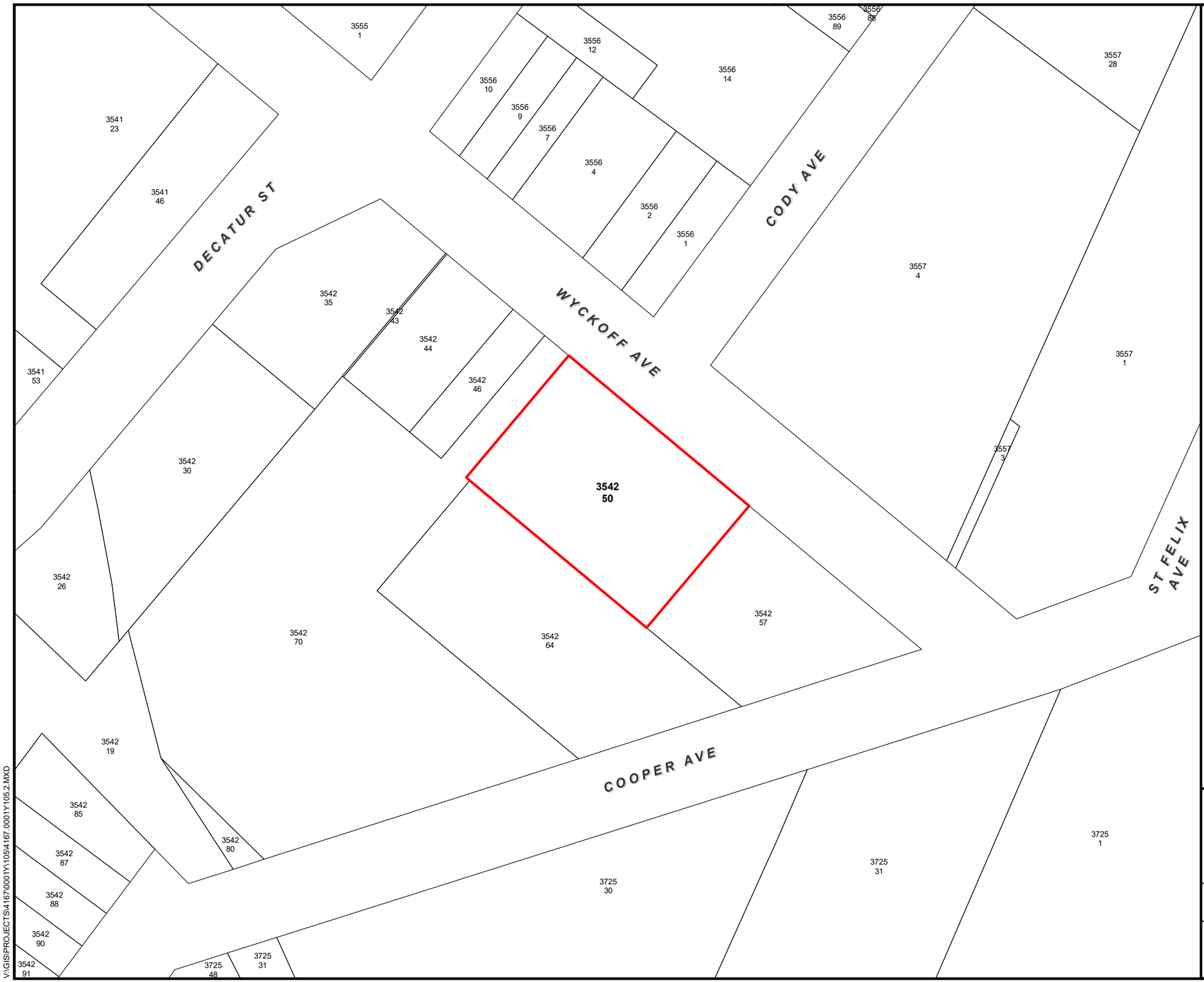

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N

LEGEND

- TAX PARCEL BOUNDARY
- SITE BOUNDARY
- 3542 BLOCK
- 50 LOT

NOTES

1. TAX PARCEL SOURCE: DEPARTMENT OF CITY PLANNING

60 0 60'

Title:

TAX MAP

11-24 WYCKOFF AVENUE
QUEENS, NEW YORK

Prepared for:

1124 WYCKOFF LLC

ROUX

Compiled by: V.S.	Date: 08/28/2024	FIGURE
Prepared by: M.S.R.	Scale: AS SHOWN	2
Project Mgr: V.S.	Project: 4167.0001Y000	
File: 4167.0001Y105.2.mxd		

N

LEGEND

SITE BOUNDARY

80 0 80'

Title:

SITE PLAN

11-24 WYCKOFF AVENUE, QUEENS, NEW YORK

Prepared for:

1124 WYCKOFF LLC

ROUX

Compiled by: A.N.	Date: 03/02/23	FIGURE
Prepared by: M.S.R.	Scale: AS SHOWN	3
Project Mgr: A.N.	Project: 4167.0001Y000	
File: 4167.0001Y101.2.mxd		

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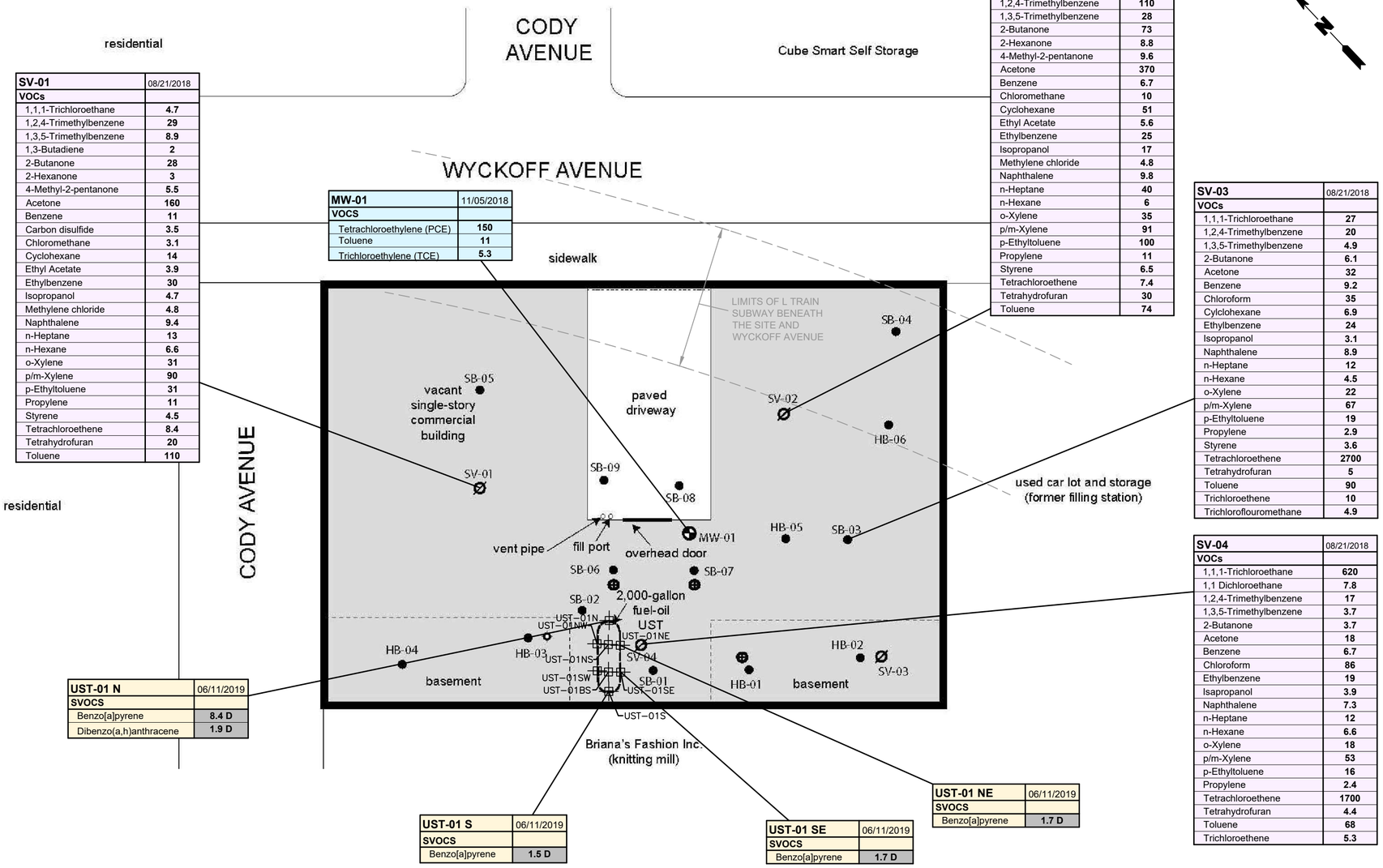


SV-01	08/21/2018
VOCs	
1,1,1-Trichloroethane	4.7
1,2,4-Trimethylbenzene	29
1,3,5-Trimethylbenzene	8.9
1,3-Butadiene	2
2-Butanone	28
2-Hexanone	3
4-Methyl-2-pentanone	5.5
Acetone	160
Benzene	11
Carbon disulfide	3.5
Chloromethane	3.1
Cyclohexane	14
Ethyl Acetate	3.9
Ethylbenzene	30
Isopropanol	4.7
Methylene chloride	4.8
Naphthalene	9.4
n-Heptane	13
n-Hexane	6.6
o-Xylene	31
p/m-Xylene	90
p-Ethyltoluene	31
Propylene	11
Styrene	4.5
Tetrachloroethene	8.4
Tetrahydrofuran	20
Toluene	110

MW-01	11/05/2018
VOCS	
Tetrachloroethylene (PCE)	150
Toluene	11
Trichloroethylene (TCE)	5.3

SV-02	08/21/2018
VOCs	
1,2,4-Trimethylbenzene	110
1,3,5-Trimethylbenzene	28
2-Butanone	73
2-Hexanone	8.8
4-Methyl-2-pentanone	9.6
Acetone	370
Benzene	6.7
Chloromethane	10
Cyclohexane	51
Ethyl Acetate	5.6
Ethylbenzene	25
Isopropanol	17
Methylene chloride	4.8
Naphthalene	9.8
n-Heptane	40
n-Hexane	6
o-Xylene	35
p/m-Xylene	91
p-Ethyltoluene	100
Propylene	11
Styrene	6.5
Tetrachloroethene	7.4
Tetrahydrofuran	30
Toluene	74

SV-03	08/21/2018
VOCs	
1,1,1-Trichloroethane	27
1,2,4-Trimethylbenzene	20
1,3,5-Trimethylbenzene	4.9
2-Butanone	6.1
Acetone	32
Benzene	9.2
Chloroform	35
Cylclohexane	6.9
Ethylbenzene	24
Isopropanol	3.1
Naphthalene	8.9
n-Heptane	12
n-Hexane	4.5
o-Xylene	22
p/m-Xylene	67
p-Ethyltoluene	19
Propylene	2.9
Styrene	3.6
Tetrachloroethene	2700
Tetrahydrofuran	5
Toluene	90
Trichloroethene	10
Trichloroflouromethane	4.9

SV-04	08/21/2018
VOCs	
1,1,1-Trichloroethane	620
1,1 Dichloroethane	7.8
1,2,4-Trimethylbenzene	17
1,3,5-Trimethylbenzene	3.7
2-Butanone	3.7
Acetone	18
Benzene	6.7
Chloroform	86
Ethylbenzene	19
Isapropanol	3.9
Naphthalene	7.3
n-Heptane	12
n-Hexane	6.6
o-Xylene	18
p/m-Xylene	53
p-Ethyltoluene	16
Propylene	2.4
Tetrachloroethene	1700
Tetrahydrofuran	4.4
Toluene	68
Trichloroethene	5.3

UST-01 N	06/11/2019
SVOCS	
Benzo[a]pyrene	8.4 D
Dibenzo(a,h)anthracene	1.9 D

UST-01 S	06/11/2019
SVOCS	
Benzo[a]pyrene	1.5 D

UST-01 SE	06/11/2019
SVOCS	
Benzo[a]pyrene	1.7 D

UST-01 NE	06/11/2019
SVOCS	
Benzo[a]pyrene	1.7 D

Legend:
- subject property border
- chain link fence
- sump
- floor drain
- boring locations
- soil vapor locations
- monitoring well location
- UST side/bottom sample

All feature locations are approximate. This map is intended as a schematic to be used in conjunction with the associated report, and it should not be relied upon as a survey for planning or other activities.

SOIL DATA NOTES

Parameter	Standards* (mg/kg)
SVOCs	
Benzo[a]pyrene	1.1
Dibenzo(a,h)anthracene	1.1

Concentrations in mg/kg
mg/kg -Milligrams per kilogram
*NYSDEC Part 375 Industrial Soil Cleanup Objectives
NYSDEC - New York State Department of Environmental Conservation
SVOCs - Semivolatile Organic Compounds
D - Diluted
- Results shown in bold black with gray background exceed NYSDEC Part 375 Industrial Soil Cleanup Objectives

GROUNDWATER DATA NOTES

Parameter	Standards* (µg/L)
VOCS	
Tetrachloroethylene (PCE)	5
Toluene	5
Trichloroethylene (TCE)	5

Concentrations in µg/L
µg/L - Micrograms per liter
* - NYSDEC AWQSGVs
NYSDEC - New York State Department of Environmental Conservation
AWQSGVs - Ambient Water Quality Standards and Guidance Values
VOCs - Volatile Organic Compounds

SOIL VAPOR DATA NOTES

Concentrations in µg/m^3
µg/m^3 - Micrograms per cubic meter
VOCs - Volatile Organic Compounds

TYPICAL DATA BOX INFORMATION

SAMPLE ID# →	MW-01	11/05/2018	← SAMPLE DATE
ANALYTES →	VOCS		
	Tetrachloroethylene (PCE)	150	← CONCENTRATIONS
	Toluene	11	
	Trichloroethylene (TCE)	5.3	

20' 0 20'

Title:
HISTORICAL SOIL AND GROUNDWATER EXCEEDANCES AND SOIL VAPOR DETECTIONS

11-24 WYCKOFF AVENUE
QUEENS, NEW YORK

Prepared for:
1124 WYCKOFF LLC

ROUX

Compiled by: V.S.	Date: 9/9/2024	FIGURE 4
Prepared by: B.H.C.	Scale: AS SHOWN	
Project Mgr: V.S.	Project: 4167.0001Y003	
File: 4167.0001Y106.02.DWG		

V:\CAD\PROJECTS\4167Y\0001Y\106\4167.0001Y106.02.DWG BCIC/O

SSMP-1/SSMP-1_IA	05/03/2023	05/03/2023
	SSMP-1	SSMP-1_IA
VOCs		
1,2,4-Trimethylbenzene	**1.4**	**1.31**
2,2,4-Trimethylpentane	**0.943**	ND
Acetone	**12.3**	**41.8**
Benzene	**0.671**	ND
Carbon Tetrachloride	ND	**0.484**
Chloromethane	**1.26**	**1.1**
Dichlorodifluoromethane	**2.35**	**2.37**
Ethanol	**25.8**	**13.6**
Isopropanol	**1.88**	**1.72**
m,p-Xylene	**2.52**	ND
Methyl Ethyl Ketone (2-Butanone)	**3.51**	ND
N-Hexane	**0.839**	**0.807**
O-Xylene (1,2-Dimethylbenzene)	**1.17**	ND
Tetrachloroethylene (PCE)	ND	**0.21**
Toluene	**2.79**	**1.9**
Trichlorofluoromethane	**1.19**	**1.2**

SSMP-3/SSMP-3_IA	05/03/2023	05/03/2023
	SSMP-3	SSMP-3_IA
VOCs		
1,2,4-Trimethylbenzene	ND	**1.74**
Acetone	**8.72**	**53.7**
Carbon Disulfide	**1.76**	ND
Carbon Tetrachloride	ND	**0.478**
Chloromethane	ND	**1.08**
Dichlorodifluoromethane	**2.32**	**2.28**
Ethanol	**71.8**	**31.1**
Isopropanol	**4.15**	**3.12**
m,p-Xylene	**2.68**	ND
Methyl Ethyl Ketone (2-Butanone)	**1.63**	ND
Methyl Isobutyl Ketone (4-Methyl-2-Pentanone)	**2.86**	ND
N-Hexane	ND	**1.13**
O-Xylene (1,2-Dimethylbenzene)	**1.08**	ND
Tetrachloroethylene (PCE)	ND	**0.251**
Toluene	**3.35**	**2.47**
Trichlorofluoromethane	**1.17**	**1.12**

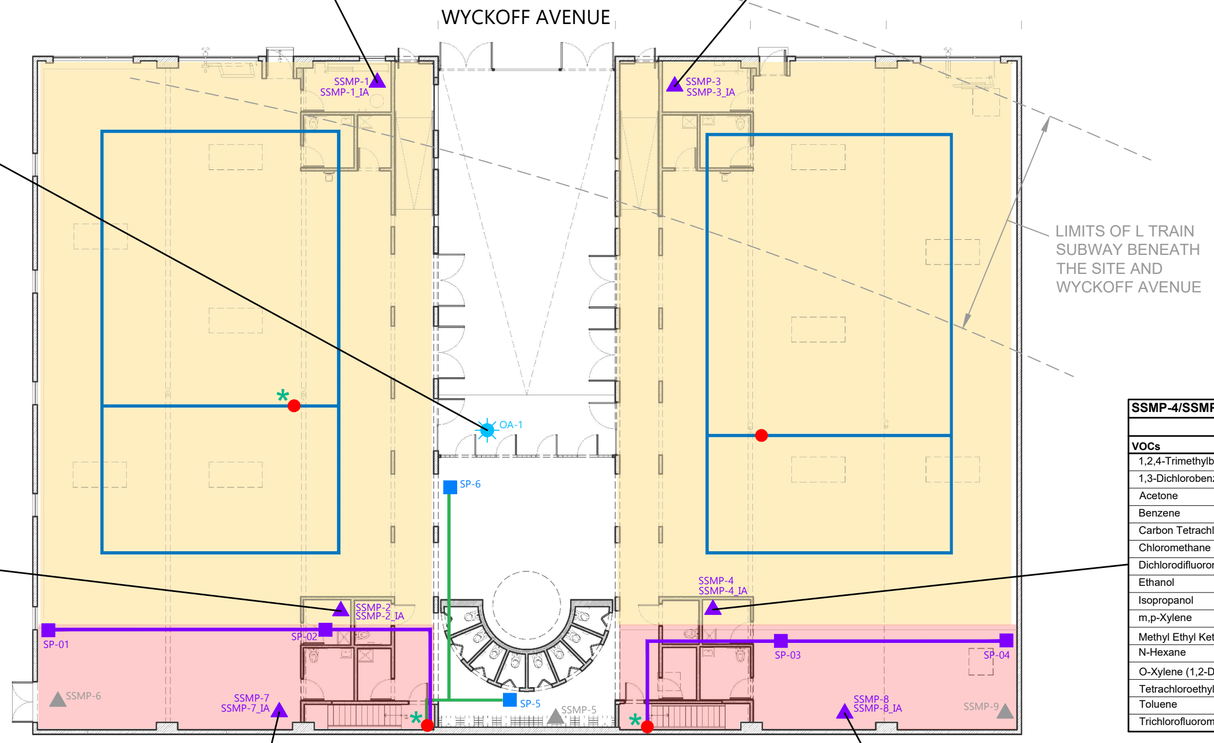
OA-1	05/03/2023
VOCs	
Acetone	**10.1**
Benzene	**0.776**
Carbon Tetrachloride	**0.459**
Chloromethane	**1.08**
Cis-1,2-Dichloroethylene	**0.083**
Dichlorodifluoromethane	**2.33**
Ethanol	**14.9**
Isopropanol	**1.59**
Methylene Chloride	**1.81**
N-Hexane	**1.33**
Tetrachloroethylene (PCE)	**1.53**
Toluene	**2.28**
Trichloroethylene (TCE)	**1.08**
Trichlorofluoromethane	**1.14**

SSMP-2/SSMP-2_IA	05/03/2023	05/03/2023
VOCs	SSMP-2	SSMP-2_IA
1,2,4-Trimethylbenzene	ND	**1.95**
Acetone	**41.8**	**58.7**
Benzene	**0.722**	ND
Carbon Disulfide	**0.722**	ND
Carbon Tetrachloride	ND	**0.465**
Chloromethane	**1.05**	**1.16**
Dichlorodifluoromethane	**2.33**	**2.39**
Ethanol	**39.8**	**15.8**
Isopropanol	**2.7**	**2.19**
m,p-Xylene	**2.24**	**1.75**
Methyl Ethyl Ketone (2-Butanone)	**2.17**	ND
Methyl Isobutyl Ketone (4-Methyl-2-Pentanone)	**2.41**	ND
N-Hexane	**0.733**	**0.899**
O-Xylene (1,2-Dimethylbenzene)	**0.921**	ND
Tert-Butyl Alcohol	**1.93**	ND
Tetrachloroethylene (PCE)	ND	**0.197**
Toluene	**2.86**	**2.11**
Trichlorofluoromethane	**1.21**	**1.23**

SSMP-4/SSMP-4_IA	05/03/2023	05/03/2023
	SSMP-4	SSMP-4_IA
VOCs		
1,2,4-Trimethylbenzene	ND	**1.96**
1,3-Dichlorobenzene	**1.51**	ND
Acetone	**41.8**	**56.1**
Benzene	ND	**0.716**
Carbon Tetrachloride	ND	**0.465**
Chloromethane	**1.06**	**1.15**
Dichlorodifluoromethane	**2.31**	**2.31**
Ethanol	**44.1**	**54.3**
Isopropanol	**3.91**	**4.84**
m,p-Xylene	**2.51**	**2.15**
Methyl Ethyl Ketone (2-Butanone)	**1.49**	ND
N-Hexane	**0.708**	**1.74**
O-Xylene (1,2-Dimethylbenzene)	**1.16**	ND
Tetrachloroethylene (PCE)	ND	**0.373**
Toluene	**2.69**	**2.95**
Trichlorofluoromethane	**1.14**	**1.16**

SSMP-7/SSMP-7_IA	05/03/2023	05/03/2023
	SSMP-7	SSMP-7_IA
VOCs		
1,1,1-Trichloroethane (TCA)	**4.3**	ND
1,2,4-Trimethylbenzene	ND	**2.12**
1,3-Dichlorobenzene	**2.45**	ND
1,3,5-Trimethylbenzene (Mesitylene)	ND	**0.605**
2-Hexanone	**0.861**	ND
4-Ethyltoluene	ND	**0.551**
Acetone	**12.9**	**45.8**
Benzene	ND	**0.738**
Carbon Tetrachloride	ND	**0.54**
Chloromethane	ND	**1.1**
Dichlorodifluoromethane	**1.91**	**2.33**
Ethanol	**98.9**	**31.8**
Ethylbenzene	**1.02**	**0.556**
Isopropanol	**10.3**	**2.8**
m,p-Xylene	**3.4**	**1.82**
Methyl Ethyl Ketone (2-Butanone)	**2.34**	ND
Methyl Isobutyl Ketone (4-Methyl-2-Pentanone)	**2.08**	ND
O-Xylene (1,2-Dimethylbenzene)	**1.58**	**0.747**
Tert-Butyl Alcohol	**2.51**	ND
Tetrachloroethylene (PCE)	**1.57**	ND
Toluene	**3.06**	**2.25**
Trichloroethylene (TCE)	**17**	ND
Trichlorofluoromethane	**1.21**	**1.17**

SSMP-8/SSMP-8_IA	05/03/2023	05/03/2023
	SSMP-8	SSMP-8_IA
VOCs		
1,1,1-Trichloroethane (TCA)	**3.4**	ND
1,2,4-Trimethylbenzene	**3.97**	**2.32**
1,3,5-Trimethylbenzene (Mesitylene)	**1.05**	ND
Acetone	**7.79**	**54.2**
Benzene	ND	**0.693**
Carbon Tetrachloride	ND	**0.453**
Chloroform	**1.62**	ND
Chloromethane	ND	**1.12**
Dichlorodifluoromethane	ND	**2.35**
Ethanol	**43.3**	**28.6**
Ethylbenzene	**1.52**	ND
Isopropanol	**3.12**	**2.85**
m,p-Xylene	**5.43**	ND
N-Heptane	**0.869**	ND
N-Hexane	**1.07**	**1**
O-Xylene (1,2-Dimethylbenzene)	**2.54**	ND
Tetrachloroethylene (PCE)	**18.6**	**0.244**
Toluene	**6.82**	**2.26**
Trichlorofluoromethane	**1.84**	**1.17**



NOTES

1. Visible expansion joints and slab cracks or openings greater than 1/16" throughout the area to be depressurized were properly sealed with Sikasil®-728 NS sealant.
2. Sump pumps and utility enclosures throughout the area to be depressurized were evaluated by a qualified environmental consultant and the SSDS installer to determine proper sealing method.
3. Six (6) suction points (SPs, 3" Ø x 12", SP-01 to SP-06) were constructed at the indicated locations, following suction pit detail.
4. Piping materials above and below ground comply with materials specified in NYC DOB Mechanical Code, Section 512, latest edition. All exterior pipes are Schedule 40 PVC and all interior pipes are cast iron, unless otherwise noted.
5. All PVC joints were sealed with plumber's cement (or similar product) applied according to the manufacturer's specifications. Cast iron no-hub couplers at all cast iron pipe unions.
6. Horizontal piping is pitched down from the riser pipe towards suction points at 1/8" per foot (1% slope) to facilitate condensation drainage.
7. Appropriate fire stop details were installed at any location in which piping penetrates a fire rated wall.
8. Piping inside and outside the building is mounted to the nearest building column, beam or supporting structure with hangers, clamps, or brackets in accordance with all applicable code and/or manufacturer's recommendations.
9. All overhead piping maintains a minimum distance of 6'-8" to floor.
10. Discharge point (DP) extends a minimum of 20" above the roof. DPs are at least 10' from other buildings, HVAC intakes, windows or doors.
11. A powered fan has been installed at each riser pipe, following construction details, by a licensed electrical contractor in accordance with NYC DOB construction code and any other applicable code and regulations utilizing a hard-wired electrical connection with a dedicated power switch and breaker.
12. Sub-slab monitoring points (SSMPs) were installed for field testing and monitoring purposes in accordance with design document specification. Penetrations through vapor barrier and slab were sealed in accordance with manufacturer specifications. Adequate negative pressure at the SSMPs are defined as equal to or less than -0.004 in. w.c.
13. U-manometers or equivalent pressure gauges were installed at each vertical pipe connected to a SP at visible locations as indicators of negative pressure.
14. A warning alarm (voice dialer system, flashing light indicator, or approved equal) was installed by a licensed electrical contractor to indicate fan failure.
15. A 4" butterfly valve (or approved equal, one per SP) was installed for system balancing purposes. Butterfly valve was installed at an accessible location along the pipe connecting the SP to the main overhead piping connecting multiple SPs.
16. All U-manometers, visible SSDS piping in the exterior and interior portions of the building, and powered fans are clearly labeled as "Sub-Slab Venting System" by means of stickers, stencil or other approved marking directly on each item.

LEGEND

- OUTDOOR AMBIENT AIR SAMPLE
- SUB–SLAB MONITORING POINT, SOIL VAPOR, AND INDOOR AIR SAMPLING LOCATION
- SUB–SLAB MONITORING POINT

GROUND FLOOR

- 3–INCH CAST IRON VERTICAL RISER
- ACCESS PANEL TO BE PROVIDED FOR MONITORING PURPOSES
- 3–INCH SCHEDULE 40 PERFORATED PVC PIPING
- 4–INCH SCHEDULE 40 NON–PERFORATED PVC PIPING
- SUCTION POINT
- EXTENT OF 6–INCH CONTINUOUS GRAVEL BELOW SLAB
- UNDERLYING BASEMENT AREA

BASEMENT

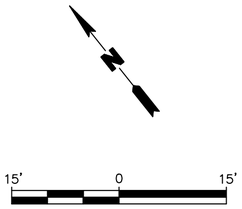
- 3–INCH CAST IRON CEILING PIPE
- SUCTION POINT

TYPICAL DATA BOX INFORMATION

SAMPLE ID# →	SSMP-8_IA	05/03/2023 ← SAMPLE DATE
	VOCs	
ANALYTES →	1,2,4-Trimethylbenzene	**2.32**
	Acetone	**54.2**
	Benzene	**0.693**
	Carbon Tetrachloride	**0.453**
	Chloromethane	**1.12**
	Dichlorodifluoromethane	**2.35**

→ CONCENTRATIONS (µg/m³)

µg/m³ – MICROGRAMS PER CUBIC METER
VOCs – VOLATILE ORGANIC COMPOUNDS



Base map provided by Cody & Wyckoff - Basement Level - Existing Floor Plan dated 6/17/20.
All feature locations are approximate. This map is intended as a schematic to be used in conjunction with the associated report, and it should not be relied upon as a survey for planning or other activities.

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Title:

SUB-SLAB DEPRESSURIZTION SYSTEM LAYOUT SHOWING DETECTIONS IN SOIL VAPOR

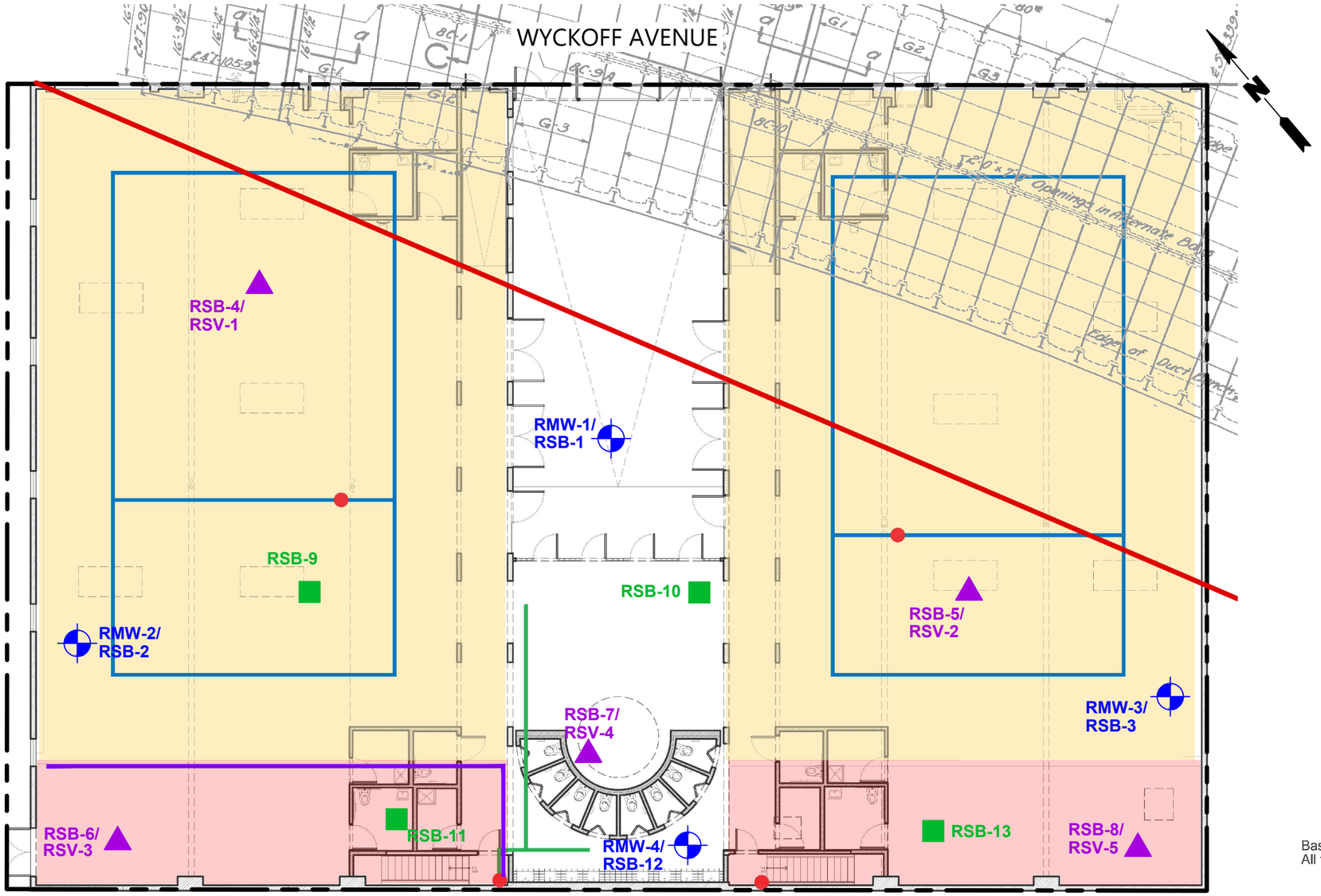
11-24 WYCKOFF AVENUE
QUEENS, NEW YORK

Prepared for:

1124 WYCKOFF LLC

ROUX

Compiled by: J.R.	Date: 9/10/2024	FIGURE
Prepared by: B.H.C.	Scale: APPROXIMATE	5
Project Mgr: S.S.	Project: 4167.0001Y002	
File: 4167.0001Y106.03.DWG		



SSDS NOTES

1. Visible expansion joints and slab cracks or openings greater than 1/16" throughout the area to be depressurized were properly sealed with Sikasil®-728 NS sealant.
2. Sump pumps and utility enclosures throughout the area to be depressurized were evaluated by a qualified environmental consultant and the SSDS installer to determine proper sealing method.
3. Six (6) suction points (SPs, 3" Ø x 12", SP-01 to SP-06) were constructed at the indicated locations, following suction pit detail.
4. Piping materials above and below ground comply with materials specified in NYC DOB Mechanical Code, Section 512, latest edition. All exterior pipes are Schedule 40 PVC and all interior pipes are cast iron, unless otherwise noted.
5. All PVC joints were sealed with plumber's cement (or similar product) applied according to the manufacturer's specifications. Cast iron no-hub couplers at all cast iron pipe unions.
6. Horizontal piping is pitched down from the riser pipe towards suction points at 1/8" per foot (1% slope) to facilitate condensation drainage.
7. Appropriate fire stop details were installed at any location in which piping penetrates a fire rated wall.
8. Piping inside and outside the building is mounted to the nearest building column, beam or supporting structure with hangers, clamps, or brackets in accordance with all applicable code and/or manufacturer's recommendations.
9. All overhead piping maintains a minimum distance of 6'-8" to floor.
10. Discharge point (DP) extends a minimum of 20" above the roof. DPs are at least 10' from other buildings, HVAC intakes, windows or doors.
11. A powered fan has been installed at each riser pipe, following construction details, by a licensed electrical contractor in accordance with NYC DOB construction code and any other applicable code and regulations utilizing a hard-wired electrical connection with a dedicated power switch and breaker.
12. Sub-slab monitoring points (SSMPs) were installed for field testing and monitoring purposes in accordance with design document specification. Penetrations through vapor barrier and slab were sealed in accordance with manufacturer specifications. Adequate negative pressure at the SSMPs are defined as equal to or less than -0.004 in. w.c.
13. U-manometers or equivalent pressure gauges were installed at each vertical pipe connected to a SP at visible locations as indicators of negative pressure.
14. A warning alarm (voice dialer system, flashing light indicator, or approved equal) was installed by a licensed electrical contractor to indicate fan failure.
15. A 4" butterfly valve (or approved equal, one per SP) was installed for system balancing purposes. Butterfly valve was installed at an accessible location along the pipe connecting the SP to the main overhead piping connecting multiple SPs.
16. All U-manometers, visible SSDS piping in the exterior and interior portions of the building, and powered fans are clearly labeled as "Sub-Slab Venting System" by means of stickers, stencil or other approved marking directly on each item.

Base map provided by Cody & Wyckoff - Basement Level - Existing Floor Plan dated 6/17/20.
All feature locations are approximate. This map is intended as a schematic to be used in conjunction

LEGEND

GROUND FLOOR

- 3–INCH CAST IRON VERTICAL RISER
- 3–INCH SCHEDULE 40 PERFORATED PVC PIPING
- 4–INCH SCHEDULE 40 NON–PERFORATED PVC PIPING
- EXTENT OF 6–INCH CONTINUOUS GRAVEL BELOW SLAB
- UNDERLYING BASEMENT AREA

BASEMENT

- 3–INCH CAST IRON CEILING PIPE

RMW-1/ RSB-1 — PROPOSED LOCATION AND DESIGNATION OF PERMANANT MONITORING WELL AND SOIL BORING TO APPROXIMATELY 70 FT BLS

RSB-4/ RSV-1 — PROPOSED LOCATION AND DESIGNATION OF SOIL BORING TO 10 FT BLS AND SOIL VAPOR SAMPLE

RSB-9 — PROPOSED LOCATION OF SOIL BORING TO 10 FT BLS

MTA UNDERGROUND EASEMENT

BCP SITE BOUNDARY

GENERAL NOTES

1. SAMPLE LOCATIONS ARE SUBJECT TO CHANGE PENDING NYCTA/MTA REVIEW.
2. AN INDOOR AIR SAMPLE WILL BE CO–LOCATED WITH EACH SOIL VAPOR SAMPLE AND ONE AMBIENT AIR SAMPLE WILL BE COLLECTED OUTDOORS.

15' 0 15'

Title:

PROPOSED SAMPLING LOCATIONS

11-24 WYCKOFF AVENUE
QUEENS, NEW YORK

Prepared for:

1124 WYCKOFF LLC

ROUX

Compiled by: V.S.	Date: 12/17/2025	FIGURE
Prepared by: B.H.C.	Scale: AS SHOWN	6
Project Mgr: V.S.	Project: 4167.0001Y000	
File: 4167.0001Y106.01.DWG		

V:\CAD\PROJECTS\4167Y0001Y106\4167.0001Y106.01.DWG GMILTON

APPENDICES

A. Previous Environmental Investigation Reports (Provided as Separate Files)

B. Quality Assurance Project Plan/Field Sampling Plan

C. Community Air Monitoring Plan

D. Site Health and Safety Plan

APPENDIX A

Previous Environmental Investigation Reports

APPENDIX B

Quality Assurance Project Plan/Field Sampling Plan

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

11-24 Wyckoff Avenue
Queens, New York 11385

December 19, 2025

Prepared for:

1124 Wyckoff LLC
2483 Coney Island Avenue
Brooklyn, New York 11223

Prepared by:

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

4167.0001Y003.107/CV

Environmental Consulting
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rouxinc.com

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2. Field and Laboratory QC Summary
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4. Preservation, Holding Times, and Sample Containers

Attachments

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

1. Introduction

Roux Environmental Engineering and Geology, D.P.C. (Roux), on behalf of 1124 Wyckoff LLC (Volunteer), 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) in accordance with the Remedial Investigation Work Plan (RIWP) for the property located at 11-24 Wyckoff Avenue, Queens, New York (Site) are of quality sufficient to meet project-specific data quality objectives (DQOs). This QAPP/FSP also includes field sampling procedures. The Volunteer has submitted an application for entry into the New York State Department of Environmental Conservation (NYSDEC) Brownfield Cleanup Program (BCP). The RI would be completed in accordance with the requirements of the BCP.

This QAPP/FSP was prepared in accordance with the guidance provided in NYSDEC's DER-10, Technical Guidance for Site Investigation and Remediation (DER-10) dated May 2010, and the United States Environmental Protection Agency's (USEPA's) Guidance on Systematic Planning using the Data Quality Objectives Process (EPA QA/G-4) dated February 2006.

1.1 Purpose

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

This QAPP/FSP was 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, locations, analytical suites, and frequency (Section 4);
- field sampling procedures (Section 5);
- sample handling, sample analysis, and QA/QC (Section 6); and
- Site control procedures and decontamination (Section 7).

2. Sampling Objectives

Previous investigations at the Site have documented the presence of soil and groundwater impacts, and the potential for soil vapor intrusion (SVI) potentially due to the former use of the Site as auto houses, auto laundry, and a knitting mill, and the presence of contaminated historic fill materials and a closed-in-place underground storage tank (UST).

The RI will evaluate soil and groundwater quality, and the potential for SVI onsite and at the property boundaries to provide the basis for remedial action selection and to determine the potential for offsite 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).

Sampling procedures are discussed in Section 5 of this QAPP/FSP. A discussion of the DQOs and QA/QC 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/Remedial Engineer

Noelle Clark, P.E. will serve as the Project Principal and Remedial Engineer. The Project Principal is responsible for defining project objectives and bears ultimate responsibility for the successful completion of the investigation. The Remedial Engineer is a registered professional engineer licensed by the State of New York. The Remedial Engineer will have primary direct responsibility for implementation of the RI and future remedial program for the Site. The Remedial Engineer will certify in the Remedial Investigation Report (RIR) that the investigation activities were observed by qualified environmental professionals under their supervision as well as any other relevant provisions of Environmental Conservation Law (ECL) 27-1419 have been achieved in full conformance with the RI.

Project Manager

Valerie Sabatasso will serve as the Project Manager. The Project Manager is responsible for defining project objectives and bears ultimate responsibility for the successful completion of the work. This individual 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

Patrick Perry 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

Laboratory analysis will be completed by Eurofins Environment Testing of Edison, New Jersey, Sacramento, California, and Burlington, Vermont, New York State Department of Health (NYSDOH) Environmental Laboratory Approval Program (ELAP)-certified laboratories (11452, 11666, and 10391, respectively). The Laboratory Project Manager is Elizabeth Flannery. 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 QA 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

Sarah Stern will serve as the Quality Assurance Officer (QAO) for this project. The QAO is responsible for conducting reviews, inspections, and audits to ensure that the data collection is conducted in accordance with the QAPP/FSP. 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 makes recommendations to the Field Team Leader.

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

The media to be sampled during the RI includes soil, groundwater, and soil vapor/indoor air/ambient air. Sampling locations, analytical suites, and frequency vary by medium. 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 QC samples, is included in Tables 2 and 3. 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, Groundwater, Soil Vapor, Indoor Air, Ambient Air Sampling Rational

The proposed soil, groundwater, soil vapor, indoor air, and ambient air sampling locations are described in the table below.

Table 1. Summary of Sample Rationale

Sample Location ID	Region of Site	Rationale	Sample Depths	Analyses
RSB-1, RSB-2, RSB-3, and RSB-12 (Soil)	Delineating upgradient and downgradient soil	Surface soil samples will be collected to evaluate exposure pathways. Other samples will provide coverage across the Site and evaluate the potential presence of soil contamination	0-2 ft bls, most impacted (if encountered, if not from middle of boring), 60-62 ft bls (2 ft above groundwater)	Target Compound List (TCL)/NYSDEC Part 375 VOCs, SVOCs (including 1,4-Dioxane), pesticides/herbicides, polychlorinated biphenyls (PCBs), Target Analyte List (TAL)/NYSDEC Part 375 metals, Per- and Polyfluoroalkyl Substances (PFAS, three samples only)
RSB-4 through RSB-11, and RSB-13 (Soil)	Delineating upgradient and downgradient soil	Surface soil samples will be collected to evaluate exposure pathways. Other samples will provide coverage across the Site and evaluate the potential presence of soil contamination	0-2 ft bls, most impacted (if encountered, if not 8-10 ft bls)	TCL/NYSDEC Part 375 VOCs, SVOCs (including 1,4-Dioxane), pesticides/herbicides, PCBs, TAL/NYSDEC Part 375 metals, PFAS (five samples only)
RMW-1 through RMW-4 (Groundwater)	Middle of site, and east and west corners of Site	To characterize onsite groundwater quality and determine groundwater flow	Groundwater level (approximately 62 ft bls)	TCL/NYSDEC Part 375 VOCs, SVOCs, pesticides, herbicides, PCBs, TAL/NYSDEC Part 375 metals, Emerging Contaminants* (ECs)

Sample Location ID	Region of Site	Rationale	Sample Depths	Analyses
RSV-1 through RSV-5 (Sub Slab Soil Vapor)	Inside building delineating upgradient and downgradient soil vapor impact	To characterize onsite soil vapor impacts and evaluate the potential for soil vapor intrusion	Immediately below the concrete slab	TO-15
IA-1 through IA-5 (Indoor Air)	Indoor Air collocated with soil vapor points	To evaluate the potential for vapor intrusion	Breathing zone collocated with soil vapor samples	TO-15
AMB-1 (Ambient Air)	Outside, middle of Site	Outdoor background atmospheric conditions	Breathing zone in outdoor space	TO-15

*1,4-Dioxane and PFAS are considered Emerging Contaminants (ECs).

ECs list includes 1,4-Dioxane and the 40 PFAS which include the 40 compounds listed in the most recent April 2023 NYSDEC guidance Sampling, Analysis, and Assessment of Per-and Polufluoroalkyl Substances (PFAS) Under NYSDEC's Part 375 Remedial Programs (NYSDEC April 2023 Guidance) and is included as Attachment 2 to this QAPP/FSP. PFAS in soil and groundwater will be analyzed via USEPA EPA Method 1633. 1,4-Dioxane in soil will be analyzed by USEPA EPA Method 8270E.

The Eurofins Standard Operating Procedures (SOPs) for completing analyses listed in the table above and reporting limits/minimum detection limits for compounds are included in Attachment 3.

Samples will be collected in laboratory-provided containers and analyzed at a NYSDOH Environmental Laboratory Approval Program-certified laboratory.

4.2 Soil Sample Analytical Suites

Soil samples will be used to determine the nature and extent of contamination at the Site and obtain a current representation of the environmental conditions at the Site. A total of 13 soil borings are proposed to be installed at the locations shown in Figure 6 of the RIWP.

Soil samples collected as part of the RI will be analyzed for the following list of parameters:

- Target Compound List (TCL)/NYSDEC Part 375 volatile organic compounds (VOCs) via USEPA method 8260D;
- TCL/NYSDEC Part 375 semivolatile organic compounds (SVOCs) via USEPA method 8270E (including the EC 1,4-dioxane);
- TCL/NYSDEC Part 375 pesticides USEPA method 8081B;
- TCL/NYSDEC Part 375 herbicides USEPA method 8151A;
- TCL/NYSDEC Part 375 polychlorinated Biphenyls (PCBs) via USEPA method 8082A;
- Total Analyte List (TAL)/NYSDEC Part 375 metals (including mercury, hexavalent chromium, trivalent chromium, and total cyanide) via USEPA method 6020B, 7471B, 7196A and 9012B; and
- PFAS via USEPA 1633 (approximately 25 percent [eight total] of the soil samples will be analyzed for PFAS).

Appropriate QA/QC samples will be collected based on applicable guidance. Additional samples may be required if evidence of impacts is observed.

4.3 Groundwater Sample Analytical Suites

To characterize onsite groundwater flow and groundwater quality, four of the soil borings (RSB-1, RSB-2, RSB-3, and RSB-12) will be converted into permanent groundwater monitoring wells RMW-1, RMW-2, RMW-3, and RMW-4, respectively) for gauging, sampling and analysis. Groundwater samples will be collected from each of the four permanent monitoring wells. The proposed RI monitoring well locations are provided in Figure 8.

Groundwater samples collected as part of the RI will be analyzed for the following list of parameters:

- TCL/NYSDEC Part 375 VOCs via USEPA method 8260D;
- TCL/NYSDEC Part 375 SVOCs via USEPA method 8270E;
- TCL/NYSDEC Part 375 pesticides via USEPA method 8081B;
- TCL/NYSDEC Part 375 herbicides USEPA method 8151A;
- TCL/NYSDEC Part 375 PCBs via USEPA method 8082A;
- TAL/NYSDEC Part 375 total and dissolved metals (including hexavalent chromium, trivalent chromium, and total cyanide) via USEPA method 6020B, 7470A, and 9012B; and
- PFAS via USEPA 1633.
- 1,4-Dioxane via USEPA Method 8270E-SIM

Groundwater samples will be filtered by the laboratory for metals and both filtered and unfiltered results will be reported.

Field parameters (pH, temperature, turbidity, conductivity, oxidation-reduction potential [ORP], and dissolved oxygen) will also be collected in the field using a water quality meter during purging prior to sample collection.

4.4 SVI Investigation Analytical Suite

As part of the SVI investigation, five soil vapor samples, five indoor air samples and one outdoor ambient air sample will be collected. All sub-slab vapor, indoor air, and outdoor ambient air samples will be collected using pre-cleaned (batch certified) 2.7-liter summa canisters with regulators calibrated to collect samples over an 8-hour period and analyzed via USEPA Method TO-15 for VOCs.

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 SVI sampling). As discussed, the sample locations are shown on Figure 6 of the RIWP. Additional details regarding sampling procedures and protocols are described in Roux's relevant SOPs, which are provided in Attachment 4.

5.1 Soil Sampling and Permanent Monitoring Well Installation

Details for the collection of soil samples and the installation of groundwater monitoring wells are provided below. At soil boring locations, pre-clearance will be completed to 5 ft bls using non-intrusive methods (i.e., hand auger, vacuum technology, etc.) prior to advancement of soil borings to verify the absence of potential underground utilities. Should a utility or other feature be observed during pre-clearance activities, the sampling location will be relocated to a nearby location.

5.1.1 Soil Sampling

Thirteen soil borings will be installed during the investigation. The soil borings will be installed using either a compact Geoprobe drill rig with direct push and hollow stem auger capabilities or a portable Geoprobe direct push drill rig for areas where the larger rig cannot access. Summarized in Table 1, soil borings will be advanced to 10 ft below land surface (bls) at 10 locations and to approximately 70 ft bls at the three locations to be converted into monitoring wells. Prior to advancement with a drill rig, each boring location will be cleared to a depth of 5 ft bls using hand tools (e.g., post-hole digger, shovel, hand-auger, etc.) and/or a vacuum excavator to identify if any shallow utilities or other obstructions are present. Soil from each soil boring will be visually inspected for evidence of impacts and screened for organic vapors in the field using a photoionization detector (PID). Soil lithology will be recorded according to the Unified Soil Classification System (USCS). The following soil samples will be collected:

- RSB-1, RSB-2, RSB-3, RSB-12: Three soil samples will be collected from each deep soil boring. The samples will be collected from 0-2 ft bls, the most impacted two-foot interval, and from either
  60-62 ft bls or the two foot interval above groundwater. If no impacted material is encountered a sample will be collected from the middle two feet of the boring.
- RSB-4 through RSB-11, and RSB-13: Two soil samples will be collected from each shallow soil boring. The samples will be collected from 0-2 ft bls, the most impacted two-foot interval, or if no impacted material is encountered, the second sample will be collected from 8-10 ft bls.

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. Soil samples will be analyzed for the parameters described above in Section 4.2. 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, included in Attachment 4.

Following sample collection, the boreholes will be backfilled with soil cuttings and patched with concrete or asphalt to restore the location to pre-existing conditions. Contaminated soil cuttings, if encountered, will be placed in sealed and labeled United States Department of Transportation (DOT) approved 55-gallon drums pending characterization and offsite disposal at a permitted facility.

All soil sampling activities will be completed in accordance with the NYSDEC April 2023 Guidance. 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 (PPE) (i.e., no materials will contain Gore-Tex or Tyvek);
- Avoid using Grundfos and bladder pumps and 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 Alconox or Liquinox for decontamination.

5.1.2 Monitoring Well Installation

Based on data from previous environmental reports, it is assumed that groundwater will be encountered at approximately 62 ft bls.

Following soil sampling activities, four monitoring wells will be installed bridging the water table and to a maximum depth of approximately 70 ft bls. Monitoring wells will be constructed of two-inch inside diameter, schedule 40 polyvinyl chloride (PVC) casing and 0.020-inch slot machined screen. Well screens will be ten feet long, and will be installed with three feet of well screen above and seven feet of well screen below the water table. A sand pack will be placed around the well screen, extending two feet above the top of the screened zone. Once the driller confirms the depth of the sand pack, a minimum two-foot-thick bentonite pellet seal will be placed above the sand pack. The permanent wells will be completed using locking well plugs, and flush-mounted, bolt down, watertight, manhole covers cemented into place.

Each of the newly installed permanent monitoring wells will be developed to remove any fine-grained material in the vicinity of the well screen and to promote hydraulic connection with the aquifer. The wells will be developed using a submersible pump, which will be surged periodically until well yield is consistent and has a turbidity below 50 Nephelometric turbidity units (NTUs).

5.2 Groundwater Sampling

Groundwater samples will be collected no sooner than one week following development of the wells. 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. Purging and sampling will be performed consistent with USEPA low-flow sampling requirements. Field parameters (pH, temperature, turbidity, conductivity, ORP, and dissolved oxygen) will be collected using a water quality meter with a flow-through cell until parameters stabilize before samples are collected. Groundwater samples will be analyzed for the parameters described above in Section 4.3.

All groundwater sampling activities will be completed in accordance with the NYSDEC April 2023 Guidance. Additional necessary precautions will be taken when sampling for ECs in the field, including, but not limited to:

- Using the proper field clothing or PPE (i.e., no materials will contain Gore-Tex or Tyvek);

- Avoid using Grundfos and bladder pumps and sampling equipment components/containers making contact with aluminum foil, LDPE, glass, or polytetrafluoroethylene materials;
- Following PFAS field sampling guidelines (i.e., using sampling materials made from 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 Alconox or Liquinox for decontamination.

All groundwater samples will be collected and placed in laboratory-supplied containers and shipped to the laboratory under chain of custody procedures in accordance with Roux's field sampling SOPs included in Attachment 4.

5.3 Soil Vapor Intrusion Sampling

Five sub-slab soil vapor samples will be collected during the RI to evaluate soil vapor concentrations at the Site. All soil vapor samples will be installed immediately beneath the concrete slab in the building using a hammer drill and prefabricated, stainless-steel vapor pins, attached to Teflon-lined polyurethane tubing.

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 NYSDOH Guidance. Following purging and verification with the tracer gas, the tubing will be connected to the pre-cleaned (batch-certified) laboratory supplied six-liter summa canister.

Additionally, five indoor air samples will be collected concurrently with the sub-slab vapor samples, as well as one ambient air sample from an outdoor location, and submitted for laboratory analysis. To the extent practicable, the indoor air samples and outdoor air sample will be collected at breathing height in open areas typical of the working environment. Indoor and outdoor air samples will be collected in pre-cleaned (batch-certified) laboratory supplied six-liter summa canisters.

6. Sample Handling, Sample Analysis, and QA/QC

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 4.

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

Samples are anticipated to be transported to the laboratory via courier, however, 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 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 Zip-lock™ bags and placed on top of the packaging material. Blue Ice cannot be used for cooling inside containers being used for shipping PFAS samples.
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 Zip-loc™ 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 QA/QC

Judy Harry, a third-party data validator, will review the analytical data for QA/QC and prepare the Data Usability Summary Report (DUSR) in accordance with NYSDEC standards. Validator resume is included in Attachment 1. A laboratory SOP for analysis of PFAS is included in Attachment 3.

The primary intended use for the data proposed to be collected is to characterize Site conditions and determine what remediation needs to be undertaken at the Site. 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 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").

Table 2 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 3 lists the number/type of field and QA/QC samples that will be collected during the RI. Table 4 lists the preservation, holding times, and sample container information.

All RI "assessment" analyses will be performed in accordance with the NYSDEC Analytical Services Protocol (ASP), using USEPA SW 846 methods, where applicable.

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 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 during the RI.

7. Site Control Procedures and Decontamination

Site control procedures, including decontamination, 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 included in Attachment 4. 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. All decontmination will be completed using the standard two step process using detergent (only Alconox will be used as , Liquinox shall not be used)) and clean, PFAS-free water for sample equipment. All water sources used for equipment decontamination during EC sampling of groundwater or soil will be verified in advance to be PFAS-free through laboratory analysis or certification, in accordance with the NYSDEC April 2023 Guidance.

7.2 Investigation Derived Waste Handling and Disposal

All investigation derived waste (IDW) materials (contaminated drill cuttings, development water, purge water, decontamination water, etc.) generated during the RI will be consolidated, and stored in appropriate labeled bulk containers (drums, etc.), and temporarily staged at a designated IDW storage area onsite. Roux will coordinate waste characterization and disposal by appropriate means. Containers will be labeled as "Non-Hazardous. Pending Analysis" until waste characterization sampling is complete and the type of waste has been determined.

TABLES

1. Summary of Sample Rationale *(embedded in text)*
2. Field and Laboratory QC Summary
3. Remedial Investigation Sampling Summary
4. Preservation, Holding Times, and Sample Containers

Table 2. Field and Laboratory QC Summary

QC Check Type	Minimum Frequency	Use
Field QC		
Duplicate	1 per matrix per 20 samples or SI	Precision
Trip Blank	1 per VOC cooler	Sensitivity
Field Blank (non-PFAS)	1 per matrix per 20 samples	Sensitivity
Field Blank (PFAS)	1 per matrix per 20 samples	Sensitivity
Laboratory QC		
Laboratory Control Sample	1 per matrix per SDG	Accuracy
Matrix Spike/Matrix Spike Duplicate/Matrix Duplica	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

Notes:

* SDG - Sample Delivery Group - Assumes a single extraction or preparation

** Provided to lab by field sampling personnel

PFAS - Per- and Polyfluoroalkyl Substances

Table 3. Remedial Investigation Sampling Summary

Sample Medium	Target Analytes	Field Samples	Replicates[1]	Trip Blanks[2]	Field Blanks[1]	Matrix Spikes[1]	Spike Duplicates[1]	Total No. of Samples
Soil	TCL/Part 375 VOCs +10	29	2	5	2	2	2	42
	TCL/Part 375 SVOCs +20	29	2	-	2	2	2	37
	TCL/Part 375 Pesticides	29	2	-	2	2	2	37
	TCL/Part 375 Herbicides	29	2	-	2	2	2	37
	TCL/Part 375 PCBs	29	2	-	2	2	2	37
	TAL Metals	29	2	-	2	2	2	37
	PFAS	8	1	-	1	1	1	12
Groundwater	TCL/Part 375 VOCs +10	3	1	1	1	1	1	8
	TCL/Part 375 SVOCs +20	3	1	-	1	1	1	7
	TCL/Part 375 Pesticides	3	1	-	1	1	1	7
	TCL/Part 375 Herbicides	3	1	-	1	1	1	7
	TCL/Part 375 PCBs	3	1	-	1	1	1	7
	TAL Metals*	3	1	-	1	1	1	7
	PFAS	3	1	-	1	1	1	7
Soil Vapor, Indoor Air, Ambient Air	TO-15 VOCs	11	1	-	-	-	-	12

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

TCL/Part 375 - USEPA Contract Laboratory Program Target Compound List

USEPA - United States Environmental Protection Agency

VOCs - Volatile Organic Compounds

SVOCs - Semivolatile Organic Compounds

PCBs - Polychlorinated Biphenyls

PFAS - Per- and Polyfluoroalkyl Substances

TAL - USEPA Contract Laboratory Program Target Analyte List

*Metals will be analyzed for both filtered and unfiltered metals in groundwater samples.

Table 4. Preservation, Holding Times and Sample Containers

Analysis	Matrix	Bottle Type	Preservation	Holding Time(a)
TAL/Part 375 Metals (total) via USEPA SW-846 6020B/7471	Soil Water	8 oz wide mouth glass, teflon lined cap 250 mL plastic, teflon lined cap	Cool to 4°C Nitric acid	180 days, Hg 28 days
Hexavalent Chromium via USEPA SW-846 7196A	Soil Water	2 oz wide mouth glass, teflon lined cap 500 mL plastic bottle	None	30 days from sample collection to extraction; 7 days following extraction 24 hours from sample collection
Total Cyanide via USEPA SW-846 9012B	Soil Water	4 oz wide mouth glass, teflon lined cap 250 mL plastic bottle	Cool to 4°C NaOH	14 days from sample collection 14 days from sample collection
PFAS via EPA 1633	Soil Water	Three 250 mL HDPE bottles Three 250 mL HDPE bottles	None Trizma	14 days to extract, 40 days to analysis 14 days from sample collection
1,4-Dioxane via SW-846 8270E 1,4-Dioxane via 8270E-SIM	Soil Water	8 oz wide mouth glass, teflon lined cap 500 mL amber glass	Cool to 4°C Cool to 4°C	14 days to extract, 40 days to analysis 7 days from sample collection
VOCS via TO-15	Air	2.7 liter Summa Canister	None	14 days from sample collection
TCL/Part 375 Volatile Organic Compounds (VOCs) via SW-846 8260D	Soil	Encore	Cool to 4°C	48 hours to be extruded into sealed DI and methanol vials; DI vial frozen to -7 to -20 degrees C. 14 days to analysis.
	Soil	Terra Core	Methanol Cool to 4°C	48 hours to freeze DI vials to -7 to -20 degrees C; 14 days to analyze DI and methanol vials
	Water	40mL voa vial, teflon lined cap	Hydrochloric Acid	14 days from sample collection
TCL/Part 375 Semivolatile Organic Compounds (SVOCs) via SW-846 8270E	Soil Water	8 oz wide mouth glass, teflon lined cap 1 liter amber glass, teflon lined cap	Cool to 4°C	14 days to extract, 40 days to analysis 7 days to extract, 40 days to analysis
TCL/Part 375 Pesticides via SW-846 8081B	Soil Water	8 oz wide mouth glass, teflon lined cap 1 liter amber glass, teflon lined cap	Cool to 4°C	14 days to extract, 40 days to analysis 7 days to extract, 40 days to analysis
TCL/Part 375 Herbicides via SW-846 8051A	Soil Water	8 oz wide mouth glass, teflon lined cap 1 liter amber glass, teflon lined cap	Cool to 4°C	14 days to extract, 40 days to analysis 7 days to extract, 40 days to analysis
TCL/Part 375 Polychlorinated biphenyls (PCBs) - SW-846 8082A	Soil Water	8 oz wide mouth glass, teflon lined cap 1 liter amber glass, teflon lined cap	Cool to 4°C	14 days to extract, 40 days to analysis 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
PFAS - Per- and Polyfluoroalkyl Substances
TCL - USEPA Contract Laboratory Program Target Compound List
USEPA - United States Environmental Protection Agency

ATTACHMENTS

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

ATTACHMENT 1

Professional Profiles

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.

PROFESSIONAL PROFILE

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EDUCATION

BS, Civil Engineering,
Manhattan College, 1991
ME, Environmental Engineering,
Manhattan College, 1994

PROFESSIONAL LICENSES

Professional Engineer in New York

Noelle M. Clarke, PE

Principal Engineer

EXPERIENCE SUMMARY

Thirty-two years' experience: Principal Engineer/Senior Engineer at Remedial Engineering, P.C./Roux; Project Engineer at Camp Dresser & McKee.

TECHNICAL SPECIALTIES

Feasibility studies, pilot testing, remedial design, implementation, construction management, and startup evaluations for remediation of soil, groundwater, soil vapor and sediment. Phase I/Phase II Environmental Site Assessments (ESA). Extensive experience at brownfields redevelopment sites, former industrial facilities, and public works facilities. Evaluation and design of storm water drainage systems. Evaluation, design, and construction management for new and existing wastewater treatment processes.

REPRESENTATIVE PROJECTS

- Principal Engineer providing due diligence support for real estate transactions on multiple projects in the New York metropolitan area. Projects have included multi-family housing (both affordable and market rate), retail/ commercial, community services and industrial properties. Services have included Phase I and Phase II ESAs.
- Principal Engineer for a Brownfield redevelopment of a property adjacent to a dry cleaning solvent distribution facility in Brooklyn, New York under the NYSDEC BCP. The site was previously a warehouse built on a former freight railyard that serviced the dry cleaning solvent facility. Offloading spillage on site and migration from the offsite facility resulted in significant soil, groundwater, and vapor contamination with chlorinated VOCs. The site was developed into multifamily housing with first floor retail use. Pre-remediation and posts-remediation Phase I ESAs were prepared by Roux. The remedy, as summarized in the Remedial Action Work Plan, consisted of soil hot spot removal, a physical barrier to limit on site groundwater migration, a permeable reactive wall to treat groundwater and mitigate offsite migration, and a sub slab depressurization system. Roux, under the direction of Ms. Clarke, provided full-time oversight of the remediation and prepared the Final Engineering Report and Site management Plan. The Certificate of Completion for the Site was obtained in October 2015 and Roux is currently providing post-remediation monitoring services.
- Principal Engineer for a Brownfield redevelopment in Brooklyn, New York at a mixed-use multifamily housing/neighborhood retail complex with a former onsite dry cleaner under the NYSDEC BCP. There is soil, groundwater, and vapor contamination from chlorinated VOCs from the former onsite dry cleaner, as well as groundwater contamination from offsite dry cleaners. The remedy, described in the Remedial Action Work Plan prepared by Roux, consisted of hot spot soil removal, *in situ* groundwater treatment and a sub slab depressurization system for vapor mitigation in the existing buildings. The NYSDEC accepted the Final Engineering Report prepared by Roux and the Site received a Certificate of Completion from NYSDEC in 2016 and Roux is currently providing post-remediation monitoring services.
- Principal Engineer for a complex dredging project for a major petroleum company on the Allegheny River in New York. The goal of the project was to remove 1,000 tons of petroleum impacted sediments from the river. Work included Site investigation, remedial investigation, alternatives evaluation, remedial design, planning and extensive regulatory permitting with multiple federal, state, and local agencies. The remediation work was completed in 2021.

- Principal engineer for the investigation and remediation of a former petroleum refinery located in Olean, New York under the NYSDEC BCP. The work included site investigation and preparation of an Alternatives Analysis Report (to document analysis of engineering options and remedy recommendation), Remedial Action Work Plan and remedial design documents to address the environmental impacts associated with the historical operations. The selected remedy includes excavation of shallow soil, in-situ stabilization of 25,000 cubic yards of deep petroleum impacted soil and capping. The work is being completed in phases beginning in 2022, with an anticipated completion date of 2024.
- Principal Engineer for the alternatives evaluation, remedy selection, regulatory negotiation, preparation of design documents (drawings, specifications, and permit applications) and permitting for all remedial components in support of redevelopment at a former metals manufacturing site in Staten Island, New York under the NYSDEC Voluntary Cleanup Program (VCP). The remedy included dredging and onsite disposal of stream sediments; consolidation and capping of fill material across the site; in-place abandonment of the Site's former sewer system; installation of drainage swales for storm water management; and wetland bank stabilization and mitigation/restoration. Required permits and regulatory approvals for the project included a Joint Permit from the USACE and NYSDEC; a NYSDEC SPDES equivalency permit for discharge of treated water to the Arthur Kill, a New York State Department of State Coastal Management Program (CMP) Federal Consistency Assessment; a New York City Waterfront Revitalization Program Consistency Assessment, a modification of topography authorization from New York City Department of City Planning; and a New York City Department of Environmental Protection permit for temporary discharge to a combined sewer. Also required by the USACE and National Marine Fisheries, was preparation of an Essential Fish Habitat Study, in support of the Joint Permit application. Permitting activities included preparation of the various permit applications, forms and supporting documentation, as well as follow up meetings and correspondence to finalize the authorizations.
- Principal Engineer for the design, bidding, contractor selection, and remedial construction phase at a former metals manufacturing facility in Staten Island under the NYSDEC VCP. Responsibilities included finalizing biddable construction documents, issuing to bidders, preparing addenda, and evaluating bids for presentation to the client. Following contractor selection Roux was heavily involved in coordinating with the client, regulators, and contractor for mobilization to the site in late 2006. During the construction Ms. Clarke provided support to the onsite construction manager regarding field changes, design revisions to account for unexpected conditions and contractor questions. The Final Engineering Report summarizing the construction activities was accepted by NYSDEC.
- Principal-in-Charge of an investigation and remediation project at a former petroleum refinery and current distribution facility located in Buffalo, New York. The site entered the NYSDEC BCP in 2006. Roux completed the BCP application and supported the application process. The work included assessing and remediating the potential environmental impacts associated with historical Site operations. These activities have included preparing multiple work plans and directing the activities of another consultant performing the fieldwork and preparing reports of results for field investigations including soil boring and sampling, well installation and groundwater sampling, aquifer pump testing, and groundwater/separate phase modeling. An in situ chemical oxidation system was designed, installed, and was operated as an IRM to remediate an area of free product and impacted groundwater discharging to the Buffalo River in OU-4.
- For the same petroleum Site in Buffalo, New York, multiple Alternatives Analysis Reports (to document analysis of engineering options and remedy recommendation), Remedial Action Work Plans and remedial design documents have been prepared to address the environmental impacts associated with the five Operable Units (OU) on the Site. Remedial construction for OU-1 was completed in 2007 and included excavation and disposal of impacted soil. The Final Construction Certification Report for OU-1 was accepted by the NYSDEC. The Alternatives Analysis Report and Remedial Design for OU-4 were submitted and approved by NYSDEC. The remedy for OU-4 included excavation and onsite consolidation of river sediments and site soil, stabilization of 1,400 linear feet of river embankment using tiered slopes, rip rap, and reinforced bioengineering, slurry wall groundwater containment, low permeability capping, a stormwater collection system and constructed wetland treatment for stormwater. Various vegetative measures were incorporated into the design in order to promote vegetative growth and enhance wildlife habitats. The remedial construction was completed in 2013 and 2014 and preparation of the Final Construction Completion Report was completed in 2015. The Alternatives Analysis Reports for OU-2 and OU-3 were submitted to NYSDEC. For OU-2, bench scale studies of stabilization/solidification agents were completed and evaluated for treatment of lead and petroleum impacted soil. In addition, field pilot studies of multiple options to treat petroleum impacted soils were completed and evaluated. Design of a stormwater collection system for portions of OU-2 and OU-3 was completed in 2010 and construction was completed in 2014 under the direction of Ms. Clarke.

- For the same petroleum terminal in Buffalo, New York, the work also included performing activities related to the operation of the remediation systems at the Site. These activities have included preparing a feasibility study work plan for improving water management systems at the site; preparing a work plan, directing the field work, and preparing an evaluation summary report for startup and testing of a portion of the groundwater extraction system at the Site; and assisting in preparation of plans to upgrade the existing treatment facilities at the Site.
- For the same petroleum terminal in Buffalo, New York, the work also included preparation of design documents and a completion report for in-place closure of the site's former in-ground oil water separator. In addition, a vapor enhanced extraction pilot study work plan was prepared and implemented at the site for recovery of separate-phase product in one portion of the site located adjacent to the Buffalo River. The results of the VER pilot testing, along with the results of chemical oxidation pilot testing conducted at the site, have been summarized in a Remedial Action Selection report, which recommended implementation of chemical oxidation in this portion of the site. A conceptual plan for implementation of chemical oxidation was submitted with the selection document. The work also included maintaining contact with regulatory agencies regarding the status of activities at the Terminal; preparing compliance monitoring reports for submittal to the regulatory agencies; overall project coordination; and budget management and tracking.
- Principal Engineer for the investigation, design, and implementation of a soil remediation project at a 4-acre former oil terminal in Cold Spring Harbor, New York under the NYSDEC spills program. The remedy completed included excavation and offsite disposal of approximately 20,000 tons of petroleum contaminated and/or hazardous lead contaminated soil in accordance with the future use of the site under an Environmental Easement. Additional activities completed by Roux at the site included asbestos remediation followed by building demolition, UST removal, and cesspool remediation. Roux prepared a Final Engineering Report, which was accepted by NYSDEC and resulted in the closure of the spill number for the Site.
- Principal Engineer for the investigation, design, and implementation of a soil remediation project at a portion of a former oil terminal in Sag Harbor, New York. The remedy completed included excavation and offsite disposal of approximately 2,000 tons of petroleum contaminated soil from beneath an active public roadway under the NYSDEC spills program. The remedy included extensive traffic control and coordination with Village of Sag Harbor officials, dewatering, water treatment, temporary water discharge of treated water to Sag Harbor and restoration of the public roadway in accordance with the Village of Sag Harbor Department of Public Works requirements. Roux prepared a Final Engineering Report, which was accepted by NYSDEC and resulted in the closure of the spill number for the Site.
- Project Manager for preparation of a work plan, direction the field activities and preparation of a summary report for investigation of the storm-water collection system at a petroleum terminal in Buffalo, New York. The objectives of the storm sewer investigation were to: prepare a detailed map of the Site's sewer system; re-establish connections that may have become blocked by debris; investigate the structural integrity of the storm sewers; locate areas of groundwater infiltration and assess infiltration rate and quality; assess wet and dry-weather flow and quality; and identify areas contributing surface water to the collection system, including hydrologic modeling using TR-55. Based on the results of the investigation, several improvements to the sewer system were recommended, including eliminating inlets to the system in areas of the site where no active operations currently take place and rehabilitation and/or installation of new sewers to restore flow by gravity to the treatment system.
- Principal Engineer for the investigation, remedial design, construction oversight and operation and maintenance of a bioventing and soil vapor extraction system at the Site of a diesel UST failure in Brooklyn. A free product recovery system was also designed, installed, and operated by Roux. Investigation activities included the use of the sonic drilling technique to advance twelve wells to 85 feet below grade through cobbles and boulders for delineation of separate phase product, soil, and groundwater impacts. Eight wells were converted to combination biovent/SVE wells. Design included specification of SVE and biovent blowers, piping, valves, and an automatic control system. Product only pumps were also designed and installed in three wells. Approximately 2,000 gallons of product were recovered by the two systems and the spill was closed by NYSDEC in 2011.
- Principal Engineer for a Brownfield redevelopment in Staten Island, New York of a former retail service station site under the NYSDEC BCP. There is soil, groundwater, and vapor contamination from petroleum-related constituents in the vicinity of the former gasoline piping and pump island (the petroleum source area), as well as historic fill across the entire site. The remedy, described in the Remedial Action Work Plan prepared by Roux, consisted of a sealed sheet pile containment wall around the petroleum source area, a Site Cover System across the entire site, comprised of concrete building slab/walkways, asphalt parking areas and limited landscaped areas and a sub-slab depressurization system to prevent vapor intrusion into the proposed retail building. A certificate of completion from NYSDEC was obtained in 2020.

- Project Manager for the remedial design at a Superfund Site in Nanuet, New York for the New York State Department of Environmental Conservation. The work included preparation of a preliminary design report, which evaluated two alternatives for handling hazardous soils and sediments at the site contaminated with volatile organic compounds. Each alternative was evaluated on the basis of technical feasibility, cost, and schedule for implementation. Based upon this evaluation, off-site disposal was recommended over on-site treatment. The report presented a site-wide conceptual plan for remediation, including soil/sediment excavation, staging and sampling; stream diversion; excavation dewatering; temporary on-site groundwater treatment; and long term monitoring. Duties also included managing and tracking all project budgets and serving as the main client contact.
- Principal Engineer for the design and specification of a large-scale (750 scfm) soil vapor extraction (SVE) pilot system with thermal oxidation off-gas treatment for a client in Brazil. Responsibilities included equipment sizing and specification, selection of materials of construction, SVE well and equipment layout, description of general startup procedures and preparation of a pilot test work plan. The pilot test work plan included a description of the pilot test operating procedures to be followed, operating parameters to be monitored and data to be collected and analyzed. The work also included conducting the pilot test activities and generating a report that included plans for expanding the SVE system across the Site. The work also included technical support for evaluating and optimizing system performance.
- Project Manager for a storm sewer study at the former metals manufacturing facility in Staten Island, New York as part of the Voluntary Cleanup Program for the Site to identify contaminated infiltration sources, provide an accurate site drainage map, and verify contributing areas to each outfall. The investigation included field inspections, surveying, dye testing, and sampling during varying tidal conditions. The storm sewer map prepared was used for future sewer closure and site redevelopment planning.
- Principal engineer for the design of a new storm water collection system for a metals manufacturing site in Staten Island, New York under the NYSDEC VCP. The design included evaluation and hydrologic modeling of the system using the USG Soil Conservation Service TR-55 hydrologic analysis model, inlet structure and pipe sizing and layout, outfall design and specification of materials and methods of construction for all system components.
- Principal-in-Charge of the operation, maintenance, monitoring and reporting activities at multiple active and former petroleum storage and distribution terminals located in New York for a large petroleum company. The work includes operation, maintenance, and performance/compliance monitoring services at the sites that currently have active remediation systems installed and monitoring, sampling, and reporting services at sites without systems. The remediation systems include groundwater extraction and treatment, free product recovery, bio-sparging, and soil vapor extraction/air sparging. At these sites, Roux is responsible for: maintaining and troubleshooting the various system components to reduce downtime to the extent possible; repairing and/or replacing equipment as needed; coordinating the upgrading of the electrical systems, as needed, to meet current building code requirements; expanding systems to meet regulatory requirements, as needed; optimizing system performance; collecting performance monitoring samples and data to track the efficiency of the treatment systems; and collecting compliance monitoring data.
- Principal Engineer for at multiple petroleum terminals in New York State for groundwater quality and surface water quality sampling and monitoring well gauging as required by the New York State Department of Environmental Conservation, as well as quarterly reporting for all sites. The work has also included collection of soil quality data at several sites and performance of an electromagnetic survey to support the divestiture and redevelopment of one of these sites. Based on these results, soil removal activities were performed at one of the former terminals in order to obtain regulatory closure of the site. Roux successfully completed the remedial activities to the satisfaction of the regulator and received closure for the client of the open spill number. Regulatory closure of another of these former terminals was obtained based upon the results of ongoing groundwater monitoring and reporting.
- Project Engineer for design of a 2.6-mgd groundwater treatment system at the Fireman's Training Center for Nassau County Department of Public Works on Long Island. The work included design of air strippers, exhaust stacks, liquid-phase GAC treatment units, and all chemical feed and storage facilities, including unit sizing, selection of materials of construction, equipment layout, and coordination with other disciplines. The work also included development of the "mass balance" for the facility.
- Task leader in charge of overseeing a bioventing pilot study conducted by a subconsultant, to treat contaminated vadose zone soils at the Fireman’s Training Center site in Nassau County, New York. The work included development of a preliminary design report for the full-scale implementation of bioventing at the site based upon the results of the pilot study.
- Project Engineer for the design, specification, construction, and operation of an air sparging and soil vapor extraction pilot at

the Long Island terminal of a large petrochemical distributor. The pilot was designed to treat contaminated ground water and vadose zone soils resulting from a one-million-gallon gasoline spill at the site. The work included development of the field sampling program and sampling and evaluation of various parameters to determine the pilot's radius of influence and effectiveness. The work also included performing data analysis and preparation of the pilot study report, which recommended full scale implementation of air sparging at the site. The site-wide implementation of air sparging and expansion of the site's existing vapor extraction system at the same Long Island petrochemical terminal was also part of the work. Responsibilities included design, specification, and layout of all mechanical equipment, vapor extraction, and air sparging wells and new vapor extraction/air sparging piping.

- Task leader responsible for investigating alternatives for the treatment of gasoline contaminated off-gas from air stripping operations a Long Island petrochemical terminal. Based on this evaluation, biofiltration was selected for piloting. Responsibilities included design of a pilot unit; development of sampling and data collection procedures; construction oversight and "troubleshooting" for the unit; coordination of data collection activities; and compilation and analysis of the pilot data.
- Project Engineer for the design of a 0.50-mgd groundwater treatment facility at a Long Island petrochemical terminal. Responsibilities included the design, specification, and layout of mechanical equipment, including the air stripping tower, vapor phase granular activated carbon off-gas treatment, centrifugal blowers, ductwork, influent pump, and concrete wet well. Responsible for shop drawing review during the construction phase.
- Project Manager for an investigation at a gasoline service station with soil and groundwater contamination. Responsible for reviewing and evaluating the work of another consultant that performed the soil and groundwater sampling and conducted remedial activities at the site including investigation summary reports; remedial designs; remediation progress reports; correspondence with regulators; and plans for future work at the Site. The work also included mapping the groundwater flow patterns in the area of the service station and mapping the areal and vertical extent of the groundwater contamination. Responsible for project coordination and budget management and tracking.
- Project Manager for the field investigation, feasibility evaluation, and remedial design at Superfund Site in Spring Valley, New York for the New York State Department of Environmental Conservation. The work included development of a work plan and site operations plan. The field investigations included Geoprobe soil borings; groundwater monitoring well installation; groundwater sampling; aquifer pump testing; and vapor extraction pilot testing. Work also included conducting the field operations for the vapor extraction pilot and producing a summary report of the field investigation results. The report presented an evaluation of the cost and feasibility of several alternatives for remediation of the site. It recommended reducing the level of effort of the remediation presented in the Record of Decision, based on lower levels of contamination encountered during the investigation. Duties also included project coordination, budget management and tracking, and development of subcontract agreements.
- Project Engineer for upgrades to the Spring Creek Auxiliary Water Pollution Control Plant for the City of New York. The work included the evaluation, design, and specification of a two-stage odor control system, chemical storage and feed facilities and new effluent disinfection system.
- Project Engineer responsible for preparation of design documents for the replacement of the sodium hypochlorite pumps and piping at the Mamaroneck Wastewater Treatment Plant for Westchester County Department of Environmental Facilities in New York.
- Project Engineer for design of upgrades to the New Rochelle Wastewater Treatment Facility for Westchester County Department of Environmental Facilities in New York. Designed upgrades to the main influent pump station, including rehabilitation of the existing influent pumps and replacement of the magnetic drives with new variable frequency drives. Responsibilities also included design of a submersible automatic duplex sump pump system, new primary sludge pumps and piping and new primary and secondary settling tank equipment. The work also included assisting the County during the bidding and contractor selection phase and preparing addenda to the contract documents.
- Project Manager for the construction of upgrades to the New Rochelle Wastewater Treatment Facility. Responsibilities included overseeing the shop drawing logging and distribution process; reviewing mechanical equipment shop drawings; addressing contractor questions regarding the contract documents; and coordinating with the resident engineer in the field and the electrical and general contractors.
- Project Engineer for the performance evaluation of the Harriman Wastewater Treatment Plant for the Orange County Department of Environmental Facilities and Services. Responsibilities included documentation of the existing conditions at the plant and evaluation of the historical and current performance of the plant with respect to its potential for expansion. A summary report was prepared, which included evaluations of the existing plant processes with respect to

standard design criteria, typical design practices and receiving water considerations. This summary report served as the basis for the facilities plan prepared as the next phase of the project.

- Project Engineer for the facilities plan for the upgrade of the Harriman Wastewater Treatment Plant. Responsibilities included evaluation of alternatives for expanding the plant's treatment capacity. A report was prepared, which recommended the conversion of the existing oxidation ditches to sequencing batch reactors (SBR) in order to increase the plant's treatment capacity to 6.0 mgd within the limited space available on the site.
- Project Engineer for the Gates-Chili-Ogden Pump Station and Force main design for Monroe County, New York. The design consisted of a new 36 mgd wet pit/dry pit pump station, influent sewer, and force main. Responsibilities included evaluating influent pumping conditions, and design of the influent sewer, manual influent bar racks and a duplex automatic submersible sump pump system for the station.
- Project Manager for the Flow Augmentation Needs Study (FANS) for Suffolk County Department of Public Works, New York. Responsibilities included coordinating and implementing the annual wetland monitoring effort for collection of data to characterize the vegetation communities and surface water and groundwater conditions at four freshwater wetland sites in Suffolk County. Duties also included maintaining a database of all vegetation data collected; developing and refining equations to characterize the groundwater table and wetlands vegetation in all monitoring areas; evaluating potential impacts of sewering on sensitive wetlands; and evaluating the need for any mitigation of these sites as triggered by vegetation changes correlated to groundwater drawdown. Responsibilities also included coordinating the production of a draft and final annual monitoring report summarizing the findings of the program.

PRESENTATIONS

Sparging Targets Submerged Residual Saturation Contamination, written with D. Bennett and L. Buchanan. Presented at the 66th New York Water Environment Federation Association Annual Meeting, New York, New York, February 1994.

Suffolk County Wetlands – Flow Augmentation Needs Study, written with M. A. Taylor and R. Southard. In Proceedings of the Annual Meeting, Hydrology and Hydrogeology of Urban and Urbanizing Areas. American Institute of Hydrology, April 1996.

PROFESSIONAL PROFILE

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EDUCATION
BS, Earth & Environmental Science, Lehigh University, 2018

Sarah Stern

Project Geologist

EXPERIENCE SUMMARY
Over five years of experience: Project Geologist, Staff Geologist, and Staff Assistant Geologist with Roux, Islandia, New York.

TECHNICAL SPECIALTIES
Project Management and Field Management of LNAPL remediation systems, LNAPL transmissivity, SPDES monitoring and compliance. Preparation and management of Remedial Action Work Plans and Construction Completion Reports. Remedial construction and soil excavation oversight, management of waste characterization and removal, environmental site assessments focusing on soil, sediment, groundwater, and soil vapor investigations using multiple sampling techniques.

REPRESENTATIVE PROJECTS

- Project manager and field supervisor for the ongoing investigation and remediation of a former lube and wax refinery and petroleum storage terminal in Queens, New York. Responsibilities include serving as technical consultant for the client assisting in the preparation of all regulatory submittals and overall strategy to achieve site closure through the NY State Spills Programs. Associated tasks include installation of LNAPL recovery systems, LNAPL transmissivity testing, scheduling and management of subcontractors and operation and maintenance team, preparation of New York State Department of Environmental Conservation (NYSDEC) quarterly monitoring reports, construction completion reporting, Remedial Action Work Plan compliance and other regulatory deliverables, organizing monitoring and sampling events.
- Project manager for environmental compliance monitoring at a power generating station in Long Island City, New York. Responsibilities include project management of weekly sampling for SPDES compliance, fluid elevation level monitoring, product recovery, monthly progress reports, semi-annual groundwater sampling, and annual groundwater sampling for Major Oil Storage Facility permit compliance.
- Project management support for ecological and environmental consulting support for the redevelopment of a 32-acre underutilized property to create a Port Facility for offshore wind material staging, assembly, and pre-commissioning of wind turbine generators (WTGs) and other offshore wind (OSW) components. This is a multi-year project and services supported to date have included sediment analysis and evaluation and dredge material management, preparation of a Remedial Action Plan and Construction Health and Safety Plan, essential fish habitat assessment support, joint permit application permitting support.
- Project manager for due diligence project in Brooklyn, NY. Activities include planning and implementation of Phase I and Phase II site assessments, dye tracer testing, lead based paint, asbestos and hazardous materials surveys, development of remedial cost estimates for planning and negotiation, SSDS pilot testing and installation.
- Project manager for a 40-acre former metals manufacturing facility in Staten Island under the NYSDEC Voluntary Cleanup program. Responsibilities include scheduling and management of Site inspection events and preparing annual progress reports.
- Project Manager and field manager responsible for Phase II Site Assessments and Remedial Investigations at various sites in Manhattan and Brooklyn including monitoring well installation and corresponding soil, soil vapor, and groundwater sampling and the preparation of investigation reports.

- Project and field management support for a site handling a petroleum hydrocarbon product release from a former refinery and petroleum storage terminal in Brooklyn, New York. Field responsibilities have included participating in quarterly site-wide fluid level gauging, annual groundwater Monitored Natural Attenuation sampling, soil vapor sampling and annual LNAPL transmissivity testing at select wells throughout the site. Reporting tasks include preparation of groundwater and soil vapor reports, assistance with quarterly progress reports, project planning, data tracking, and notices/letters to NYSDEC.
- Field manager responsible for providing environmental support for LIRR Railyard site including overseeing soil excavation and movement, overseeing disposal of soil, proper handling of waste manifests, waste characterization and daily reporting of site activities for development of residential buildings in Pacific Park, Brooklyn, New York.
- Assisted in the implementation of a large-scale waste characterization program for a major redevelopment project in Elmont, New York. Project included coordination and oversight of in situ waste characterization sampling of over 405,000 tons of soil.
- Participated in research for the Tecumseh insurance litigation project including reviewing and entering nearly 2,000 contractor invoices.
- Implemented Community Air Monitoring Programs during excavation and disposal activities at various sites in Manhattan and Brooklyn. Responsible for monitoring 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 and for signing manifests for the shipment of soil to approved facilities.
- Site Safety Officer for various remedial investigation sites. Responsibilities include preparation of health and safety plans (HASPs), job safety analysis (JSA) documents development and review, onsite safety meeting management, safety document preparation (Lessons Learned, Near Loss, Field Audits, etc.), and planning/execution of corrective actions.

PROFESSIONAL TRAININGS

OSHA 40-Hour HAZWOPER Training, 2018

OSHA 10-Hour Construction Safety Training, 2018

Transportation Worker Identification Credential, 2018 & 2023

OSHA 30-Hour Construction Safety Training, 2019

First Aid and CPR Training, 2021

DOT Hazmat/RCRA Training, 2023

PROFESSIONAL PROFILE

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EDUCATION
BS, Physics, Stony Brook University, 2014

Valerie Sabatasso

Senior Scientist I

EXPERIENCE SUMMARY
Over eight years of experience: Senior, Project, and Staff Scientist at Roux, Islandia, New York.

TECHNICAL SPECIALTIES
Design, implementation, and management of Remedial Investigations and Remedial Actions for sites in regulatory programs including United States Environmental Protection Agency Superfund program, New York State Brownfields Cleanup Program, and New York City Office of Environmental Remediation Voluntary Cleanup Program; Management of due diligence Phase I & II Environmental Site Assessments; Preparation and management of Remedial Investigation Work Plans, Remedial Investigation Reports, Remedial Action Work Plans, and Remedial Action Reports; Investigation and evaluation of petroleum-related contamination and per- and poly fluoroalkyl substances (PFAS)-related contamination; Management of Environmental Site Assessments focusing on soil, groundwater, and soil vapor investigations. Performance of sampling for the following media: soil, groundwater, surface water, soil vapor, and sediment; and excavation sampling and oversight and waste tracking.

REPRESENTATIVE PROJECTS

- Project Manager for the ongoing large scale excavation of a former airport long-term parking lot in Queens, New York. Redevelopment includes a unique, large-scale warehouse meant to ease transportation of goods for the airport. The project is enrolled in the NYC OER VCP, which has transferred over 70,000 CY, most of which has been through the Clean Soil Bank, and will obtain a Track 1 Cleanup. Management of excavation oversight has included remediation for the closure of 28 drywells, nonfibrous asbestos abatement, and the removal of three unregistered USTs discovered during excavation.
- Project Manager for a series of clusters of sites across East New York and the Bronx, New York. Currently, seven Phase I ESAs and eight Phase II ESAs have been completed have been completed. A RAWP is being completed for one of the properties, which has an E designation and is under guidance of the NYCOER.
- Project Manager for an affordable housing redevelopment site under NYCDEP guidance in Bronx, New York. This project included a due diligence environmental investigation; remedial investigations (soil, groundwater, and soil vapor); site-wide in situ waste characterization sampling program; a Remedial Action Work Plan, the management of soil and bedrock excavation, and a Remedial Closure Report.
- Project Manager for the ongoing remediation of over 20 drywell structures at a strip mall facility in Setauket, New York. The drywell remediation project was conducted in accordance with the Suffolk County Article XII requirements and entailed coordination with Suffolk County Department of Health Services (SCDHS).
- Project Manager for remedial investigation at a former gas station located in Brooklyn, New York. Historical site operations adversely affected the subsurface through petroleum hydrocarbon impacts. Responsibilities included creating and managing an initial sample plan for soil and groundwater, designing a remediation plan that included a small scale excavation and the use of RegenOx™ oxygen-releasing pellets, and maintaining communication between subcontractors.
- Field Manager for the installation of over 400 points for a Vapor Mitigation System in an active warehouse in New Jersey. Responsible for semi-annual indoor air sampling consisting of 130

air samples collected following each tenant's specific schedule as well as consistent SSDS monitoring to ensure the system is running correctly and efficiently.

- Project Manager for underground storage tank (UST) discovery, inventory, and removal. Field responsibilities involved subcontractor oversight for excavation and removal of UST, tank cleaning, and waste management.
- Field Manager responsible for implementation of a remedial investigation at a former Manufactured Gas Plant (MGP) site in Brooklyn, New York. Tasks included management of remedial investigation, collection of forensic samples, and management of on-site Health and Safety.
- Staff Scientist as a former alumina manufacturing facility in Corpus Christi, Texas. Tasks included intrusive site investigation, health and safety management, collection of samples for laboratory analysis from various media including soil, sediment, surface water, and groundwater, and completion of pH tests of surface water above 13 pH. Areas of concern being addressed by the investigation included the manufacturing area, wastewater percolation ponds, on-site landfills, and various surface water features.
- Performed numerous Phase I Environmental Site Assessments for due diligence in connection with property transfers for the Metro New York Area. Most properties included commercial properties, former automobile service stations, and residential/office buildings.
- Field manager responsible for soil excavation and waste removal oversight for development of residential buildings in Brooklyn, New York. Responsibilities included overseeing excavation, organization and proper handling of waste manifests and ensuring compliance with the Site Environmental Management Plan.
- Field Manager responsible for implementation of Remedial Investigation Work Plan (RIWP) at an industrial warehouse in Brooklyn, New York that is currently being developed for residential apartments. Responsible for groundwater level monitoring which included recording, entering, and reviewing data with in situ electronic transducers.
- Field manager responsible for implementation of Community Air Monitoring Plan (CAMP) during excavation at a hospital in Greenwich Village, 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. Intrusive activities included removing concrete and rebar and backfilling with clean soil. In addition to CAMP activities, assisted project engineer and construction manager with contractor oversight, material review, health and safety oversight, and daily reporting.
- Staff Scientist responsible for Phase II Site Assessment and preparation of investigation reports for soil boring installation, monitoring well installation and corresponding soil, soil vapor, and groundwater sampling.
- Project execution manager for various projects at multiple locations in Manhattan, the Bronx, Staten Island, Queens, and Brooklyn, New York. Activities include subcontractor coordination, scheduling, bottleware and sample management, subcontractor contract preparation, scope of work project design, subcontractor oversight, system operations and maintenance, tenant relations, and health and safety management.
- Site Safety Officer for various remedial investigation sites. Responsibilities include preparation of health and safety plans (HASPs), job safety analysis (JSA) documents development and review, onsite safety meeting management, safety document preparation (Lessons Learned, Near Loss, Field Audits, etc.), and planning/execution of corrective actions.

PROFESSIONAL TRAININGS

OSHA 40-hour HAZWOPER Training, 2015

OSHA 30-hour Construction Safety Training, 2019

OSHA 8-hour Refresher Training, 2016 - 2019

OSHA 10-hour Construction Safety Training, 2015

First Aid and CPR Certified

Transportation Worker Identification Credential (TWIC)

Loss Prevention System (LPS) Awareness, 8-Hour Certified

MTA LIRR Roadway Worker Protection Training

ATTACHMENT 2

NYSDEC PFAS 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 PFOS (ppb) 3.7	PFOA (ppb) 0.8 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 (…) 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 (…) 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. " [Interim SCO Table] "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. 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. 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. [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: a. any other individual PFAS (not PFOA or PFOS) is detected in water at or above 100 ng/L; or 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

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.

ATTACHMENT 3

Laboratory's Standard Operating Procedures

Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
Volatile Organic Compounds in Ambient Air	TO15	Air Summa Can	Dichlorodifluoromethane	75-71-8	2.47	0.544	1.98	ug/m3	61	142	25	70	130			
			Chlorodifluoromethane	75-45-6	1.77	0.424	1.41	ug/m3	60	147	25	70	130			
			1,2-Dichlorotetrafluoroethane	76-14-2	1.40	0.336	1.12	ug/m3	71	141	25	70	130			
			Chloromethane	74-87-3	1.03	0.310	0.826	ug/m3	56	141	25	70	130			
			n-Butane	106-97-8	1.19	0.475	0.951	ug/m3	53	151	25	70	130			
			Vinyl chloride	75-01-4	0.200	0.0537	0.409	ug/m3	61	135	25	70	130			
			1,3-Butadiene	106-99-0	0.442	0.0863	0.354	ug/m3	58	139	25	70	130			
			Bromomethane	74-83-9	0.777	0.276	0.621	ug/m3	72	124	25	70	130			
			Chloroethane	75-00-3	1.32	0.475	1.06	ug/m3	68	130	25	70	130			
			Bromoethene(Vinyl Bromide)	593-60-2	0.875	0.219	0.700	ug/m3	75	125	25	70	130			
			Trichlorofluoromethane	75-69-4	1.12	0.281	0.899	ug/m3	70	129	25	70	130			
			1,1,2-Trichlorotrifluoroethane	76-13-1	1.53	0.406	1.23	ug/m3	70	121	25	70	130			
			1,1-Dichloroethene	75-35-4	0.200	0.103	0.634	ug/m3	68	120	25	70	130			
			Acetone	67-64-1	11.9	3.80	9.50	ug/m3	54	154	25	70	130			
			Isopropyl alcohol	67-63-0	12.3	3.93	9.83	ug/m3	53	142	25	70	130			
			Carbon disulfide	75-15-0	1.56	0.405	1.25	ug/m3	71	138	25	70	130			
			3-Chloropropene	107-05-1	1.57	0.376	1.25	ug/m3	50	150	25	70	130			
			Methylene Chloride	75-09-2	1.74	0.625	1.39	ug/m3	59	137	25	70	130			
			tert-Butyl alcohol	75-65-0	15.2	3.64	12.1	ug/m3	66	132	25	70	130			
			Methyl tert-butyl ether	1634-04-4	0.721	0.130	0.577	ug/m3	70	127	25	70	130			
			trans-1,2-Dichloroethene	156-60-5	0.793	0.0912	0.634	ug/m3	69	137	25	70	130			
			n-Hexane	110-54-3	1.76	0.388	1.41	ug/m3	63	138	25	70	130			
			1,1-Dichloroethane	75-34-3	0.809	0.101	0.648	ug/m3	66	130	25	70	130			
			Methyl Ethyl Ketone (2-Butanone)	78-93-3	1.47	1.45	1.47	ug/m3	72	124	25	70	130			
			cis-1,2-Dichloroethene	156-59-2	0.200	0.0833	0.634	ug/m3	72	121	25	70	130			
			Bromochloromethane	74-97-5				ug/m3								
			Chloroform	67-66-3	0.977	0.200	0.781	ug/m3	73	124	25	70	130			
			Tetrahydrofuran	109-99-9	14.7	3.83	11.8	ug/m3	60	149	25	70	130			
			1,1,1-Trichloroethane	71-55-6	1.09	0.240	0.873	ug/m3	72	127	25	70	130			
			Cyclohexane	110-82-7	0.688	0.200	0.551	ug/m3	76	124	25	70	130			
			Carbon tetrachloride	56-23-5	0.220	0.138	1.01	ug/m3	71	133	25	70	130			
			2,2,4-Trimethylpentane	540-84-1	0.934	0.178	0.748	ug/m3	68	131	25	70	130			
			Benzene	71-43-2	0.639	0.141	0.511	ug/m3	73	119	25	70	130			
			1,2-Dichloroethane	107-06-2	0.809	0.376	0.648	ug/m3	68	135	25	70	130			
			n-Heptane	142-82-5	0.820	0.225	0.656	ug/m3	60	142	25	70	130			
			1,4-Difluorobenzene	540-36-3				ug/m3								
			Trichloroethene	79-01-6	0.200	0.134	0.860	ug/m3	73	122	25	70	130			
			Methyl methacrylate	80-62-6	2.05	0.573	1.64	ug/m3	73	129	25	70	130			
			1,2-Dichloropropane	78-87-5	0.924	0.434	0.739	ug/m3	69	128	25	70	130			
			1,4-Dioxane	123-91-1	18.0	0.296	14.4	ug/m3	66	129	25	70	130			
			Bromodichloromethane	75-27-4	1.34	0.335	1.07	ug/m3	75	127	25	70	130			
			cis-1,3-Dichloropropene	10061-01-5	0.908	0.204	0.726	ug/m3	74	125	25	70	130			
			4-Methyl-2-pentanone (Methyl isobutyl ketone)	108-10-1	2.05	0.533	1.64	ug/m3	58	144	25	70	130			
			Toluene	108-88-3	0.754	0.234	0.603	ug/m3	75	122	25	70	130			
			trans-1,3-Dichloropropene	10061-02-6	0.908	0.245	0.726	ug/m3	74	128	25	70	130			
			1,1,2-Trichloroethane	79-00-5	1.09	0.404	0.873	ug/m3	75	126	25	70	130			
			Tetrachloroethene	127-18-4	1.36	0.142	1.09	ug/m3	70	125	25	70	130			
			Methyl Butyl Ketone (2-Hexanone)	591-78-6	2.05	0.615	1.64	ug/m3	57	143	25	70	130			
			Dibromochloromethane	124-48-1	1.70	0.537	1.36	ug/m3	73	125	25	70	130			
			1,2-Dibromoethane	106-93-4	1.54	0.323	1.23	ug/m3	78	122	25	70	130			
			Chlorobenzene-d5	3114-55-4				ug/m3								
			Chlorobenzene	108-90-7	0.921	0.203	0.737	ug/m3	76	119	25	70	130			
			Ethylbenzene	100-41-4	0.868	0.300	0.695	ug/m3	74	122	25	70	130			
			m,p-Xylene	179601-23-1	2.17	0.413	1.74	ug/m3	76	121	25	70	130			
			o-Xylene	95-47-6	0.868	0.274	0.695	ug/m3	73	123	25	70	130			
			Styrene	100-42-5	0.852	0.251	0.682	ug/m3	74	125	25	70	130			
			Bromoform	75-25-2	2.07	1.24	1.65	ug/m3	53	149	25	70	130			
			Cumene	98-82-8	0.983	0.202	0.787	ug/m3	73	123	25	70	130			
			1,1,2,2-Tetrachloroethane	79-34-5	1.37	0.295	1.10	ug/m3	74	126	25	70	130			
			n-Propylbenzene	103-65-1	0.983	0.231	0.787	ug/m3	73	127	25	70	130			
			4-Ethyltoluene	622-96-8	0.983	0.241	0.787	ug/m3	75	129	25	70	130			
			1,3,5-Trimethylbenzene	108-67-8	0.983	0.231	0.787	ug/m3	72	126	25	70	130			
			2-Chlorotoluene	95-49-8	1.04	0.238	0.828	ug/m3	74	126	25	70	130			
			tert-Butylbenzene	98-06-6	1.10	0.258	0.878	ug/m3	71	125	25	70	130			
			1,2,4-Trimethylbenzene	95-63-6	0.983	0.393	0.787	ug/m3	71	129	25	70	130			
			sec-Butylbenzene	135-98-8	1.10	0.247	0.878	ug/m3	70	128	25	70	130			
			4-Isopropyltoluene	99-87-6	1.10	0.335	0.878	ug/m3	68	130	25	70	130			
			1,3-Dichlorobenzene	541-73-1	1.20	0.445	0.962	ug/m3	69	131	25	70	130			
			1,4-Dichlorobenzene	106-46-7	1.20	0.535	0.962	ug/m3	67	132	25	70	130			
			Benzyl chloride	100-44-7	1.04	0.456	0.828	ug/m3	60	136	25	70	130			
			n-Butylbenzene	104-51-8	1.10	0.604	0.878	ug/m3	65	137	25	70	130			
			1,2-Dichlorobenzene	95-50-1	1.20	0.397	0.962	ug/m3	68	129	25	70	130			
			1,2,4-Trichlorobenzene	120-82-1	3.71	2.45	2.97	ug/m3	50	150	25	70	130			
			Hexachlorobutadiene	87-68-3	2.13	1.17	1.71	ug/m3	58	130	25	70	130			
			Naphthalene	91-20-3	2.00	1.57	2.10	ug/m3	50	150	25	70	130			
			BFB	460-00-4	1.43	1.43	1.43	ug/m3								

eurofins	Always check on-line for validity. **EPA Methods TO-15 and TO-3, Determinations of VOCs in Ambient Air**	Level: **Standard Operating Procedure**
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16.6) Appendix A: Terms and Definitions
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Revision Log

Revision: 16.0	**Effective Date**	This version
Section	**Justification**	**Changes**
Throughout	Added	Cross-reference links to related documents in D4
Section 5	Updated	Current standard Safety language
Sections 6 & 7	Added	Catalog # disclaimer and storage conditions
Section 10.2.2.1	Added	Percent Error acceptance criteria for individual points in the calibration

Revision: 15.0	**Effective Date**	21 April 2023
Section	**Justification**	**Changes**
Entire Document	Format	Update to D4 format
Section 10.2.4	Updated	Removed reference to DoD specific QA requirements.
Section 10.3.4	Added	Client specific QA requirements

Previous revisions maintained by QA Department

References

- EPA Compendium Method TO-15, "Determination of Volatile Organic Compounds in Ambient Air using Specially Prepared Canisters and Analyzed by Gas Chromatography/Mass Spectrometry", US EPA, January, 1999.
- USEPA Compendium Method TO-3 “Method for the Determination of Volatile Organic Compounds In Ambient Air Using Cryogenic Pre Concentration Techniques and Gas Chromatography with Flame Ionization and Electron Capture Detection”, USEPA, April 1984
- New Jersey Department of Environmental Protection Site Remediation Program Vapor Intrusion Technical Guidance, Version 3.1, March 2013.

Cross References

- *SOP52862 Quality Assurance Manual*
- *SOP48255 Canister Cleaning, Certification and Sampling Kit Preparation*
- *QP46060 NDSC HSE Manual*
- *SOP49961 Burlington Corporate Safety Manual Addendum*
- *SOP52272 Traceability, Handling and Storage of Standards and Reagents*
- *SOP49959 Sample Management*
- *SOP49967 Nonconformance and Corrective Action Procedures*
- *SOP43862: Manual Integrations*
- *SOP49966 Management, Retention Storage and Archive of Data Records*
- *SOP52264 Employee Training and Analyst Demonstration of Proficiency*
- *SOP47863 Tracking and Collecting of Hazardous Waste*
- *SOP48269 Procedure for Acceptance and Handling of USDA Regulated Domestic and Foreign Soil*

1) Scope and Application

This SOP describes the laboratory procedure for the analysis of polar and non-polar volatile organic compounds (VOCs) in ambient and non-ambient air. The procedure is applicable to those VOCs that have been evaluated by the laboratory for their consistent performance in meeting the control criteria put forth in Compendium Method TO-15.

While the compendium method is specifically written for the analysis of ambient air samples collected in leak-free passivated stainless-steel canisters, it may be applied to the analysis of samples that have employed the use of other collection devices such as Tedlar bags and are from sources other than ambient air such as indoor air, vapor intrusion impacted air, soil gas, landfill gas, gas cleaning apparatuses and stack emissions.

This procedure may also be used to report a variety of carbon ranges, constituent groups like TVOC (total volatile organic compounds), or as unresolved complex mixtures (e.g. Total Hydrocarbons).

1.1) Analytes, Matrix(s), and Reporting Limits

The target compound list and reporting limits for each compound are provided in the Appendix.

2) Summary of Method

An aliquot of sample is pulled from the canister through a solid multi sorbent bed trap which reduces the water content of the sample. The sample is thermally desorbed, and the VOCs are carried onto a GC column coupled to a mass spectrometer. Compounds are identified by comparison of the mass spectra for individual peaks in the total ion chromatogram to the fragmentation patterns of ions corresponding to VOCs including the intensity of primary and secondary ions as well as the patterns of stored spectra acquired under similar conditions.

The concentration of the target compound is calculated by internal standard technique using the average response factor or slope and intercept of that compound as determined by the initial calibration.

If the laboratory has modified the method, a list of these adjustments may be found in the Method Modifications section of this SOP.

3) Definitions

A list of terms and definitions are provided in the Appendix. Refer to the Laboratory *SOP52862 Quality Assurance Manual* (QAM) for additional Glossary of Terms, Definitions, and Acronyms.

4) Interferences

Contamination may occur if canisters or other equipment is not properly cleaned before use. The laboratory procedures for canister and flow controller cleaning are provided in *SOP48255 Canister Cleaning, Certification and Sampling Kit Preparation*.

5) Safety

Employees must abide by the policies and procedures in the Eurofins NDSC-US EHS-*QP46060 NDSC HSE Manual*, the *SOP49961 Burlington Corporate Safety Manual Addendum* and this document. This procedure may involve hazardous material, operations and equipment.

This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

5.1) Specific Safety Concerns or Requirements

The analytical system contains zones with elevated or depressed temperatures that are capable of causing injury upon direct contact. The analyst needs to be aware of the locations of those zones and allow them to return to room temperature prior to maintenance activities or take measures to avoid contact with hot and/or cold surfaces. There are areas of high voltage in the analytical system. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

Liquid nitrogen (LN2) is used for cryogenic purposes. In addition to avoiding contact with LN2 cooled surfaces, analysts must be aware of the potential for oxygen depletion in a confined space in the event of an unexpected large release of the product. Users should evacuate a confined space in which large amounts of LN2 have been released.

Sample canisters are occasionally pressurized for cleaning or sample dilution purposes. Lab systems are designed to ensure that the cans are not pressurized above 40 psi. Eye protection must be worn when cans are pressurized in the event of a canister failure.

5.2) Primary Materials Used

There are no materials used in this method which have a serious or significant hazard rating.
NOTE: This section does not include all materials used in the method. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the SDS for each material before using it for the first time or when there are major changes to the SDS.

6) Equipment and Supplies

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts are cautioned to be sure equipment meets the specification of this SOP.

6.1) Sampling Equipment

- 6L and 1L SUMMA® Canisters: Leak-Free, Passivated Stainless Steel, with Swagelok DSS4 Valves, or equivalent. Maximum rated pressure 40 psig.
- 6L SUMMA® Canisters: Silicon Lined-Leak-Free, Passivated Stainless Steel, with Swagelok DSS4 valves or equivalent. Maximum rated pressure 40 psig.
- Flow Controllers: Restek Catalog #24239 or equivalent.
- Flow Controller Orifice: Various sizes ranging from 0.008” to 0.060”, Restek or equivalent.
- Flow Controller Vacuum Gauges: Capable of measuring vacuum to an absolute vacuum of -30” Hg, and pressure up to 30 psi, Grainger Catalog #5WZ37 or equivalent
- Rain Guard: Stainless Steel Tubing ¼”, 10ft. Grainger or Equivalent. Cut 8” and bend into a J shape using a pipe bender.
- Stainless Steel Pre-Filter (7 um): Swagelok Catalog# SS-4F-T7-7 or equivalent
- Teflon Tape: Home Depot Brand or equivalent.

6.2) Analytical System

- Mass Spectrometer: Agilent 5973, 5975, 5975B, 5977C MSD or equivalent.
- Gas Chromatograph: Agilent 6890, 7890, 8890 or equivalent.
- VOC Autosampler: Entech 7016CA or equivalent.
- Cryogenic Concentrator: Equipped with an electronic mass flow controller that maintains a constant flow for carrier gas and sample over a range of 0-200 cc/min. Entech 7100A, 7200, 7200A or equivalent.
- Low Pressure Liquid Nitrogen: Air Gas or equivalent.
- Glass Bead Cryotrap: Capable of effectively removing water while trapping polar and non-polar compounds. Entech catalog# 01-04-11320.
- TENAX Sorbent Trap: Capable of removing CO2 and trapping the polar and non-polar compounds. Entech catalog # 01-04-11330.Primary Column: Fused silica capillary column (60 m x 0.32 mm x 1.8 µm), Restek RTX-624 or equivalent.
- Data System: PC software for Entech instrumentation. Hewlett-Packard ChemStation data acquisition software, Environment Testing Chrom and Environment Testing LIMS (TALS).
- NIST Mass Spectral Library and Search Program, 2020 release or newer

6.3) Cleaning System

- Canister Cleaner Module and Software: Capable of filling canisters with humidified air and evacuating canisters to 50 mtorr, Entech Model 3100A or equivalent.
- Vacuum Pump: Capable of evacuating sample canisters to full vacuum. Vacuubrand or equivalent.
- Cleaning Manifold: Equipped with stainless steel and Teflon transfer lines and connections for cleaning up to twelve canisters simultaneously.
- Heating Belts: Individual thermal-stated heating belts used to heat canisters to 100ºC during the manifolds cleaning cycles. Entech or equivalent.
- Cleaning oven: Capable of cleaning 6 Summa Cans simultaneously at a temperature of 100ºC. Entech or equivalent.
- Flow Controller Cleaning Manifold: Capable of flushing hot Nitrogen through 24 flow controllers simultaneously for cleaning.

6.4) Miscellaneous Supplies

- Mass Flow Controller, NIST Traceable: Capable of flow rate of 70 mL/min, Alicat MC5005CCM or equivalent. Use for the preparation of calibration and working standards.
- Syringes: Gas tight with a Luer-Lok tip, assorted sizes ranging from 1.0 mL to 1.0 L, SGE or equivalent.
- Digital Pressure Gauges, NIST Traceable: Capable of measuring pressure in the range of -30" Hg to 100 psi, Dwyer Models DPGA-12 and 67100 or equivalent
- Digital Flow Meter, NIST Traceable: Alltech or equivalent.

- Nitrogen Gas
- Digital Stop watch, NIST traceable, Thomas Scientific or equivalent

7) Reagents and Standards

Catalog numbers listed in this SOP are subject to change at the discretion of the vendor. Analysts may use equivalent-quality materials based on factors including availability. Permanent changes will be noted in future SOP revisions. All reagent and standards must be labeled with their Name and/or ID #; the Expiration Date; and the Date Prepared.

All reagents and standards are stored at room temperature in the Air Lab work area, unless noted otherwise.

For additional information refer to the *SOP52272 Traceability, Handling and Storage of Standards and Reagents*

7.1) Reagents

- Argon - Gas off bulk Liquid Argon tank. UHP Air Gas or equivalent vendor
- Nitrogen- Gas off bulk Liquid Nitrogen tank. UHP Air Gas or equivalent vendor
- Helium- UHP from Airgas or equivalent
- VOA free water

7.2) Standards

Certified stock standard solutions are purchased from approved commercial vendors. From these, intermediate and working standard solutions are prepared by diluting a known volume of standard in an appropriate solvent to the desired final concentration.

Formulations for the preparation of intermediate and working standard solutions are provided in the Appendix. Unless otherwise specified, assign an expiration date of 12 months from date of preparation, unless the parent component expires earlier in which case use the earliest expiration date.

There are small variations in analyte concentrations in the preparation of air standards. For simplicity, the nominal concentrations are referenced in this SOP.

Note: *When entering the concentrations of source standards in TALS, use the nominal concentration and not the actual concentration. Unless the actual has a > 15% difference from the nominal value. In these cases, use the actual value.*

Purchase the following stock standard mixtures from commercial vendors:

- Mixed Gas Stock Standard: - Commercially prepared standard that includes internal standard and tune standard compounds: Bromofluorobenzene, Bromochloromethane, 1,4-Difluorobenzene, and Chlorobenzene-d5, at a concentration of 100 ppbv each. Linde or equivalent.
- Calibration Stock Standard: - Commercially prepared custom gaseous stock standard that includes all target analytes except ethanol at a concentration of 1.0 ppmv. Linde or equivalent.
- Calibration Ethanol Neat Material. >99.5%
- ICV / LCS Stock Standard: - Custom made gaseous stock standard prepared from a different lot(s) of the source material(s) used to manufacture the calibration stock standard. The ICV/LCS stock standard includes all target analytes at a concentration of 1.0 ppmv. Linde.
- ICV/LCS Ethanol Neat material. >99.5% from a source other than the calibration source.

Prepare calibration and working standards mixtures by diluting a known volume of the stock standard with nitrogen to a specified volume into a humidified summa canister. The summa canister is humidified by adding 100ul of VOA free reagent water. The volume of the standard added to the canister is calculated using the set flow rate of 70mL/min using a mass flow meter multiplied by the time of the standard addition, plus the inclusion of the 25ml volume for the tubing connecting the mass flow meter to the summa canister. The formulations for standard preparation are provided in the Appendix along with recommended expiration dates and storage conditions.

Each stock standard is assigned a 1-year expiration date from manufacture and recertified annually. See *SOP52272 Traceability, Handling, and Storage of Standards and Reagents* for details on the recertification process. The ethanol neat material is assigned the expiration date given by the manufacturer.

If a new standard cannot be received prior to the expiration of the standard presently in use, the lab will notify the QA manager. The QA manager will perform a control chart review (see *SOP49965 Control Charts*). If the standard is determined to be acceptable, the lab may assign a new expiration date of not more than 30 days from the date of the last data point in the control chart review. When the new standard ID is created in TALS, the COA and the Control Chart verifying the standards stability will be attached to the reagent ID.

8) Sample Collection, Preservation, Shipment and Storage

The laboratory does not perform sample collection, so these procedures are not included in this SOP. Sampling requirements may be found in the published reference method.

Matrix	Sample Container	Minimum Sample Size	Preservation	Holding Time	Reference
Air	1L or 6L Passivated Summa Canister	1L	NA	30 days from collection	EPA TO-15

1-liter Tedlar bags are only provided upon client request; however, clients are discouraged from using Tedlar bags due to the shortened holding time of 72 hours from collection.

All samples should be collected in passivated stainless-steel canisters that have been certified clean prior to sampling. The laboratory will provide certified clean canisters to the client upon request. The procedures for clean canister certification are provided in Laboratory SOP 48255: Canister Cleaning, Certification and Sampling Kit Preparation.

The laboratory can also provide flow controllers set to the appropriate flow rate for the sampling time required by the client.

The laboratory ships air canisters in custom made boxes. The boxes are equipped with custom-made foam inserts to hold the pre-set flow-controllers. The shipping materials are designed to prevent damage of equipment to and from the sampling site. The laboratory checks the equipment to ensure it is in proper working order before shipment to the client and additional checks are performed on return of the equipment to the laboratory. Sampling instructions are provided with each sampling kit. The sampling crew is advised to handle the sampling equipment using the instructions provided by the laboratory to ensure optimum performance.

The laboratory's sample acceptance policy for air samples in canisters requires that the sampling crew record the ID of the flow controller used for sample collection on the tag attached to each canister, but the association may also be recorded on the Field Test Data Sheet or a COC. With this information the laboratory can review the history of use of the FC as needed to troubleshoot potential equipment problems. Without the association, the history of use of the FC is unknown. The laboratory strongly recommends that field samplers be instructed to provide this information for each sampling event.

The return pressure of each canister should be between -10" Hg to -1"Hg, except for "grab" samples or samples with a collection rate of 100 or 200 mL/min, which must have a pressure greater than or equal to -10" Hg. The residual vacuum criteria ensure that the sample was collected over the time period the flow controller was calibrated for.

9) Quality Control

9.1) Sample QC

The following quality control samples are prepared with each batch of samples.

QC Item	Frequency	Acceptance Criteria
Method Blank (MB)	1 in 20 or fewer samples	See QC Summary Table
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	See QC Summary Table
Internal Standard (ISTD)	Every Sample	See QC Summary Table
Laboratory Control Sample Duplicate (LCSD)	Client request or if insufficient sample for Sample Duplicate	See QC Summary Table
Sample Duplicate (SD)	1 in 20 or fewer samples if sample volume allows	See QC Summary Table
Trip Blank (TB)	Client Request	See QC Summary Table

NOTE: *The compendium reference method does not require the analysis of a laboratory control sample (LCS) or provide criteria for the evaluation of an LCS. The laboratory performs an LCS at the above-mentioned frequency as an evaluation of percent recovery in a blank matrix. The laboratory uses statistically derived control limits for the LCS.*

Unless otherwise specified by the client during project initiation, the LCSD will be used to measure precision only. The LCS will be used for evaluations for percent recovery and to determine if corrective action is necessary.

9.2) Instrument QC

The following instrument QC is performed:

QC Item	Frequency	Acceptance Criteria
Tune Standard (BFB)	Each Analytical Window	See Appendices
Initial Calibration (ICAL)	Initially; when ICV or CCV fail	See Appendices
Initial Calibration Verification (ICV)	Once, after each ICAL	See Appendices
RT Window Establishment	Once per ICAL	See Appendices

Relative Retention Time (RRT)	With each sample	See Appendices
Continuing Calibration Verification (CCV)	Daily, after each BFB	See Appendices

10) Procedure

10.1) Support Equipment Calibration

Verify the calibration is valid for the following;

- Mass flow controller used to prepare standards.
- Digital flow meter used to set and check the flow rates of the FC(s) used for sample collection.
- Digital pressure gauges used to check return canister pressure.
- Stopwatch used to measure standard addition time is current to the year.

Immediately notify the QA department if the calibration is not current and wait for further instruction. Equipment whose calibration has expired may not be used without documented approval from the QA department.

NOTE: *The QA department schedules the annual calibrations of the support equipment and maintains all Certificates of Calibration. The flow controllers are checked against a NIST traceable standard. This check is performed by the manufacturer of the equipment, when possible, or by an approved vendor that provides certification service.*

10.2) Instrument Calibration

All Calibration Standards and Quality Control samples have the same concentration of Internal Standards and Surrogates that are added for field sample analysis. Refer to the Analytical Sequence section for additional information.

10.2.1) Tune Standard

Analyze a tune standard (BFB) prior at the beginning of each analytical window. The tune standard is a commercially prepared mixed gas stock standard that includes Bromofluorobenzene (BFB) at a concentration of 100 ppbv.

To analyze the tune standard:

1. Establish the instrument operating conditions specified in the Sample Analysis section of this SOP. Once established these settings must be maintained for the analysis of all quality control and environmental samples.
2. Attach the mixed gas stock standard to the Entech concentrator by attaching the cylinder to the line dedicated for introduction of the internal standard (ISTD). The concentrator directly injects 20 mL of the 100 ppbv stock standard onto the instrument to yield an on-column concentration of 10 ppbv.
3. Acquire the data and evaluate the results against the acceptance criteria in the Tune Standard Criteria table in the appendix.
4. Criteria must be met prior to further analysis.
5. The official start time of the 24-hour analytical window is the injection time of a passing tune standard. All samples must be injected within 24 hours of that time.

NOTE: *The data processing system averages three scans (apex scan, scan prior, and scan following) and performs background subtraction of the single scan prior to the elution of BFB.*

10.2.2) Initial Calibration (ICAL)

The instrument must be calibrated with a minimum of five calibration standards for each target analyte at concentrations that span the working range of the method.

The laboratory routinely analyzes 8 standards at the recommended concentrations of 0.035, 0.20, 0.50, 5.0, 10.0, 15.0, 20 and 40 ppbv, except for Ethanol. For Ethanol, a 6-point curve is analyzed at the following concentrations: 5, 10, 15, 20, 40, and 100 ppbv.

Note: *These are nominal concentrations, dependent on parent can stock concentrations. The nominal 0.035 ppbv concentration is prepared to ensure the actual low calibration standard is below the reporting limit (RL).*

Even though eight calibration standards are routinely analyzed not every calibration standard is used for each analyte. Each analyte has been assigned to an analyte group that includes a calibration range of at least five standards. The analyte group associations for each target analyte are provided in Tables in the appendix. The calibration range for each analyte group is as follows:

- Group A: This analyte group is associated with a seven-point calibration curve. The calibration range is 0.20 to 40 ppbv with the 0.035 ppbv standard excluded. The limit of quantitation (LOQ) for this group of analytes is 0.20 ppbv

- Group B: This analyte group is associated with a six-point calibration curve. The calibration range is 0.50 to 40 ppbv with the 0.04 and 0.20ppbv standards excluded. The limit of quantitation (LOQ) for this group of analytes is 0.50 ppbv.

- Group C: This analyte group is associated with a five-point calibration curve. The calibration range is 5.0 to 40 ppbv with the 0.04, 0.20, and 0.50ppbv standards excluded. The limit of quantitation (LOQ) for this group of analytes is 5.0 ppbv.

- Group D: This analyte group is an eight-point calibration curve. The calibration range is 0.035 to 40 ppbv. The limit of quantitation (LOQ) for this group of analytes is 0.035 ppbv.

- Group E: (Ethanol): This analyte has a six-point calibration curve. The calibration range is 5 to 100 ppbv. The limit of quantitation (LOQ) for this analyte is 5 ppbv.

Prepare the calibration standards using the formulations provided in Appendix B.

Analyze the standards in a sequence from lowest to highest concentration using the instructions provided in the Sample Analysis section.

10.2.2.1) Initial Calibration Acceptance Criteria

The data processing system calculates a relative response factor (RRF), for each analyte and isomer pair using the assigned internal standard. The internal standard associations for each target analyte are provided in tables in the Appendix. The data processing system also calculates a mean relative response factor, relative standard deviation (RSD), relative retention time (RRT) and the mean RRT.

The following criteria must be met for a calibration to be considered acceptable:

- The RSD for each target analyte must be <30% with at most 2 exceptions up to a limit of 40%. If linear regression is used the R2 must be greater than 0.996

- The area response for the primary quantitation ion for the internal standard for each ICAL standard must be within 40% of the mean area response over the calibration range for each internal standard.

    - The RRT for each target compound at each calibration level must be within 0.06 RRT units of the mean RRT for the compound. The retention time shift for each of the internal standards at each calibration level must be within 20 seconds of the mean retention time over the initial calibration range for each internal standard.

If these criteria are not met inspect the system for problems and perform corrective action. See the corrective action section and the QC Summary Table in the appendix for guidance.

Whichever calibration model is selected, it must be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount.

Calculate the concentration of each calibration standard from the final calibration curve. Calculate the %Error for each point. All points must have an absolute %Error of < 30%, except the low point % Error must be < 50%.

If these conditions are not met, perform corrective action and recalibrate the instrument.

The laboratory CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD / Linear Response Factor.
%Error is the absolute value of the difference between the calculated concentration and the true concentration divided by the true concentration x100%.

- For example, if the 0.20mg/L standard reads back as 0.17mg/L:
  $[(0.20 - 0.17)/0.20] \times 100\% = 15\%$

Repeat initial calibration whenever instrument operating conditions are changed, a new column is installed, when significant instrument maintenance has been performed, and when the result of the CCV indicate the calibration is no longer valid.

10.2.3) Second Source Calibration Verification (ICV)

Immediately following an acceptable initial calibration verify the accuracy of the calibration by the analysis of the second source calibration verification standard (ICV).

Prepare the ICV following the formulation provided in the Appendix.

Analyze the ICV following the instructions provided in Sample Analysis section.

The percent recovery (%R) for each target analyte must be within 70-130%. If criteria are not met, perform corrective action. Recommended corrective actions are provided in QC Summary Table. If corrective action is not successful, remake your standards and recalibrate.

If after successful analysis of the ICV, time remains in the 24-hour analytical window, QC and field samples may be analyzed without analysis of a continuing calibration verification check standard. If time does not remain in the analytical window, a new analytical sequence must be initiated with a Tune Standard followed by daily calibration (CCV).

10.2.4) Continuing Calibration Verification (CCV)

Analyze the CCV immediately after the tune standard unless the analytical window includes ICAL, in which case, a CCV is not required.

Prepare the CCV standard using the formulation given in the Appendix. The recommended concentration of the CCV for each target analyte is 10.0 ppbv.

Analyze the CCV following the instructions provided in Sample Analysis section. The data system calculates a response factor for each analyte and calculates the percent difference (%D) of the RRF relative to the mean RRF in the most recent initial calibration.

- The %D for each target analyte must be within ±30%. If the above criteria are not met, repeat the analysis of the CCV once. If the second CCV meets criteria, continue with the analytical sequence. If it fails, evaluate the data to determine if one of the following conditions is met. If these conditions are not met corrective action must be taken. Guidance for troubleshooting is provided in the Troubleshooting and Corrective Action sections. After corrective action the analytical sequence may be continued only if two immediate, consecutive CCVs at different concentrations are within acceptance criteria. If these two CCVs do not meet the criteria, recalibration is required prior to further analysis.

- If the CCV criteria are exceeded high, indicating a high bias, and the associated samples have non-detects for those analytes, the analytical data may be considered usable. In the absence of instructions otherwise, proceed with analysis.

- If the CCV criteria are exceeded low, indicating a low bias, analytical results may be reported if those results exceed the project's regulatory decision level. In other words, if the analytical results are sufficiently high to counter the low bias, results may be reported. Consult with the project manager to determine if the exception is allowable for each project.

10.2.5) Troubleshooting

Check the following items in case of calibration failures:

- Loss of sensitivity or unstable ISTD recoveries are usually the result of a leak. Check the union between the GC column and Entech transfer line.
- Loss of sensitivity for individual compounds may be a result of either an active site in a transfer line or a bad trap. Troubleshoot and perform maintenance as necessary.
- Poor chromatography usually requires GC column maintenance, perform as necessary.
- Carryover is usually caused by excessive amounts of analyte introduced to the system. Analyze blanks until the system is cleaned or replace the traps and transfer lines if necessary.

10.3) Sample Preparation

10.3.1) Post Sampling Canister Pressure Check Procedure

The post-sampling canister pressure check is performed at the time of sample login by sample management staff so that any problems found are quickly identified and communicated to the client.

Refer to the current version of *SOP49959 Sample Management* for the procedure to take and record the post sampling canister pressure check.

10.3.2) Sample Screening

At the laboratory's discretion unknown samples may be screened prior to initial analysis to determine if the sample requires dilution. Unless otherwise requested by the client the laboratory does not provide screen data with the data package report even when primary dilutions are performed based on the results of the screen analysis.

To prepare a sample for screen analysis, connect the sample canister to the autosampler connected to screening instrument and analyze 20 mL of sample. Acquire and evaluate the results. If the results of screen analysis indicate that a target compound is above its upper range of calibration, calculate a recommended dilution factor (DF) by dividing the concentration of analyte found by 30. Record the recommended DF on the screen worksheet.

NOTE: *Samples are screened on a GC/MS instrument that is programmed with the operating conditions referenced in this SOP. The calibration is checked weekly with a single point calibration standard at a concentration of 10 ppbv for all target analytes. The calibration is checked more frequently when the results of instrument analysis do not correlate well with the results of the screen analysis.*

10.3.3) Sample Dilutions & Pressure Adjustment

Field samples should be diluted prior to initial analysis when the screen results indicate that the concentrations are above calibration range or when the laboratory has sufficient knowledge of the sample (history) to know that the sample will require dilution. Field samples must be reanalyzed at a dilution when the concentration of target compounds in initial analysis exceed of the upper range of calibration.

When the return negative pressure of a canister is greater than -10” Hg, make-up gas may be added to provide sufficient volume of make-up gas in order to have an adequate sample volume for analysis. The addition of make-up gas is considered a canister dilution. Some concentrators are able to pull full sample volume even if the residual vacuum is lower than -10” Hg so make-up air may not be necessary.

For samples analyzed for constituent groups (e.g., TVOC as Toluene), ranges, and or unresolved complex mixtures (Total Hydrocarbons), samples must be diluted such that the maximum peak height of any sample constituent under consideration does not exceed the equivalent peak height generated by highest calibration level standard. For evaluation it may be useful to graphically overlay sample chromatograms with that of the high point calibration standard.
To dilute the sample:

1) Attach the sample canister to the nitrogen gas line equipped with a pressure gauge that reads negative pressure in (“Hg) and positive pressure in (psig).

2) Ensure the valve of the nitrogen gas line is closed then open the valve of the sample canister. Record the negative pressure reading in the Canister Dilution Worksheet or on the canister’s tag.

3) Slowly open the valve of the nitrogen gas line and fill the canister until canister pressure gauge reads -10” Hg. Do not open the valve to such an extent that the nitrogen gas line pressure drops below 15 psig and do not allow the nitrogen gas line to reach equilibrium otherwise you will contaminate the nitrogen gas line.

4) When the desired pressure is achieved, close the canister valve and the valve on the nitrogen gas line; wait 15 seconds. The pressure should not exceed 40 psig.

5) Open the canister valve and record the final pressure reading in psig.

6) Close the canister valve and remove the valve from the zero air-line.

7) Record the initial and final pressure readings in the TALS canister dilution tracking module. If the final pressure is below ambient, the pressure will be in “Hg. Be sure to change the units in the TALS dilution tracking module.

When the return pressure of a canister is positive, the pressure must be adjusted to near ambient (0” Hg) prior to analysis. To adjust the pressure to ambient, vent the canister to ambient in a fume hood by opening the canister value for ~4-5 seconds, close the valve. For higher pressure canisters, open the valve and listen for a release of air then close the valve when the sound recedes.

If a trip blank is provided under vacuum, pressurize the trip blank canister to 10 psig. The pressurization of the trip blank is not considered a dilution.

10.3.4) QC Sample Preparation

To prepare the method blank (MB): Fill a clean canister that has never been used to collect environmental samples and has never left the laboratory to 20 psig with nitrogen gas. Continue to use this canister as the MB until the pressure of the canister reaches 0 psig, at which time, recharge with nitrogen gas to 20 psig and reuse.

To prepare the LCS: Follow the instructions provided in the Appendix for preparation of the working ICV/LCS standard. If an LCSD is requested, analyze the LCSD from the same canister as the LCS, unless client makes a specific request for an independent LCSD can.

10.4) Sample Analysis

10.4.1) Instrument Operating Conditions

Optimize the GC and MS conditions for compound separation and sensitivity.

The recommended operating conditions are as follows:

Thermal Desorb:	Initial Trap #1 Temperature: -110ºC. Desorb Temperature from Trap #1 to #2: 0 ºC. Total Volume Transfer by Mass Flow Controller: 40 mL Initial Trap #2 Temperature: -15 ºC Desorb Temperature from Trap #2 to #3: 200ºC. Transfer time 3.5 minutes Initial Trap #3 Temperature: -165 ºC Injection Trap #3 Temperature: 70ºC Injection Time: 1.5 minutes Trap #3 Temperature after Injection: -165 ºC
Carrier Gas:	Helium, Ultra High Purity
Cryogenic Focusing Gas:	Liquid Nitrogen
Flow Rate:	~1.5 mL/min
Temperature Program:	Initial Temperature: 40ºC Initial Hold Time: 4 minutes Ramp1 Rate: 20ºC/min. to 200ºC. Ramp 2 Rate: 40ºC/min. to 220ºC Final Temperature: 220ºC Final Hold Time: 6.5 minutes
Electron Energy:	70 electron volts
Mass Range:	35-265 amu
Scan Time:	≥1 scan per second.

These operating conditions may be changed but once the operating conditions are established for initial calibration the same conditions must be used until a new calibration is performed.

The mass spec tune profile may be changed to optimize the BFB tune analysis but once set, it cannot be changed until another BFB tune is analyzed.

10.4.2) Daily Instrument Maintenance

Prior to analysis initiate the flushing sequence using the Entech software. Then initiate the bake program using the Entech software.

10.4.3) Bi-Weekly Instrument Maintenance

At a minimum frequency of once every two weeks, perform an autosampler leak check. Cap all autosampler ports and initiate the leak check program using the Entech software. Record this check in the instrument maintenance log.

10.4.4) Analytical Sequence

All Calibration Standards, batch QA and field samples, except for the BFB analysis, have 20mls of the Mixed Gas Stock Standard added in conjunction with any other additions. This addition adds the internal standards and surrogates.

An example analytical sequence that includes initial calibration (ICAL) is provided below. When ICAL is not performed, the sequence begins with the tune standard and is followed by the CCV, LCS, LCSD, and method blank. If sufficient time remains in the 24 hours analytical window after initial calibration, QC and field samples may be analyzed without the CCV and the ICV will serve as the LCS for the sequence. The MB, LCS and LSCD must be analyzed at a frequency of every 20 samples or with each analytical sequence whichever is more frequent.

1. Tune Standard (BFB)
2. ICAL
3. ICV
4. CCV
5. LCS (repeat every 20 samples)
6. LCSD (when requested)
7. MB (repeat every 20 samples)
8. Field Samples (including trip blanks)

Attach the canisters to the autosampler inlet in the order of the analytical sequence then initiate the analytical sequence. The autosampler introduces 200 mL of sample volume from each canister to the instrument system and adds 20 mL of the mixed gas

standard to each sample.

Acquire the data and evaluate the results to confirm qualitative identification and quantification.

10.5) Corrective Action

An out-of-control situation may occur that is not adequately addressed in the corrective action table or troubleshooting section. The analyst must use their best analytical judgment and available resources to determine the corrective action to be taken. The analyst should seek the assistance of their immediate supervisor, QA manager or other experienced staff if they are uncertain of the cause of the out-of-control situation.

The analysis must not be resumed until the source of the problem and an in-control status is re-established. All samples associated with the out-of-control situation must be reanalyzed after in-control status has been re-established.

If authorization is received from the Supervisor or QA Manager for release with data qualification, a Non-Conformance Memo (NCM) must be created to notify the project manager, who then notifies the client of the out-of-control situation. Also, the situation may be significant enough that the Supervisor or QA Manager would create a Corrective Action Report (CAR). Refer to the *SOP49967 Nonconformance and Corrective Action Procedures* for further guidance.

11) Calculations/Data Reduction

11.1) Qualitative Identification

The data processing system tentatively identifies target analytes by comparing the retention time of the peaks to the window set around the continuing calibration standard, and searches in that area for the primary ion and up to two secondary ions characteristic of the target analyte.

All tentative identifications made by the computer are reviewed and either accepted or rejected by the primary analyst. The identification made by the system is accepted when the following criteria are met:

- The target analyte is identified by comparison of its background subtracted mass spectrum to a reference spectrum in the NIST14 database. In general, all ions that are present above 10% relative abundance in the mass spectrum of the standard should be present in the mass spectrum of the sample component and their relative abundances should agree within 20%. For example, if an ion has a relative abundance of 30% in the standard spectrum, its abundance in the sample spectrum should be in the range of 10-50%. Some ions, particularly the molecular ion, are of special importance if a tentative identification is to be made and should be evaluated even if they are below 10% relative abundance.
- The GC retention time for the target analyte should be within 0.06 RRT units of the daily standard.

Identification requires expert judgment when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When GC peaks obviously represent more than one sample component (i.e., broadened peak with shoulder(s) or valley between two or more maxima), appropriate analyte spectra and background spectra can be selected by examining plots of characteristic ions for tentatively identified components.

When analytes coelute (i.e., only one GC peak is apparent), the identification criteria can be met but each analyte spectrum will contain extraneous ions contributed by the coeluting compound. If the data system does not properly integrate a peak, perform manual integration. All manual integration must be performed and documented in accordance with corporate *SOP43862 Manual Integrations*.

11.2) Quantification of Target Analytes

After a compound has been identified, the data system quantifies the on-column concentration of the target compound based on the integrated abundance of the characteristic ion from the EICP. If there is matrix interference with the primary ion, a secondary ion may be used for quantification by calculating a mean RF factor for that ion and using that ion to quantify the analyte in the sample. When secondary ion calculations are required, include this information in the non-conformance report and project narrative.

Samples analyzed for constituent groups, ranges, and or unresolved complex mixtures are quantified as follows;

11.2.1) TVOC as Toluene

An internal standard type of quantification response factor is generated using the RIC peak response measured for Toluene and its associated internal standard (as detailed in the calculation section). That response factor is used to quantify all RIC chromatographic sample response with the exception of those peak responses associated with internal standards. The results are reported as 'TVOC (as Toluene)'.

Retention Time Range: Propene to Naphthalene

11.2.2) GRO as Octane

An internal standard type of quantification response factor is generated using the RIC peak response measured for Octane and its associated internal standard (as detailed in the calculation section). That response factor is used to quantify all RIC chromatographic sample response with the exception of those peak responses associated with internal standards that eluted within the retention time range as defined by the elution of 2-Methyl-Butane through Decane (C5-C10). The results are reported as 'GRO (as Octane).

Retention Time Range: 2-Methyl-Butane to n-Decane

11.2.3) Unresolved Complex mixtures

An internal standard type of quantification response factor is generated using the RIC peak response measured for the un-resolved complex mixture (i.e. the gasoline envelope) and its associated internal standard (as detailed in the calculation section). That response factor is used to quantify all similarly chromatographing RIC sample response with the exception of those peak responses associated with internal standards. The results are reported using the name of the unresolved complex mixture used in the calibration (e.g. Total Hydrocarbons).

Retention Time Range: Propene to Naphthalene

Final results are calculated in TALS

11.3) Calculations

Analytical results are calculated as follows:

Dilution Factor

DF = (V2/V1) x (V4/V3)

Where:
V1 = Pre-Dilution Canister Volume
V2 = Post-Dilution Canister Volume
V4 = Base Sample Amount (200 mL)

Relative Response Factor (RRF)

RRF = (Ax)(Cis)/(Ais)(Cx)

Where:
Ax = Area of the quantitation ion of the analyte
Ais = Area of the quantitation ion of the internal standard
Cx = Concentration of analyte in concentration units (ppbv)
Cis = Concentration of internal standard in concentration units (ppbv)

Sample Concentration

Cx = [(Ax)(Cis)/(Ais)(Mean RRF)] x (DF)

Where:
Cx = Compound concentration (ppbv)
Cis = Concentration of associated internal standard (ppbv)
Ais = Area of quantitation ion for associated internal standard
Ax = Area of quantitation ion for compound
DF = Dilution Factor
Mean RRF = Average Relative Response Factor from initial calibration.

Unit Conversion from ppbv to ugm3

Analytical Result(ug/m3) = Result(ppbv) x (MW/24.45)

Where:
MW = molecular Weight

Example:
Benzene Result = 56 ppbv
Benzene mw = 78.108

Analytical Result(ug/m3) = 56ppbv x (78.108/24.45)

Result(ug/m3) = 178.9 ug/m3 reported as 180 ug/m3

Percent Recovery (%R)

%R = (Cs/Cn) x 100

Where:
Cs = Concentration of the spiked sample (ppbv)
Cn = Nominal concentration of spike added (ppbv)

Precision (%RPD)

%RPD = [(|C1-C2|)/((C1+C2)/2) x 100

Where:
C1 = Measured concentration of the first sample aliquot
C2 = Measured concentration of the second sample aliquot

Formula of Standard Addition:

Volume (mL) = (FR x T) + 25 mL

Where:
FR = flow rate in mL/min
T= time in minutes

Example:
FR = 70ml/min
Time = 10.69 minutes
Volume = 25+ (70 x 10.69) = 773 mL

Total Volume of diluted standard

Volume (mL) = CV x P

Where:
CV = canister volume in mL
P = pressure in Bar (14.7psi per Bar)

Example:
CV = 6185 mL
P= 2.5 Bar
Volume = 15462 mL

11.4) Data Review

11.4.1) Primary Review (Performed by Primary Analyst)

Upload the data files to TALS. Enter batch editor information and add the standards and reagents to the TALS batch. Review the results against acceptance criteria. If acceptance criteria are not met, make arrangements to perform corrective action.

Check the results of samples analyzed immediately after high concentration samples for signs of carry-over. Reanalyze the sample if carry over is suspected.

Dilute and reanalyze samples whose results exceed the calibration range. The diluted analysis should result in a determination within the upper half of the calibration curve.

If samples are run at dilution, there should be a target peak that is at least 20% of the calibrated range but preferably in the upper half of the range or there should be an abundance of TICs which preclude a more concentrated analysis, or the sample should show matrix effects which preclude a more concentrated analysis. If a sample is diluted due to TICs or matrix, an NCM should be written explaining the reason for dilution.

Set results to primary, secondary, acceptable or rejected as appropriate.

Verify corrective action was taken for all results not within acceptance criteria. If corrective action is not taken or was unsuccessful, record all instances where criteria are not met with a nonconformance memo (NCM). Be sure to provide

explanation of your decision making in the internal comment section of the NCM. The internal comment section should list the reason the NCM is suspected, which action (if any) was taken and why, and the outcome of the action taken. Refer to the *SOP49967 Nonconformance and Corrective Action Procedures* for further guidance.

Review project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements to ensure those project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Review that QC links have been properly made and run the data review checker (DRC) and address any anomalies.

Set the batch to 1st level review and record your review on the data review checklist.

11.4.2) Secondary Review (Performed by Peer Reviewer)

Review the project documents such as the Project Plan (PP), Project Memo or any other document/process used to communicate project requirements and verify project requirements were met. If project requirements were not met, immediately notify the project manager (PM) to determine an appropriate course of action.

Review records (these records include but are not limited to the Pre-Shipment Clean Canister Certification Report, Flow Controller Set Flow Rate and Leak Check Record, Field Test Data Sheets, and the Air Canister Return Pressure Check Record) associated with release and return of air sampling equipment to ensure all anomalies are properly recorded. Compare any problems noted in these documents with the analytical results and record any findings in the narrative note program or otherwise communicate your findings to the PM for inclusion in the project narrative.

Review the TALS batch editor to verify information is complete. Review the batch to verify that the procedures in this SOP were followed. If discrepancy is found, resolve the discrepancy and verify any modifications to the SOP were approved and properly documented.

Spot-check 15% of samples in the batch to verify quantitative and qualitative identification. If the samples are being analyzed under DoD methods 100% of data must be checked during secondary review.

If manual integrations were performed:

- Review all integrations to verify that the integration meets the requirements for manual integration as specified in corporate SOP CA-Q-S-002. If an error is suspected or found, consult with the analyst that performed the integration and request correction or notify the Department Manager or QA Manager. Do not "fix" the integration. Reintegration by a secondary data reviewer must not be performed except in limited circumstances as approved by the department supervisor or other laboratory management. If those instances where the secondary reviewer performs the integration, this person is now considered the primary analyst and each integration performed by the secondary reviewer must be subsequently reviewed by a peer analyst or the department supervisor to verify the integration is consistent and compliant with the requirements specified in corporate SOP CA-Q-S-002.
- Check to ensure an appropriate technical reason code is provided for all manual integrations.

Verify that the performance criteria for the QC items listed in TO15 QC Summary & Recommended Corrective Action table in Appendix were met. If the results do not fall within the established limits, verify that corrective actions were performed. If corrective action was not performed; verify the reason is provided and that the situation is properly documented with an NCM. Set samples to 2nd level review. Record second level review on the data review checklist.

Run and review the deliverable. Fix any problems found. When complete set the method chain to lab complete and forward any paperwork to report/project management.

11.5) TO15 Poor Performing Analytes

NOTE: *Statistically derived limits will be no wider than 50-150%R for poor performing compounds.*

Compounds with a high boiling point which is beyond the range of typical gas compounds.

- Dodecane
- 1,2,4-Trichlorobenzene
- Hexachlorobutadiene
- Naphthalene
- 1,2,3-Trichlorobenzene

The following compounds change how they act on the traps depending on the humidity of the standard.

- 3-chloro-1-propene
- Bromoform

The following compounds are very polar and are linked to the water in the standard. Since we remove most of the water during the trapping process, a little change can produce a big result. For example removing 95% of the water instead of 94% of the water will have a negligible affect on non-polar compounds, but a 20% difference for polar compounds.

- Ethanol (made from neat material and is not part of the standard TO-15 calibration gas mix due to high reactivity
- Ethyl ether
- Acrolein
- Acetone
- Isopropyl alcohol
- 2-butanone
- Tetrahydrofuran
- 2-hexanone

11.6) Data Reporting

Report analytical results above the reporting limit (RL) as the value found. Report analytical results less than the RL, to the adjusted RL with a "U" data qualifier. If the data is to be reported to the method detection limit (MDL) the analytical results between the RL and the MDL are reported with an "estimated" footnote. Adjust the RL and MDL for sample dilution/concentration. The unadjusted RL for each target analyte is provided in Routine Compound List and TO-3 Analyte List tables. For Method TO3 the laboratory does not report values below the reporting limit.

Data reporting and creation of the data deliverable is performed by TALS using the formatters set by the project manager during project initiation.

Electronic and hardcopy data are maintained as described in laboratory *SOP49966 Management, Retention Storage and Archive of Data Records*.

12) Method Performance

12.1) Method Detection Limit Study (MDL)

Perform a method detection limit (MDL) study at initial method set-up following the procedures specified in laboratory SOP BR-QA-005,

12.2) Training Requirements and Demonstration of Capabilities

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. The Group Leader is also responsible for the documentation of this training.

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the portion of the method they will be performing prior to working independently. Annually thereafter, the analyst must document continuing demonstration of capability (CDOC). See requirements for demonstration of analyst proficiency in *SOP52264 Employee Training and Analyst Demonstration of Proficiency*.

13) Pollution Control

It is Eurofins Burlington's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in NDSC-USEHS-*QP46060 NDSC HSE Manual*.

14) Waste Management

Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to *SOP47863 Tracking and Collecting of Hazardous Waste* and *SOP48269 Procedure for Acceptance and Handling of USDA Regulated Domestic and Foreign Soil*.

The following waste streams are produced when this method is carried out:

- None

15) Method Modifications

- This SOP utilizes and alternative detector (mass spectrometer) vs that of a FID and/or ECD as listed in the published method. (TO3)

- This SOP describes a method where an aliquot of a whole air sample collected in a passivated stainless-steel canister is cryogenically trapped and concentrated prior to injection into the MS detector equipped GC. (TO3)
- This Method SOP utilizes an RTX-624 capillary column (60M x 0.32 mm ID x 0.18um). (TO3, TO15)
- Additional quality control aspects are employed in the use of this method based on the alternative detector type, specifically, the detector tune is verified at the beginning of each period of analysis prior to the acquisition on any standard, blank, QC sample or field sample. (TO3)
- This Method SOP does not utilize sub ambient column oven temperature programming. (TO3, TO15)
- Sample traps consist of a multi bed system employing Tenax and glass beads (see manufactures system description: Entech Model 7100 Sample Pre-Concentrator). (TO15)
- Sampling apparatus consists of passivated stainless-steel canister specifically design for the collection of ambient air samples for volatile analysis (TO3)
- Nafion dryers are not used. The analytical pre-concentration system employs a moisture control system consisting of multi sorbent bed traps. (TO15)
- System calibration is verified every 24 hours within which samples are analyzed. System linearity is not verified every 4-6 hours as described. (TO3)
- Published Method requires weekly multi-point calibration be performed. Laboratory performs multi-point calibrations as necessary see section 10.2 (TO3)
- System linearity is determined through multi point calibration utilizing average response factors and internal standard technique quantification (see Section 10).(TO3)
- This SOP is applied to more than just the ambient air listed as a matrix by the method (TO15)

16) Appendices

16.1) Routine Compound List, Reporting Limit, Internal Standard and Ion Assignments

Analyte	CAS #	6L RL (ppbv)	1L RL (ppbv)	Quantifier Mass	Qualifier Mass	Qualifier Mass	ISTD Group	Analyte Group
Dichlorodifluoromethane	75-71-8	0.5	5	85	87		1	B
Chlorodifluoromethane	75-45-6	0.5	5	51	67	69	1	B
1,2-Dichlorotetrafluoroethane	76-14-2	0.2	2	85	135	87	1	A
Chloromethane	74-87-3	0.5	5	50	52		1	B
n-Butane	106-97-8	0.5	5	43	41	58	1	B
Vinyl Chloride	75-01-4	0.035	0.35	62	64		1	D
1,3-Butadiene	106-99-0	0.035	0.35	54			1	D
Bromomethane	74-83-9	0.2	2	94	96		1	A
Chloroethane	75-00-3	0.5	5	64	66		1	B
Isopentane	78-78-4	0.2	2	43	57	56	1	A
Bromoethene (Vinyl Bromide)	593-60-2	0.2	2	106	108	81	1	A
Trichlorofluoromethane	75-69-4	0.2	2	101	103		1	A
Pentane	109-66-0	0.5	5	43	57	56	1	B
EthylEther	60-29-7	0.2	2	59	45	74	1	A
Acrolein	107-02-8	5	50	56	55	37	1	C
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.2	2	101	151	103	1	A
1,1-Dichloroethene	75-35-4	0.035	0.35	96	161	63	1	A
Acetone	67-64-1	5	50	43	58		1	C
Isopropyl Alcohol	67-63-0	5	50	45	43		1	C
Carbon Disulfide	75-15-0	0.5	5	76			1	B
3-Chloropropene (Allyl Chloride)	107-05-1	0.5	5	41	76		1	B
Acetonitrile	75-05-8	5	50	41	40	39	1	C
Methylene Chloride	75-09-2	0.5	5	49	84	86	1	B
tert-Butyl Alcohol	75-65-0	5	50	59	41	43	1	C
Methyl tert-Butyl Ether	1634-04-4	0.5	5	73	43		1	B
trans-1,2-Dichloroethene	156-60-5	0.2	2	61	96		1	A
n-Hexane	110-54-3	0.2	2	57	86		1	B
1,1-Dichloroethane	75-34-3	0.035	0.35	63	65	83	1	A
Methyl Ethyl Ketone	78-93-3	0.5	5	72	43		1	B
cis-1,2-Dichloroethene	156-59-2	0.035	0.35	96	98	83	1	A
Tetrahydrofuran	109-99-9	5	50	42	72		2	C
Chloroform	67-66-3	0.2	2	83	85		1	A

1,1,1-Trichloroethane	71-55-6	0.2	2	97	99	61	2	A
Cyclohexane	110-82-7	0.2	2	84	56		2	A
Carbon Tetrachloride	56-23-5	0.035	0.35	117	119		2	D
2,2,4-Trimethylpentane	540-84-1	0.2	2	57	41	43	2	A
1,2-Dichloroethene (total)	540-59-0	0.2	2	61	96		1	A
Benzene	71-43-2	0.2	2	78	77		2	A
1,2-Dichloroethane	107-06-2	0.2	2	62	98		2	A
n-Heptane	142-82-5	0.2	2	43	71		2	A
Trichloroethene	79-01-6	0.035	0.35	95	130	132	2	D
Methyl Methacrylate	80-62-6	0.5	56	69	41	39	2	B
1,2-Dichloropropane	78-87-5	0.2	2	63	41		2	A
1,4-Dioxane	123-91-1	5	50	88	58		2	C
Dibromomethane	74-95-3	0.2	2	174	93	172	2	A
Bromodichloromethane	75-27-4	0.2	2	83	85		2	A
cis-1,3-Dichloropropene	10061-01-5	0.2	2	75	110		2	A
Methyl Isobutyl Ketone	108-10-1	0.5	5	43	58		2	B
n-Octane	111-65-9	0.2	2	43	57	114	2	A
Toluene	108-88-3	0.2	2	92	91		3	A
trans-1,3-Dichloropropene	10061-02-6	0.2	2	75	110		2	A
1,1,2-Trichloroethane	79-00-5	0.2	2	83	97	85	3	A
Tetrachloroethene	127-18-4	0.035	0.35	166	168	129	3	D
Methyl Butyl Ketone	591-78-6	0.5	5	43	58		3	B
Dibromochloromethane	124-48-1	0.2	2	129	127		3	A
1,2-Dibromoethane	106-93-4	0.2	2	107	109		3	A
Nonane	111-84-2	0.2	2	57	71	128	3	A
Chlorobenzene	108-90-7	0.2	2	112	77	114	3	A
Ethylbenzene	100-41-4	0.2	2	91	106		3	A
Xylene (m,p)	1330-20-7	0.5	5	106	91		3	A
Xylene (o)	95-47-6	0.2	2	106	91		3	A
Styrene	100-42-5	0.2	2	104	78		3	A
Bromoform	75-25-2	0.2	2	173	175	171	3	A
Cumene	98-82-8	0.2	2	105	120	77	3	A
1,1,2,2-Tetrachloroethane	79-34-5	0.2	2	83	131	85	3	A
Xylene (total)	1330-20-7	0.2	2	106	91		3	A
n-Decane	124-18-5	0.5	5	57	71	142	3	B
n-Propylbenzene	103-65-1	0.2	2	91	120	92	3	A
1,2,3-Trichoropropane	96-18-4	0.5	5	75	110	112	3	B
4-Ethyltoluene	622-96-8	0.2	2	105	120		3	A
1,3,5-Trimethylbenzene	108-67-8	0.2	2	105	120		3	A
2-Chlorotoluene	95-49-8	0.2	2	91	63		3	A
tert-Butylbenzene	98-06-6	0.2	2	119	91	134	3	A
1,2,4-Trimethylbenzene	95-63-6	0.2	2	105	120		3	A
sec-Butylbenzene	135-98-8	0.2	2	105	134	91	3	A
4-Isopropyltoluene	99-87-6	0.2	2	119	134	91	3	A
1,3-Dichlorobenzene	541-73-1	0.2	2	146	111	148	3	A
1,4-Dichlorobenzene	106-46-7	0.2	2	146	111	148	3	A
n-Undecane	1120-21-4	5	50	57	71	156	3	C
Benzyl Chloride	100-44-7	0.2	2	91	126	65	3	A
n-Butylbenzene	104-51-8	0.2	2	91	134	92	3	A
1,2-Dichlorobenzene	95-50-1	0.2	2	146	111	148	3	A
n-Dodecane	112-40-3	5	50	57	71	170	3	C
1,2,4-Trichlorobenzene	120-82-1	0.5	5	180	182		3	B
1,3-Hexachlorobutadiene	87-68-3	0.2	2	225	223		3	A
Naphthalene	91-20-3	0.5	5	128			3	B
1,2,3-Trichlorobenzene	87-61-6	0.2	2	180	182	145	3	A
Propylene	115-07-1	5	50	41	42	39	1	C

Vinyl Acetate	108-05-4	5	50	43	86		1	C
Ethyl Acetate	141-78-6	5	50	43	74		1	C
Ethanol	64-17-5	5	50	46	45		1	E
Bromochloromethane	74-97-5	NA	NA	128	49	130	1	NA
1,4-Difluorobenzene	540-36-3	NA	NA	114			2	NA
Chlorobenzene-d5	3114-55-4	NA	NA	117			3	NA

16.2) TO-3 Analyte List, Reporting Limit, Internal Standard and Ion Assignments

Analyte	CAS No.	RL (ppbv)	Qualifier Ion	Qualifier Ion	Qualifier Ion	ISTD Group
n-Octane	111-65-9	0.2	43	57	114	2
Toluene	108-88-3	0.2	92	91		3
TVOC as Toluene	NA	11				NA
GRO as Octane	NA	14				NA
Total Hydrocarbons	NA	76 ug/m3				NA
Internal Standards						
Bromochloromethane	74-97-5	NA	128	49	130	1
1,4-Difluorobenzene	540-36-3	NA	114			2
Chlorobenzene-d5	3114-55-4	NA	117			3

16.3) Tune Standard Criteria

Mass	Ion Abundance Criteria
50	8.0 to 40.0 percent of mass 95
75	30.0 to 66.0 percent of mass 95
95	Base Peak, 100 percent relative abundance
96	5.0 to 9.0 percent of mass 95
173	Less than 2.0 percent of mass 174
174	50.0 to 120.0 percent of mass 95
175	4.0 to 9.0 percent of mass 174
176	93.0 to 101.0 percent of mass 174
177	5.0 to 9.0 percent of mass 176

16.4) QC Summary & Recommended Corrective Action (Routine and DoD)

QC Check	Frequency	Acceptance Criteria	Recommended Corrective Action
Tune Standard	Prior to calibration and every 24 hours	See Tune Standard Criteria table	Correct Problem. Reanalyze. No samples may be analyzed without a valid tune.
Initial Calibration	Prior to sample analysis and when CCV fails	RSD for each analyte ≤ 30% with 2 exceptions up to 40% (if allowed). Individual Calibration points: < 30% Error, except < 50% for the lowest calibration point	Correct problem and repeat calibration

RRT	With each calibration	RRT of each target analyte in each calibration standard within ± 0.006 RRT units	Correct Problem. Repeat ICAL
ICV	Once after each ICAL	%R 70-130 for all analytes	Correct Problem. Reanalyze, re-make, re-verify & re-analyze. If that fails, re-make all standards and repeat calibration
CCV	Daily before sample analysis after tune standard	%Difference ≤ 30	Correct Problem. Reanalyze once. If that fails, see Section 10.3.9 for instruction.
LCS	Each batch or every 20 samples, whichever is sooner.	%Recovery 70-130%	Reanalyze LCS, re-prep and reanalyze LCS, and all associated samples if sufficient sample volume is available. If corrective action not successful, initiate nonconformance report and qualify sample results.
LCSD	Each batch or every 20 samples, only if there is no Sample Duplicate, or per client request	Relative Percent Difference (RPD) ≤ 25%	Reanalyze LCSD, re-prep and reanalyze LCS, LCSD and all associated samples if sufficient sample volume is available. If corrective action not successful, initiate nonconformance report and qualify sample results.
Method Blank	Each batch or every 20 samples, whichever is sooner.	< Reporting Limit	Reanalyze along with associated samples, unless detects for same compounds found in blank are greater than 10X the concentration found in the blank.
Internal Standards	All standards, field and QC samples	+/- 40% area response from last acceptable calibration. RT +/- 0.33 min (20 seconds) from last acceptable calibration.	Inspect system for malfunction. Reanalyze samples. Qualify data.
Sample Duplicate	Each batch or every 20 samples, if volume allows	Relative Percent Difference (RPD) ≤ 25%	Consult with PM. Reanalyze or qualify data.

16.5) In-house LCS limits

Analyte	CAS #	In-House Lower Limit (%R)	In-House Upper Limit (%R)	Relative Percent Difference (%)
Dichlorodifluoromethane	75-71-8	61	142	25
Chlorodifluoromethane	75-45-6	60	147	25
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	71	141	25
Chloromethane	74-87-3	56	141	25
n-Butane	106-97-8	53	151	25
Vinyl Chloride	75-01-4	61	135	25
1,3-Butadiene	106-99-0	58	139	25
Bromomethane	74-83-9	72	124	25
Chloroethane	75-00-3	68	130	25
Isopentane	78-78-4	56	150	25
Bromoethene (Vinyl Bromide)	593-60-2	75	125	25
Trichlorofluoromethane	75-69-4	70	129	25
Pentane	109-66-0	59	152	25
EthylEther	60-29-7	71	143	25
Acrolein	107-02-8	50*	150*	25
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	70	121	25
1,1-Dichloroethene	75-35-4	68	120	25
Acetone	67-64-1	54	154	25
Isopropyl Alcohol	67-63-0	53	142	25
Carbon Disulfide	75-15-0	71	138	25
3-Chloropropene (Allyl Chloride)	107-05-1	50*	150*	25

Acetonitrile	75-05-8	60	154	25
Methylene Chloride	75-09-2	59	137	25
tert-Butyl Alcohol	75-65-0	66	132	25
Methyl tert-Butyl Ether	1634-04-4	70	127	25
trans-1,2-Dichloroethene	156-60-5	69	137	25
n-Hexane	110-54-3	63	138	25
1,1-Dichloroethane	75-34-3	66	130	25
Methyl Ethyl Ketone	78-93-3	72	124	25
cis-1,2-Dichloroethene	156-59-2	72	121	25
Tetrahydrofuran	109-99-9	60	149	25
Chloroform	67-66-3	73	124	25
1,1,1-Trichloroethane	71-55-6	72	127	25
Cyclohexane	110-82-7	76	124	25
Carbon Tetrachloride	56-23-5	71	133	25
2,2,4-Trimethylpentane	540-84-1	68	131	25
1,2-Dichloroethene (total)	540-59-0	73	126	25
Benzene	71-43-2	73	119	25
1,2-Dichloroethane	107-06-2	68	135	25
n-Heptane	142-82-5	60	142	25
Trichloroethene	79-01-6	73	122	25
Methyl Methacrylate	80-62-6	73	129	25
1,2-Dichloropropane	78-87-5	69	128	25
1,4-Dioxane	123-91-1	66	129	25
Dibromomethane	74-95-3	68	126	25
Bromodichloromethane	75-27-4	75	127	25
cis-1,3-Dichloropropene	10061-01-5	74	125	25
Methyl Isobutyl Ketone	108-10-1	58	144	25
n-Octane	111-65-9	62	144	25
Toluene	108-88-3	75	122	25
trans-1,3-Dichloropropene	10061-02-6	74	128	25
1,1,2-Trichloroethane	79-00-5	75	126	25
Tetrachloroethene	127-18-4	70	125	25
Methyl Butyl Ketone	591-78-6	57	143	25
Dibromochloromethane	124-48-1	73	125	25
1,2-Dibromoethane	106-93-4	78	122	25
Nonane	111-84-2	70	131	25
Chlorobenzene	108-90-7	76	119	25
Ethylbenzene	100-41-4	74	122	25
Xylene (m,p)	1330-20-7	76	121	25
Xylene (o)	95-47-6	73	123	25
Styrene	100-42-5	74	125	25
Bromoform	75-25-2	53	149	25
Cumene	98-82-8	73	123	25
1,1,2,2-Tetrachloroethane	79-34-5	74	126	25
Xylene (total)	1330-20-7	75	122	25
n-Decane	124-18-5	68	134	25
n-Propylbenzene	103-65-1	73	127	25
1,2,3-Trichoropropane	96-18-4	71	125	25
4-Ethyltoluene	622-96-8	75	129	25
1,3,5-Trimethylbenzene	108-67-8	72	126	25
2-Chlorotoluene	95-49-8	74	126	25
tert-Butylbenzene	98-06-6	71	125	25
1,2,4-Trimethylbenzene	95-63-6	71	129	25
sec-Butylbenzene	135-98-8	70	128	25
4-Isopropyltoluene	99-87-6	68	130	25
1,3-Dichlorobenzene	541-73-1	69	131	25
1,4-Dichlorobenzene	106-46-7	67	132	25
n-Undecane	1120-21-4	62	136	25
Benzyl Chloride	100-44-7	60	136	25
n-Butylbenzene	104-51-8	65	137	25

1,2-Dichlorobenzene	95-50-1	68	129	25
n-Dodecane	112-40-3	50*	150*	25
1,2,4-Trichlorobenzene	120-82-1	50*	150*	25
1,3-Hexachlorobutadiene	87-68-3	58	130	25
Naphthalene	91-20-3	50*	150*	25
1,2,3-Trichlorobenzene	87-61-6	50*	150*	25
Propylene	115-07-1	50*	150*	25
Vinyl Acetate	108-05-4	59	149	25
Ethyl Acetate	141-78-6	70	131	25
Ethanol	64-17-5	50*	150*	25

*These are poor performing compounds. Limits for poor performing compounds will be no wider than 50-150%.
**The limits in this table are those in effect as of the published date of this SOP. These limits are based on historical data and are subject to change. Current in-house limits are populated in the LIMS database. Contact a laboratory representative for the most current set of limits. Limit Ref: 2019CCTO15

16.6) Appendix A: Terms and Definitions

Acceptance Criteria: Specified limits placed on characteristics of an item, process or service defined in requirement documents.
Accuracy: The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.
Analyte: The specific chemicals or components for which a sample is analyzed. (EPA Risk Assessment Guide for Superfund, OSHA Glossary).
Batch: Environmental samples that are prepared and/or analyzed together with the same process, using the same lot(s) of reagents. A preparation/digestion batch is composed of one to 20 environmental samples of similar matrix, meeting the above criteria. An analytical batch is composed of prepared environmental samples (extracts, digestates and concentrates), which are analyzed together as a group.
Calibration: a set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material and the corresponding values realized by the standards.
Calibration Curve: the graphical relationship between the known values or a series of calibration standards and their instrument response.
Calibration Standard: A substance or reference used to calibrate an instrument.
Continuing Calibration Verification (CCV): An analytical standard gas mixture containing all target analytes and internal standard compounds that is used to evaluate the performance of the instrument system with respect to a defined set of method criteria.
Corrective Action: the action taken to eliminate the cause of an existing nonconformity, defect or other undesirable occurrence in order to prevent recurrence.
Cryogen: A refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen (bp -195.8°C) or liquid argon (bp -185.7°C).
Demonstration of Capability (DOC): procedure to establish the ability to generate acceptable accuracy and precision.
Holding Time: the maximum time that a sample may be held before preparation and/or analysis as promulgated by regulation or as specified in a test method.
Initial Calibration: Analysis of analytical standards for a series of different specified concentrations used to define the quantitative response, linearity and dynamic range of the instrument to target analytes.
Initial Calibration Verification (ICV): An analytical standard mixture containing all target analytes and internal standard compounds that are prepared from a source independent of the source of the initial calibration standards. The purpose of the ICV is to verify that the initial calibration is in control.
Intermediate Standard: a solution made from one or more stock standards at a concentration between the stock and working standard. Intermediate standards may be certified stock standard solutions purchased from a vendor and are also known as secondary standards.
Internal Standards (IS): Non-target analytes that are similar to the target analytes but are not expected to be found in environmental media (generally, isotopically labeled target analytes are used for this purpose). IS are added to every standard, quality control sample, and field sample at a known concentration prior to analysis. IS responses are used as the basis for quantitation of target analytes.
Laboratory Control Sample (LCS): A QC sample of known composition spiked with analytes of interest. The LCS evaluates method performance and ability to successfully recover target analytes from a clean matrix. LCS recovery is typically expressed as percent recovery and provides a measure of accuracy. A LCSD is a duplicate LCS prepared and analyzed from a separate canister to provide a measure of replicate precision.
Method Blank (MB): A canister of humidified ultra pure zero air that is treated exactly as a sample. The MBLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.
Method Detection Limit (MDL): the minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific measurement system. The MDL is a statistical estimation at a specified confidence interval of the concentration at which relative uncertainty is ±100%. The MDL represents a range where qualitative detection occurs. Quantitative results are not produced in this range.
Non-conformance: an indication, judgment, or state of not having met the requirements of the relevant specification, contract or regulation.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves.
Quality Control Sample (QC): a sample used to assess the performance of all or a portion of the measurement system.
Reporting Limit (RL): the level to which data is reported for a specific test method and/or sample.
Stock Gas Mixture: A Commercially purchased concentrated gas mixture containing one or more method analytes

16.7) Appendix B: Standard Preparation Tables

The standard formulations contained in this appendix are recommended and are subject to change. If the concentration or volume of any of the stock standard changes, the standard preparation instructions must be adjusted accordingly. See laboratory *SOP52272 Traceability, Handling, and Storage of Standards and Reagents* for further guidance on the preparation of standard solutions.

Prepare all standards using the McMillan Company 80SD mass flow controller. Prepare the standard in zero air, demonstrated to be analyte free. Store the standard at ambient temperature. Unless otherwise specified, assign an expiration date of 30 days from date of preparation unless the parent standard expires earlier, in which case, use the earliest expiration date.

Intermediate Calibration Standard

Parent Standard	Vendor	Stock Standard Concentration (ppmv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Custom Calibration Stock Standard	Spectra Gases Custom Made	1.0	7500	37.5	200

Prepare in 15 L Summa Canister. Expiration Period 3months
This standard contains all the target analytes listed in the Routine Compound Table.

Working Calibration Standards

Parent Standard	Calibration Standard	Parent Standard Concentration (ppbv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Cal Standard 20 ppbv	Cal Standard 0.2 ppbv	20	155	15.46	0.2
Cal Standard 20 ppbv	Cal Standard 0.5 ppbv	20	386	15.46	0.5
Intermediate Calibration Standard	Cal Standard 5 ppbv	200	386	15.46	5
Intermediate Calibration Standard	Cal Standard 10 ppbv	200	773	15.46	10
Intermediate Calibration Standard	Cal Standard 15 ppbv	200	1160	15.46	15
Intermediate Calibration Standard	Cal Standard 20 ppbv	200	1546	15.46	20
Intermediate Calibration Standard	Cal Standard 40 ppbv	200	3092	15.46	40

Prepare in 6 L Summa Canister Expiration Period 3 months
Each calibration standard contains all the analytes listed in the Routine Compound Table at the above concentrations.

Intermediate ICV/LCS Standard

Parent Standard	Vendor	Stock Standard Concentration (ppmv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
ICV Stock Standard	Spectra Gases Custom Made	1.0	7500	37.5	200

Prepare in 15L Summa Canister Expiration period 3 months
This standard contains all target analytes listed in Routine Compound Table.

Working ICV/LCS Standard

Parent Standard	Calibration Standard	Stock Standard Concentration (ppbv)	Volume Added (mL)	Final Volume (L)	Final Concentration (ppbv)
Intermediate ICV/LCS Standard	ICV Standard 10 ppbv	200	773	15.46	10

Prepare in 6L Summa Canister Expiration period 3 months
This standard contains all target analytes listed in Routine Compound Table.

Calibration Level	Working Calibration Standard	Volume Analyzed (mL)	Concentration on Column (ppbv)
Calibration Level 1	Cal Standard 0.2 ppbv	40	0.04
Calibration Level 2	Cal Standard 0.2 ppbv	200	0.2

Calibration Level 3	Cal Standard 0.5 ppbv	200	0.5
Calibration Level 4	Cal Standard 5 ppbv	200	5
Calibration Level 5	Cal Standard 10 ppbv	200	10
Calibration Level 6	Cal Standard 15 ppbv	200	15
Calibration Level 7	Cal Standard 20 ppbv	200	20
Calibration Level 8	Cal Standard 40 ppbv	200	40
ICV	Intermediate ICV 10ppb	200	10

Prepare in 6L Summa Canister

Intermediate Ethanol Calibration Standard at 500ppbv/v

1. Fill a 44 ml VOA vial with VOA free water. Remove 197ul of water from the vial.
2. Add 197 ul of >99.5% Ethanol neat material
3. Cap and shake/roll vial for 1 minute
4. Inject 10ul of the prepared water/ethanol mix into a fully evacuated 15 liter summa canister
5. Pump the syringe plunger 5 times to insure complete transfer of material
6. Immediately fill the canister to 22 psig with zero air.

Calibration Level	Working Calibration Standard	Volume added (mL)	Concentration on Column (ppbv)
Calibration Level 1	Cal Standard 0.5 ppbv	124	5
Calibration Level 2	Cal Standard 5.0 ppbv	309	10
Calibration Level 3	Cal Standard 10ppbv	464	15
Calibration Level 4	Cal Standard 15 ppbv	618	20
Calibration Level 5	Cal Standard 20 ppbv	1237	40
Calibration Level 6	Cal Standard 40 ppbv	3092	100

DC-SOP49966 Management, Retention, Storage and Archive of Data Records
EHS-SOP47863 Tracking and Collecting of Hazardous Waste
EHS-SOP48269 Procedure for Acceptance and Handling of USDA Regulated Domestic and Foreign Soil
EHS-SOP49961 Burlington Laboratory Corporate Safety Manual Addendum and Contingency Plan
EQ-SOP48255 Canister Cleaning, Certification and Sampling Kit Preparation
EQ-SOP52272 Traceability, Handling, & Storage of Standards & Reagents
ML-SOP49965 Procedures for Control Charts, Trend Analysis and In-House Limits
NC-SOP49967 Nonconformance and Corrective Action Procedures
QA-QM52862 Quality Assurance Manual
SM-SOP49959 Sample Management
TR-SOP52264 Employee Training and Analyst Demonstration of Competence
NDSC-ETHC-SOP43862 Manual Integrations
NDSC-US-EHS-QP46060 NDSC HSE Manual

End of document

Version history

Version	Approval	Revision information	
14	03.FEB.2022		
15	21.APR.2023	Conversion to HTML format and full review	
16	29.JAN.2024	SOP Review & Update. Due to database migration, some review by analysts and approval by supervisors are documented outside of D4 in QA SOP directory.	

Title: Analysis of Organochlorine Herbicides by SW846 Method 8151A

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP describes Eurofins Edison's procedure for the analysis of select organochlorine herbicides in solid and aqueous matrices by USEPA SW846 Method 8151A using dual capillary column gas chromatography (GC) with electron capture detector (ECD). Water samples are prepared using the extraction techniques detailed in Eurofins Edison SOP No. ED-ORP-015, *Extraction of Organochlorine Herbicides in Water by SW846 Method 8151A,* current revision. Solid sample extraction procedures are detailed in Eurofins Edison SOP No. ED- ORP-023, *Extraction of Organochlorine Herbicides in Soil by SW846 Method 8151A,* current revision. Table 1 (below) lists the method target compounds as well as the analytical reporting limit for each compound by matrix.

Table 1: Analytes and Reporting Limits

Parameter	CAS No.	Soil Reporting Limits (ug/kg)	Water Reporting Limits (ug/L)	Leachate Reporting Limits (ug/L)	Waste Reporting Limits (ug/kg)
2,4-D	94-75-7	17	0.50	0.080	33
2,4,5-TP (Silvex)	93-72-1	17	0.50	0.080	33
2,4,5-T	93-76-5	17	0.50	0.080	33
Dalapon	75-99-0	17	0.50	n/a	33
Dinoseb	88-85-7	17	0.50	n/a	33
2,4-DB	94-82-6	17	0.50	n/a	33
DIcamba	1918-00-9	17	0.05	n/a	33
Dichlroprop	120-36-5	17	0.05	n/a	33
MCPA	94-74-6	1700	50.0	n/a	3300
MCPP	93-65-2	1700	50.0	n/a	3300
Pentachlorophenol	87-86-5	17	0.05	n/a	33
Picloram	1918-02-01	17	0.05	n/a	33

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 9 (Review of Work) and 19 (Test Methods and Method Validation) in the Eurofins Edison Quality Assurance Manual.

2.0 Summary of Method

2.1. A measured volume or weight of sample (30 g for soil, 1000 ml for water, 15 ml for leachates) is extracted using the appropriate preparation SOP (see Section 1.1). The final volume is 10 ml in hexane (soil and water) and 5 ml in hexane (leachates).

2.2. Water samples are extracted with diethyl ether and then esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

2.3. Soil and waste samples are extracted and esterified with diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

2.4. Samples are analyzed after all the necessary calibration and QC checks have been performed as described herein. Since quantitative values for organochlorine herbicide analyses are based on the methyl ester derivatives of the free acid herbicides, the results must be adjusted for the difference in molecular weight between the free acid and the methyl ester. (See Attachment 1 for Conversion Factors)

2.5. All data undergoes two documented levels of review prior to release to clients.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. 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.

4.1.1. 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 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. For complete details reference Eurofins Edison SOP No. ED-GEN-013, *Glassware Cleaning*, most current revision.

4.1.2. The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

4.2. Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably depending upon the nature and diversity of the material being analyzed.

4.3. Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure. The determination using pentafluorobenzylation is more sensitive, and more prone to interferences from the presence of organic acids or phenols than by methylation.

4.4. 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. 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.

4.5. Organochlorine 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.

4.6. Sample extracts should be dry prior to methylation or poor recoveries may result.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation:

6.1.1. The system used is an Agilent Technologies (Avondale, PA) model 5890A or 6890 Gas Chromatograph (GC). It is equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine herbicides. All operations are as automated as possible with the equipment utilized.

6.1.2. Injection system: Dual tower Agilent Technologies 7673 Autosampler, or equivalent. Sample injection is accomplished by dual autoinjectors. A robot arm that shuttles samples between the sample tray and the injector turret services the auto injectors.

6.1.3. The samples are injected into a split/splitless injection port. The injection port is normally operated in splitless mode during injection with electronic pressure control (EPC) providing for constant flow during oven temperature programming.

6.1.4. Liners: The injection ports are each fitted with replaceable, heavy-walled glass liners. Each liner contains a plug of Hexane extracted, Teflon wool approximately 1 cm in length. The Teflon wool is positioned in the liner 1 cm from the bottom for the 5890 system. The Teflon wool is positioned in the middle of the goose neck of the siltek liner used in the 6890 system.

6.1.5. GC Oven: capable of integrated temperature control between 35^{O}C and 400^{O}C. Temperature programming of the gas chromatograph is employed.

6.1.6. Two dissimilar columns are used for analysis. An Agilent Technologies DB-5, 30m x 0.53mm ID column is used for sample quantitation. The secondary column is an Agilent Technologies DB-608, 30m x 0.53mm ID megabore column.

6.1.7. Detectors: The GC is equipped with dual Electron Capture Detectors (ECD), one for each column. Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron

plasma. This is in addition to the gas exiting the column. The make-up gas consists of Ultra high purity (99.999%) Argon (95%)/Methane (5%) (a.k.a. P-5 Mixture) (P-5) and is fed from a supply separate from the carrier or injection port gas streams.

6.1.8. Data System:

6.1.8.1 **Chemstation:** The Chemstation is utilized for automation of runs and acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

6.1.8.2 **Eurofins Chrom** chromatography data processing software package. Chrom is used for post acquisition GC data processing, reporting and storage and is fully integrated with Eurofins's LIMs (TALS).

6.1.9. Final processed data is uploaded to the Eurofins LIMS (TALS).

6.2. Supplies

6.2.1 4mm splitless sleeve (Restek Catalog # 11868-773 or equivalent) - This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

6.2.2 0.8mm ID, Gold Inlet Seal (Restek Catalog # 21241 or 21318 or equivalent)

6.2.3 Snoop Leak Detector solution or equivalent.

6.2.4 Gas-tight syringes (various sizes)

6.2.5 Injection port septa

6.2.6 Pre-silanized glass wool (Supelco 2-0411 or equivalent)

6.2.7 Syringes, Assorted sizes 10ul - 1000ul; gas-tight

6.2.8 Bottles, 10 and 5ml amber screw cap with Teflon liner

6.2.9 Vials, 2ml amber screw cap with Teflon liner

6.2.10 Wheaton microvials 100ul or equivalent

7.0 Reagents and Standards

7. 1. Reagents

7.1.1. **Gases:** Ultra high purity (99.999%) Helium is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. Ultra high purity (99.999%) Argon (95%) / Methane (5%) (a.k.a. P-5 Mixture) is used as make-up gas. It is introduced to the GC via the make-up gas adapter at the end of the capillary column. They are supplied by M-G Industries (Valley Forge, PA). Both gases are supplied at tank pressures of 2000-2400 psig., for a 300 cft. tank. The tank pressure is regulated to an outlet pressure of 70 psig. Each tank is used until the tank pressure drops to less than 500 psig.

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the GC The traps are as follows:

7.1.1.1.1. Hydrocarbon trap
7.1.1.1.2. H_2O trap
7.1.1.1.3. O_2 scrubber

7.1.2. All sample extracts are in hexane (reference the sample prep SOPs detailed in Section 2.0 above).

7.1.2.1. Hexane, Pesticide Grade, Baxter 217-4 or equivalent.

7.1.3. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7. 2. Standards

7.2.1. Standards are purchased as concentrated solutions (see Section 7.2.2). Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent as described in Section 7.3.

7.2.2. **Standard mixes and sources:**

Table 2:
Herbicide Standard Mixes and Sources*

Standard Name ("Lab Name")	Concentration (ug/ml)	Source
Acid Herbicide Spiking Mix 2 ("Spiking Mix")	varies (in acetone)	Supelco Catalog # 861259
Underivatized Chlorinated Herbicides ("10 Compound Spiking Mix")	Varies (in methanol)	Accustandard Catalog #M-8150A)
EPA 8270 Herbicide Ester Mix ("Calibration Mix")	2000 (in hexane)	Supelco Catalog # 48474
Pentachloroanisole ("Pentachloroanisole")	100 (in methanol)	Accustandard Catalog # P-199S
Picloram methyl ester ("Picloram methyl ester")	Neat	Chem Service Catalog # F2155
Methyl Derivatives of Chlorinated Herbicides ("Calibration Mix - 10 compound")	Varies (in methanol)	AccuStandard Catalog # M-8150
Pentachlorophenol ("Pentachlorophenol spike")	1000	Spex Certiprep Catalog #

Table 2: Herbicide Standard Mixes and Sources*		
Standard Name ("Lab Name")	**Concentration (ug/ml)**	**Source**
		S-2950
Picloram (4-amino-3,5,6-trichloropicolinic acid) ("Picloram Acid Spiking Standard")	Neat	Chem Service. Catalog No. F2041
2,4-DCAA Acid Spike Mix ("Surrogate Spiking Solution")	100 (in acetone)	Supelco Catalog # 861271
2,4-DCAA methyl ester ("Surrogate Solution")	1000 (in hexane)	Supelco Catalog # 861272

* May be substituted with equivalent standards from alternate sources.

Table 3: Components of Herbicide Standard Mixes			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
2,4-dichlorophenoxyacetic acid (2,4-D)	Supelco 861259	Spiking Mix	50
2-(2,4,5-Trichlorophenoxy)propionic acid (2,4,5-TP aka Silvex)	Supelco 861259	Spiking Mix	20
2,4,5-Trichlorophenoxyacetic acid (2,4,5-T)	Supelco 861259	Spiking Mix	20
2,4-D methyl ester	Supelco 48474	Calibration Mix	2000
2,4,5-T methyl ester	Supelco 48474	Calibration Mix	2000
Silvex methyl ester	Supelco 48474	Calibration Mix	2000
2,4-D methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
Dalapon methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
2,4-DB methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
Dicamba methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
Dichloroprop methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
Dinoseb methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
MCPA methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	10000
MCPP methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	10000
2,4,5-T methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
2,4,5-TP methyl ester	Accustandard M-8150	Calibration Mix – 10 compound	100
2,4-D	Accustandard M-8150A	Spiking Mix – 10 compound	100
2,4-DB	Accustandard M-8150A	Spiking Mix – 10 compound	100
2,4,5-T	Accustandard M-8150A	Spiking Mix – 10 compound	100
Silvex	Accustandard M-8150A	Spiking Mix – 10 compound	100

Table 3:
Components of Herbicide Standard Mixes

Parameter	Catalog Nos.	Lab Standard Name	Concentration (ug/ml)
Dalapon	Accustandard M-8150A	Spiking Mix – 10 compound	100
Dicamba	Accustandard M-8150A	Spiking Mix – 10 compound	100
Dichloroprop	Accustandard M-8150A	Spiking Mix – 10 compound	100
Dinoseb	Accustandard M-8150A	Spiking Mix – 10 compound	100
MCPA acid	Accustandard M-8150A	Spiking Mix – 10 compound	10000
MCPP acid	Accustandard M-8150A	Spiking Mix – 10 compound	100000
Pentachloroanisole	Accustandard P-199S	Pentachloroanisole	100
Pentachlorophenol	Spex Certiprep S-2960	Pentachlorophenol spike	1000
Picloram methyl ester	Chem Service Catalog # F2155	Picloram methyl ester	neat
Picloram (4-amino-3,5,6-trichloropicolinic acid)	Chem Service. Catalog No. F2041	Picloram Acid Spiking Standard	neat
2,4-Dichlorophenylacetic Acid	Supelco 861271	Surrogate Spiking Solution	100
2,4-Dichlorophenylacetic Acid methyl ester	Supelco 861272	Surrogate Solution	1000

7. 3. Standards Preparation

7.3.1. All standard stock solutions are diluted to the working concentrations with hexane or acetone (as indicated) using Class A volumetric glassware.

7.3.2. Total Herbicide Calibration Standards:

7.3.2.1 Total Herbicide Calibration Standards (10 compound analysis): The ten (10) compound/surrogate total herbicide calibration standard range is prepared when analyzing for the expanded list of herbicides. Table 4 details the preparation of the five calibration concentration levels using the 10 compound calibration mix. The standards are prepared using a methyl ester source with the final concentration adjusted to the free acid equivalent concentration using the conversion factors detailed in Attachment 1.

Table 4:
Preparation of Total Herbicide Calibration Standards (10 compound analysis)

Parameter	Level 1 Conc. (ug/ml) [1] 25.0 ul '8151-Herbicide Mix (10 compounds)' & 5ul 'Surrogate Solution" into 50ml Hexane	Level 2 Conc. (ug/ml) [1] 125 ul '8151-Herbicide Mix (10 compounds)' & 25ul 'Surrogate Solution" into 50ml Hexane	Level 3 Conc. (ug/ml) [1] 375 ul '8151-Herbicide Mix (10 compounds)' & 75ul 'Surrogate Solution" into 50ml Hexane	Level 4 Conc. (ug/ml) [1] 750 ul '8151-Herbicide Mix (10 compounds)' & 150ul 'Surrogate Solution" into 50ml Hexane	Level 5 Conc. (ug/ml) [1] 750 ul '8151-Herbicide Mix (10 compounds)' & 150ul 'Surrogate Solution" into 25ml Hexane
2,4-D methyl ester	0.047	0.235	0.705	1.41	2.82
2,4,5-T methyl ester	0.047	0.235	0.705	1.41	2.82
2,4,5-TP (Silvex) methyl ester	0.048	0.238	0.712	1.42	2.85
Dalapon methyl ester	0.046	0.228	0 .682	1.36	2.73
2,4-DB methyl ester	0.047	0.235	0.705	1.41	2.82
Dicamba methyl ester	0.047	0.235	0.705	1.41	2.82
Dichloroprop methyl ester	0.047	0.235	0.705	1.41	2.82
Dinoseb methyl ester	0.047	0.235	0.705	1.41	2.82
MCPA methyl ester	4.67	23.4	70.1	140	280
MCPP methyl ester	4.67	23.4	70.1	140	280
2,4-DCAA methyl ester (surr)	0.096	0.478	1.43	2.86	5.73

[1] The calculated final standard concentrations have been converted from the methyl ester concentration to the free acid equivalent concentration using the factors found in Attachment 1

7.3.2.2 Pentachlorophenol Calibration Standards: A pentachlorophenol /surrogate total herbicide calibration standard range is prepared when analysis is required for this compound. Table 5 details the preparation of the five calibration concentration levels. The standards are prepared using a source containing the derivatized form of the compound (pentachloroanisole) the final concentration adjusted to the free acid equivalent concentration using the conversion factors detailed in Attachment 1.

Table 5:
Preparation of Pentachlorophenol Calibration Standards

Parameter	Level 1 Conc. (ug/ml) [1] 50 ul 'Pentachloroanisole' & 10ul 'Surrogate Solution" into 100ml Hexane	Level 2 Conc. (ug/ml) [1] 25 ul 'Pentachloroanisole' & 5ul 'Surrogate Solution" into 10ml Hexane	Level 3 Conc. (ug/ml) [1] 75 ul 'Pentachloroanisole' & 15ul 'Surrogate Solution" into 10ml Hexane	Level 4 Conc. (ug/ml) [1] 150 ul 'Pentachloroanisole' & 30ul 'Surrogate Solution" into 10ml Hexane	Level 5 Conc. (ug/ml) [1] 300 ul 'Pentachloroanisole' & 60ul 'Surrogate Solution" into 10ml Hexane
Pentachlorophenol	0.048	0.237	0.712	1.42	2.85
2,4-DCAA methyl ester (surr)	0.096	0.478	1.43	2.87	5.73

[1] The calculated final standard concentrations have been converted from the deriviatized concentration to the free acid equivalent concentration using the factors found in Attachment 1

7.3.2.3 Picloram Methyl Ester Calibration Standards: A Picloram methyl ester/surrogate calibration standard range is prepared when analysis is required for this compound. Table 6 details the preparation of the five calibration concentration levels. The standards are prepared using a source containing the picloram methyl ester and the final concentration is adjusted to the free acid equivalent concentration using the conversion factors detailed in Attachment 1.

7.3.2.3.1 Prior to the preparation of the picloram calibration standards a working stock solution must be prepared using the neat material described in Table 2. This working stock solution is prepared at 9000 ug/ml by dissolving 9.00 grams of neat picloram methyl ester into 1000 ml of hexane using class A volumetric glassware. This solution is used as described in Table 6.

Table 6:
Preparation of Picloram Calibration Standards

Parameter	Level 1 Conc. (ug/ml) [1] 20 ml of Level 3 Picloram methyl ester into 100ml Hexane	Level 2 Conc. (ug/ml) [1] 33 ml of Level 3 Picloram Methyl ester into 100ml Hexane	Level 3 Conc. (ug/ml) [1] 8.5 ul Picloram Methyl ester stock (9000ug/ml) & 150 ul 'Surrogate Solution" into 100ml Hexane	Level 4 Conc. (ug/ml) [1] 17 ul Picloram Methyl ester stock (9000ug/ml) & 300 ul 'Surrogate Solution" into 100ml Hexane	Level 5 Conc. (ug/ml) [1] 34 ul Picloram Methyl ester stock (9000ug/ml) & 600 ul 'Surrogate Solution" into 100ml Hexane
Picloram	0.047	0.236	0.709	1.42	2.84
2,4-DCAA methyl ester (surr)	0.095	0.477	1.43	2.86	5.73

[1] The calculated final standard concentrations have been converted from the deriviatized concentration to the free acid equivalent concentration using the factors found in Attachment 1

7.3.3. Herbicide Spiking Standard (LCS/MS/MSD):

7.3.3.1 Herbicide QC Spiking Standard Mix (10 compound analysis): The ten (10) compound spiking standard is prepared and used when analyzing for an expanded list of herbicides. The 10 compound spiking mix is prepared using volumetric glassware as detailed in Table 7 (below) using a concentrated solution of free acid herbicides (Accustandard Catalog # M-1850A; see Tables 1 and 2):

Table 7: Preparation of Total Herbicide 10 Compound Spiking Mix	
Parameter	**Final Conc. (ug/ml)** 5.0 ml 'Spiking Mix -10 compounds)' into 10ml Acetone
2,4-D	50
2,4,5-T	50
2,4,5-TP (Silvex)	50
Dalapon	50
2,4-DB	50
Dicamba	50
Dichloroprop	50
Dinoseb	50
MCPA	5000
MCPP	5000

The concentrate is used for spiking of the following quality control sample types: MS, MSD, and LCS. For spiking instructions see the appropriate herbicide prep SOP referenced in Section 1.1.

7.3.3.2 **Pentachlorophenol QC Spiking Standard:** The pentachlorophenol spiking standard is prepared and used when analyzing for this herbicide. The pentachlorophenol spiking mix is prepared using volumetric glassware by diluting 1ml of the concentrated pentachlorophenol solution Spex # S-2950; see Tables 1 and 2) into a final volume of 10ml acetone. The concentrate is used for spiking of the following quality control sample types: MS, MSD, and LCS. For spiking instructions see the appropriate herbicide prep SOP referenced in Section 1.1.

7.3.3.3 **Picloram Acid QC Spiking Standard:** The picloram acid spiking standard is prepared and used when analyzing for this herbicide. The standard is prepared using a source containing the picloram free acid. Prior to the preparation of the picloram acid spiking standard a working stock solution must be prepared using the neat material described in Table 2. This working stock solution is prepared at 10000 ug/ml by dissolving 10.00 grams of neat picloram free acid into 1000 ml of hexane using class A volumetric glassware

7.3.3.3.1 Prepare the 5000 ug/ml picloram spiking solution by diluting 500ml of the working stock solution (see Sec. 7.3.3.4) into 1000 ml of hexane using class A volumetric glassware. For spiking instructions see the appropriate herbicide prep SOP referenced in Section 1.1

7.3.4. **Herbicide Surrogate Spiking Standard (**2,4-DCAA Acid Spike Mix**):** The herbicide surrogate spiking solution is a concentrated 10 ml solution of 2,4-DCAA at a concentration of 100 ug/ml. (Supelco Catalog # 861271, see Tables 1 and 2). The concentrate is used without further dilution prior to the spiking of samples.

7.3.5. **Initial Calibration Verification Standard:** The ICV is prepared as detailed in Section 7.3.2 (dependent upon the compound being analyzed) and must be from a separate lot or separate source from the standards used in the Initial Calibration Range (the analyst must confirm and document that a separate source or lot is used).

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass	1L amber	Cool 4 ± 2ºC	7 days to extraction; 40 days to analysis	USEPA SW846
Soil/Sediment	Glass	4 oz wide mouth	Cool 4 ± 2ºC	14 Days to extraction; 40 days to analysis	USEPA SW846

8.1. Extracts must be stored under refrigeration (Cool 4 ± 2ºC) in the dark and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD is randomly selected by the organic prep lab, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into TALS (LIMS).

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to ensure that the analytical system is in control. It is also used to assess performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated acceptance criteria. If the spiked sample recovery results fall outside the laboratory generated limits (refer to the current active TALS method limit group database), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QC batch must be re-extracted and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to refer to the current active TALS method limit group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in 9.1.2.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with surrogate standard solution containing 2,4-DCAA (see Section 7.3.4). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to refer to the current active TALS method limit group database). If the surrogate recovery limits are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.2 Instrument QC

9.2.1. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.1.1. ***Initial Calibration Range*:** A five-point calibration range is analyzed for each target compound using herbicide methyl ester standard mixes. Standards are prepared following the instructions in Section 7.3.

9.2.1.2. ***Initial Calibration Verification (ICV):*** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed.

The ICV is prepared as detailed in Section 7.3.2 (dependent upon the compound being analyzed) and must be from a separate lot or separate source from the standards used in the Initial Calibration Range (the analyst must confirm and document that a separate source or lot is used).

9.2.2. Continuing Calibration Verification (CCV): A mid-point Continuing Calibration Verification (CCV) must be analyzed every 12-hours or 20 samples (whichever is more frequent) and at the end of each analytical sequence. The CCV consists of the Level 3 calibration standard (~700 ppb) detailed in Section 7.3

9.2.3. Calibration Acceptance Summary

9.2.3.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. All gas chromatographs used for pesticides analysis at Eurofins Edison are equipped with electronic pressure control (EPC). The use of EPC results in little retention time variability between analyses. Accordingly, retention time variability for the purpose of retention time window determination for standards analysis is extremely small. The default retention time window option must therefore be employed as follows to accommodate the excellent precision of EPC equipped systems.

9.2.3.1.1 Obtain the retention time for all single component compounds from the analysis of the midpoint standard for the calibration curve. Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. Apply the retention time window data in the table in Attachment 2 to its corresponding compound (ex., mid-point RT ±0.10 minute). Calculate absolute retention time windows for each analyte and surrogate on each chromatographic column and instrument.

9.2.3.1.2 New retention time windows must be established when whenever a chromatographic column is replaced or a new detector is installed .Whenever the observed retention time of each analyte and surrogate is outside of the established retention time window, the analyst is advised to determine the cause and correct the problem before continuing analyses.

9.2.3.2. Initial Calibration Range: External standard calibration is employed for this method. The response factor (defined as the

ratio of the area to the standard concentration) is calculated for each analyte at each calibration concentration. The average response factor may be used for quantitation if the % RSD across the 5 point range is <20%. Alternatively, linear regression may be used if the correlation coefficient (r1) is ≥ 0.990 (note: if the linear regression model is used the curve must NOT be forced through the origin). Calibration is checked every 12 hours or after every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single response pesticide standards.

9.2.3.2.1 Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

RF = Response Factor

9.2.3.2.2 If the % RSD across the 5 point range is <20% the calibration can be assumed to be linear and the average response factor can be used to calculate concentrations of target compounds in samples.

9.2.3.2.3 If the % RSD is >20% for any given compound, a first order linear regression can be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r^1 (Correlation Coefficient) value must be ≥ 0.990 for the calibration to be acceptable. Calibration is checked every 12 hours or after every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single component pesticide standards.

9.2.3.3. Relative Standard Error: The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation. The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.

Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (r) and Coefficient of Determination (r_2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on r. As a result, a curve may have a very good correlation coefficient (>0.990) while also having > 100% error at the low point

9.2.3.4. Initial Calibration Verification (ICV): An ICV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed immediately after an initial calibration range. The calculated concentration of the ICV must be within ±15%D of the expected concentration. Should the %D exceed 15% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 15% after a single ICV reinjection, a new Initial Calibration Range must be analyzed.

9.2.3.5. Continuing Calibration Verification (CCV): A CCV will consist of an ICAL standard at or near the midpoint of the Initial Calibration Range analyzed after each 10 samples (every 12 hours at minimum). The calculated concentration of the CCV must be within ±15%D of the expected concentration. Should the %D exceed 15% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 15% after a single CCV reinjection, a new Initial Calibration Range must be analyzed.

Step	*Standards*	*Type*	*Control Limit*	*Frequency*
Method # 8151A Instrument QC Summary				
Initial Calibration	*5 point*	*Average response factor or 1st order linear regression*	*For average RF: <20%RSD all analytes. For linear regression: r ≥0.990 or <20%RSE*	*As required when ICV or CCV do not meet requirements*
ICV	*Separate source (midpoint)*	*Average*	*± 15%D*	*After every initial calibration*
CCV	*ICAL source (midpoint)*	*Average*	*± 15%D*	*1 in 10 or fewer samples (every 12 hours minimum)*

10.0 Procedure

10.1. Gas Chromatograph Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified.

Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. Injection System: A split/splitless injection port with electronic pressure control (EPC) is used. Thirty seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port. The EPC is used in constant flow mode.

10.1.2.1. For herbicide analysis the normal operating conditions of the injection port are as follows:

Injection port Temperature:	275^{o}C
Temperature Rate:	10 deg C/minute
Inlet pressure	4.0 psi
Initial Temperature:	55 deg C
EPC:	Constant flow
Detector temperature	300C

10.1.2.1.1 For pentachlorophenol analysis the following injection port settings are used:

Injection port Temperature:	250^{o}C
Temperature Rate:	4 deg C/minute
Inlet pressure	7.9 psi
Initial Temperature:	190 deg C
EPC:	Constant flow
Detector temperature	300C

10.1.2.2. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. The plug of glass wool is located in the liner between the double goose neck.

10.1.2.3. This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.4. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be

changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.5. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.6. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.7. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.3. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.3.1. The standard oven program for total herbicide analysis is :

Initial Temp	Time	Rate	Final Temp	Time
55°C	3.0 min	10°/min	244°C	11.5 min

10.1.3.2. The standard oven program for TCLP herbicide analysis is :

Initial Temp	Time	Rate	Final Temp	Time
190°C	3.0 min	4.0°/min	238°C	0.00 min

10.1.4. Detectors: Detectors operate at 300°C and need to be supplied with 40-60 ml/min total flow. They are essentially maintenance free on a day-to-day basis. They are routinely baked out at 325°C to remove persistent contaminants. On occasion the detectors may be baked out at a higher temperature to remove contaminants with an extremely high boiling point (CAUTION: Do not exceed the maximum detector temperature of 380°C).

10.1.4.1. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.5. Chemstation: The HP Chemstation application is used for automation of runs and acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.6. Eurofins's Chrom chromatography data processing application is utilized for the processing of ChemStation chromatography data files. Calibrations, verification standards and samples are processed using this database. All of the processed files are imported into the TALS database. Data is processed and first level reviewed by the analysts. Senior analysts or managers do the second level review and update the status of the job to 'Lab Complete' in TALS. All client reports are generated in TALS.

10.1.7. Eurofins LIMS (TALS): All data files processed in Chrom are then imported to into TALS. The data files are reprocessed in TALS after linking the appropriate reagents and standards to the batch. All samples and quality control runs are reviewed. A TALS non-conformance memo (NCM) is written for any QC outlier or situation requiring additional documentation. Samples and QC results are assigned to primary/secondary column as per reporting requirements. Files that are not reported are marked rejected. All data files that are being reported are first level reviewed in TALS by the analyst. Second level TALS review is done by a trained peer or supervisor. Once second level review is completed in TALS reports are generated and reviewed after which the job is marked complete.

10.2. Analytical Sequences

10.2.1. Calibration Ranges:

10.2.1.1 Herbicides are calibrated using a five-point calibration range. Standards are prepared as detailed in Section 7.3 and analyzed under the instrumental conditions detailed in Section 10.1 as appropriate for total or TCLP herbicide analysis.

10.2.1.2 A calibration factor, defined as the ratio of the response to the amount injected, is calculated for each analyte at each standard concentration. The average calibration factor (if the %RSD across the five-point range is <20%) or a linear calibration not through the origin (if the correlation coefficient (r1) is $\geq$ 0.99) is used for quantitation. Calibration is

checked every 12 hours or after every ten (10) samples, whichever comes first, by injection a calibration check standard.

10.2.1.3 Generating a calibration table: After the five calibration standards have been run, the data files are copied over to the Chrom data system. A new Chrom batch is created and the method file from the previous Chrom batch is copied to the new Chrom batch.

10.2.1.4 The data files representing the five levels of calibration are also copied to the new Chrom batch and processed with the method file. The integration of the five levels is checked for consistency in Chrom Review.

10.2.1.5 Linear Calibration. Check the percent relative standard deviation (% RSD) of the calibration factors for each individual analyte. If the % RSD is less than 20% over its working range, the linearity of the range is assumed. The average value of the calibration factors is used for quantitation of all the samples and continuing standards.

10.2.1.6 Linear Calibration Using Least Squares Regression. If the % RSD is >20% for any given compound, a first order linear regression can be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r^1 (Correlation Coefficient) value must be $\geq$ 0.99 for the calibration to be acceptable.

10.2.1.7 Since the calibration is performed using standards made from methyl ester compounds (compounds not esterified by application of this method), then the calculation of free acid herbicide concentrations must include a correction for the molecular weight of the methyl ester versus the acid herbicide (see Attachment 1).

10.2.2. Calibration Check Standards:

10.2.2.1. **Initial Calibration Verification** (ICV) is analyzed and evaluated immediately after the initial calibration range as detailed in Sections 7.3 (standard prep) and 9.2 (instrument QC).

10.2.2.2. **Continuing Calibration Verification** (CCV): is analyzed and analyzed after each 10 sample injections as further detailed in Sections 7.3 (standard prep) and 9.2 (instrument QC). The CCV is also analyzed at the end of each analytical sequence as a closing calibration check standard.

10.2.2.3. Note that samples are always quantified against the average calibration factor from the initial calibration range (or from the initial calibration curve). Calibration check standards serve only

to confirm the validity of the initial calibration range and are never used to quantify sample concentrations.

Calibration Controls	Sequence	Control Limit
Calibration Standards	5-point (minimum) linearity	≤20% RSD
Cont. Cal. Verif. (CCV)	Prior to / after every 10 injections	Prior to/after every 10 injections
RT Windows (RTW)	Init. CCV determines midpt. of RTW	±3X SD

10.2.3. Idealized Analytical Sequence

10.2.3.1. Analysis Sequence: The automation of the GC runs is accomplished via the "SEQUENCE" macro of the Chemstation.

10.2.3.2. The Sequence File: The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle

10.2.3.3. It is common practice to evaluate the instrument status, and then complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro.

10.2.3.4. An idealized analytical sequence is presented below:

Ideal Analysis Sequence*
Instrument Blank (solvent injection)
Initial Calibration Range Standards (5)
ICV
CCV
Method Blanks and LCS
Client Samples (10)
CCV
Client Samples (10)
CCV*

*After the analysis of ten sample injections or 12 hours of analysis, a CCV must be successfully analyzed.

10.3. Documentation

10.3.1. After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor, method, job

number, QC Batch number, extraction date, lab prep batch, Chrom batch, signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (e.g., 'O.K'., 're-run at dilution', 'non-inject', etc.).

10.4. Dual Column Approach

10.4.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. If the difference between the dual columns results in ≤40% RPD report the higher concentration.

10.4.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows established from the most recent preceding calibration verification.

10.4.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P.

10.4.4. If the surrogates on one column are very different (>40% RPD) compared to the other column, this may be indicative of a bad injection or column blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.4.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the compliant column. If the falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.4.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.4.6.1 An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.

10.4.7 In summary, the flow chart in Attachment 3 presents a recommended rational approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.5. Data Processing

10.5.1. Process all samples, QC samples, and blanks using Chrom. Evaluate the QC samples (Method Blank, MS/MSD, and LCS) against the criteria detailed in Section 9.1. Take corrective action as detailed in that section.

10.5.2. Evaluate chromatograms for carryover and cross-contamination. Any samples suspected of having carryover must be re-prepped and re-analyzed. To make sure there is no carryover, reagent water is injected before the re-prepped sample.

10.5.3. Any compound concentration that exceeds the concentration of the calibration range must be diluted and re-analyzed.

10.5.4. The reporting limit is based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and %moisture (No unqualified analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. $$\text{Concentration = mg/kg or L} = \frac{C \times V \times D}{W}$$

Where:
C = sample concentration in extract (ppm)
V = Volume of extract (mL)
D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared

11.4. Relative Standard Error (RSE)

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i-1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level i

.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification:

The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.4. Training Requirements

Refer to Eurofins Edison SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

The following waste streams are generated when using this method:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the

waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

Teris Profile Number: 50016624
Onyx Profile WIP Number: 545240

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method 8151A: Organochlorine Herbicides by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2. Eurofins Edison SOP No. ED-ORP-015, Extraction of Organochlorine Herbicides in Water by SW846 Method 8151A, current revision.

15.3. Eurofins Edison SOP No. ED- ORP-023,Extraction of Organochlorine Herbicides in Soil by SW846 Method 8151A, current revision.

15.4. Eurofins Edison Document No. ED-QA-LQM, Laboratory Quality Manual, current revision.

15.5. Eurofins Corporate Document No. CW-E-M-001, Environmental Health and Safety Manual, current revision.

15.6. Eurofins Corporate Quality SOP No. CA-Q-S-001, Solvent & Acid Lot Testing & Approval, current revision.

15.7. Eurofins Edison SOP No. ED-GEN-023, Bulk Solvent Testing and Approval, current revision.

15.8. Eurofins Edison SOP No. ED-GEN-013, Glassware Cleaning, most current revision

15.9. Eurofins Edison SOP No. ED-GEN-022, Training, current revision.

16.0. Method Modifications:

N/A

17.0. Attachments

Attachment 1: Herbicide Free Acid Conversion Chart

Attachment 2: Retention Time (RT) Windows for Primary Herbicides/Surrogate

Attachment 3: Dual Column Reporting Flowchart

18.0. Revision History

- Revision 12, dated 17 Oct 2022
    - Updated throughout to reflect Eurofins branding.
    - Section 9.2.3.3: Included details of evaluation of initial calibration for Relative Standard Error (RSE).
    - Section 11.4: added formula for calculation of RSE.
    - Section 12.2 added: details the annual LLOQ verification requirement.

- Revision 11, dated 22 March 2021
    - Added Section 10.5.4 which details the establishment of the reporting limit (RL).

- Revision 10, dated 10 December 2020
    - Updated throughout to reflect Eurofins branding.
    - Revised references to Quality Manual section numbers as required

- Revision 9, dated 22 February 2016
    - Revised throughout to correct the spelling of Picloram (4-amino-3,5,6-trichloropicolinic acid).

- Revision 8, dated 12 August 2013
    - Revised throughout to reflect the lab's implementation of the TestAmerica Chrom data system.
    - Deleted Section 7.3.2.1 (Total Herbicide Calibration Standards, 3 Compound Analysis). Renumbered remaining sections accordingly.
    - Deleted Section 7.3.1.1 (Herbicide QC Spiking Standard, 3 Compound Analysis). Renumbered remaining sections accordingly.
    - Deleted Table 4 (Preparation of Total Herbicide Calibration Standards, 3 Compound Analysis). Renumbered remaining tables accordingly.
    - Revised 2,4,5-TP (Silvex) methyl ester concentrations (all levels) and Dalapon (level 3) in Table 4 (formerly Table 5)
    - Revised Pentachlorophenol concentrations (levels 2 and 3) in Table 5 (formerly Table 6)
    - Revised Picloram concentrations (all levels) and 2,4-DCAA methyl ester (level 4) in Table 6 (formerly Table 7)

- Revision 7, dated 15 August 2011
    - Section 7.3.3.4: Corrected weight of neat Picloram from 9 grams to 10 grams
    - Attachment 3: revised to reflect current practice.
    - Revised 'Distribution' footer on Cover Page to reflect current practice.

- Revision 6, dated 28 October 2009

    - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
    - Section 1.1. Added Table 1, *Analytes and Reporting Limits*
    - Section 1.1 Added reference to Quality Assurance Manual for method modifications.
    - Section 1.1: Expanded to include references to applicable prep and cleanup SOPs.
    - Section 3: revised to reference new location for definitions.
    - Section 5: Revised to include most up to date corporate health and safety references and information.
    - Section 7.2.2: Added table detailing components found in the various standards mixes.
    - Section 7.3: Updated the instructions for preparation of standards.
    - Section 7.3: Added tables with calibration standards prep details.
    - Section 8: Updated with additional details including a table outlining containers, preservation and holding times for waters and soils.
    - Section 9.1: Expanded QC sample preparation, analysis, evaluation and corrective action details.
    - Section 9.2: Expanded details of preparation, analysis, evaluation and corrective action for initial and continuing calibration and calibration verifications.
    - Section 10.2.3: added procedures for dual column evaluation.
    - Section 10.4.1: Updated Idealized Analytical Sequence table.
    - References: Expanded to include more specific SOP references
    - Section 18: Added this Revision History section
    - Throughout document: added references to TestAmerica LIMS (TALS).
    - Attachments: Added Attachment 3: Dual Column Reporting Flowchart

Attachment 1

Herbicide Free Acid Conversion Chart

Analyte	Free Acid Molecular Weight	Methyl Ester Molecular Weight
Dalapon	142.97	156.97
2, 4 - D	221.00	235.00
2, 4, 5 - TP (Silvex)	269.50	283.50
2, 4, 5 - T	255.50	269.50
Dinoseb	240.22	254.22
2, 4 - DB	249.09	263.09
Dicamba	221.04	235.04
Dichloroprop	235.05	249.05
Picloram	241.48	255.48
Pentachlorophenol	266.50	280.50
MCPA	200.63	214.63
MCPP	200.63	214.63
DCAA	297.13	311.13

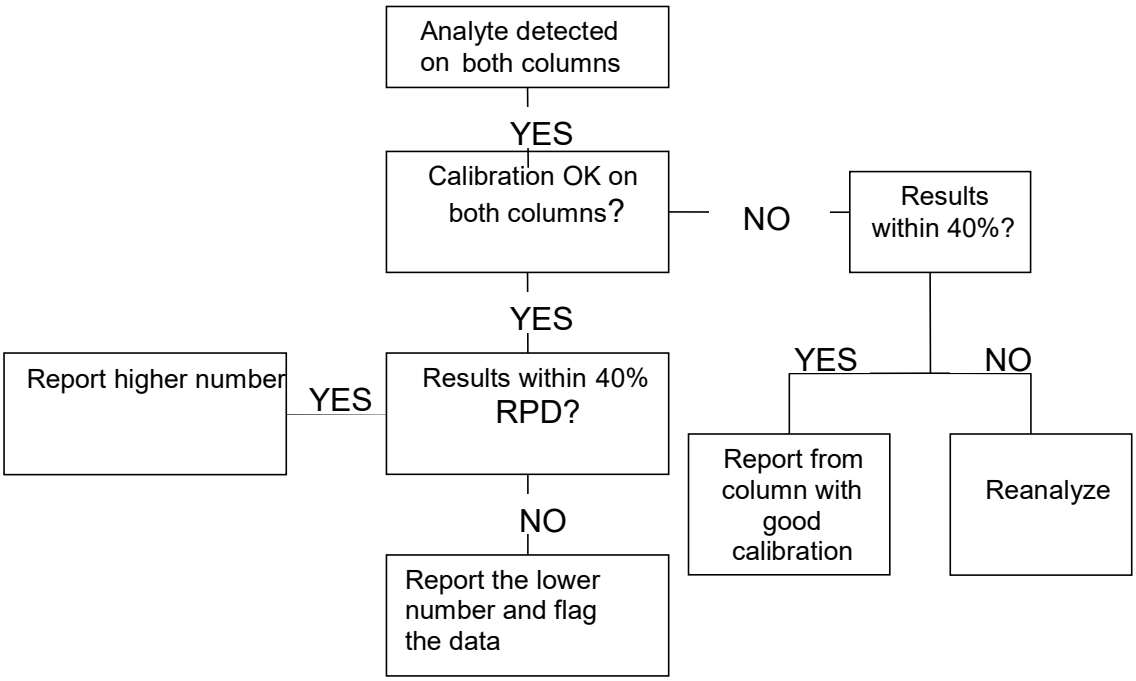
$$[\text{Free Acid}] = \frac{[\text{Methyl Ester}] \times \text{Free Acid Molecular Weight}}{\text{Methyl Ester Molecular Weight}}$$

Attachment 2

Retention Time (RT) Windows For Herbicides/Surrogate

Compound	RT Window (minutes)	
	Total Herbs	TCLP Herbs
2,4-D	*± 0.10*	*± 0.05*
2,4,5-TP (Silvex)	*± 0.10*	*± 0.05*
2,4,5-T	*± 0.10*	*± 0.05*
Dalapon	*± 0.10*	
2,4-DB	*± 0.10*	
Dicamba	*± 0.10*	
2,4-DP (Dichloroprop)	*± 0.10*	
Picloram	*± 0.10*	
Pentachlorophenol	*± 0.10*	
MCPA	*± 0.10*	
MCPP	*± 0.10*	
Picloram	*± 0.10*	
DCAA (surrogate)	*± 0.10*	*± 0.10*

Attachment 3
Dual Column Reporting Flowchart



Title: SW846 Method 8082A, Analysis of Polychlorinated Biphenyls by Gas Chromatography

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Approvals (Signature/Date):

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1.0 Scope and Application

This method is used to quantify specific polychlorinated biphenyls (PCBs) as Aroclors (see Table 1 below) in extracts from aqueous, soil, sludge, leachate, wipe or oil matrices by direct injection dual capillary column gas chromatography using SW846 Method 8082A. An electron capture detector (ECD) is employed for detection.

1.1 Analytes, Matrix(s), and Reporting Limits

The specific analytes determined by this method are identified in Table 1.

Table 1
Polychlorinated Biphenyls

Compound Name	CAS Registry No.
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

The routine Eurofins Edison reporting limits (RLs) by analyte and matrix are summarized in Table 2 (below).

Table 2
Reporting Limits by Matrix

Parameter	Soil	Soil	Water	Leachate	Oil	Wipe
	Reporting Limits (ug/kg) LOW Level	Reporting Limits (ug/kg) MED Level	Reporting Limits (ug/L)	Reporting Limits (mg/L)	Reporting Limits (ug/kg)	Reporting Limits (ug/wipe)
Aroclor-1016	67	500	0.50	0.0050	1000	0.40
Aroclor-1221	67	500	0.50	0.0050	1000	0.40
Aroclor-1232	67	500	0.50	0.0050	1000	0.40
Aroclor-1242	67	500	0.50	0.0050	1000	0.40
Aroclor-1248	67	500	0.50	0.0050	1000	0.40
Aroclor-1254	67	500	0.50	0.0050	1000	0.40
Aroclor-1260	67	500	0.50	0.0050	1000	0.40
Aroclor-1262	67	500	0.50	0.0050	1000	------
Aroclor-1268	67	500	0.50	0.0050	1000	------

The most current MDLs and RLs for this method can be found in the active Eurofins LIMS (TALS) SW846 8082A Method Limit Group (MLG) database.

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and Section 19 (*Test Methods and Method Validation)* of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Samples undergo a preparation step prior to analysis by SW846 Method 8082A. A measured volume or weight of sample is extracted using the appropriate matrix-specific sample extraction technique (reference the applicable Organic Sample Prep SOPs listed below). The extract is exchanged into hexane and concentrated to a final volume between 1 and 20 ml depending upon the prep technique used.

2.1.1. Aqueous and leachate samples are extracted at a neutral pH using either SW846 Method 3510C (SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel*) or SW846 Method 3511 (SOP No. ED-ORP-051: *Extraction of Semivolatile Organic Compounds in Water by Microextraction using SW846 Method 3511*)

2.1.2. Wipe samples are extracted using SW846 Method 3550B: Sonication (*SOP No. ED-ORP-018: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B*)

2.1.3. Solid samples are extracted using SW846 Method 3546 (*SOP No. ED-ORP-0044: Procedure for the Microwave Extraction of Solids,SW846 Method 3546*).

2.1.4. Organic liquids are prepared using SW846 Method 3580A (*SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A*).

2.1.5. Extract cleanup steps are employed as need depending on the nature of the matrix interferences encountered. Suggested cleanups include SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*), SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*) and SW846 Method 3665A (*SOP No. ED-ORP-022, Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*) for heavy organic interferences.

2.2. After cleanup, the extract is analyzed by injecting a known volume of sample into a gas chromatograph equipped with a dual wide-bore fused silica capillary columns and dual electron capture detectors (GC/ECD). The GC is temperature programmed to separate and detect the analytes recovered during the extraction step. Quantitation is accomplished by comparing the area response of each target analyte relative to an internal standard established through a five-point initial calibration (six points for second order regression). Specific calibration and quality control steps are detailed in this SOP and meet the specification of SW846 Method 8082A.

2.3. Samples are analyzed only after all the necessary calibration and QC checks have been performed.

2.4. Acquired data from sample analysis is manually reviewed. Secondary column confirmation of target compounds and quantitation are conducted by the analyst as required.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences from phthalate esters introduced during sample preparation can pose major difficulties for PCB determinations.

4.1.1. Interferences from phthalate esters can be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting PCBs. Sulfur contamination should be expected with sediment samples. Employ SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*) for removal of sulfur.

4.3. Co-eluting chlorophenols are eliminated by using SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*),

4.4. Interferences from other organic compounds can effectively be removed using a sulfuric acid treatment, SW846 Method 3665A (*SOP No. ED-ORP-022, Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*). This destructive technique can be employed only when the sample extract is being analyzed solely for PCBs (i.e., it is not to be used prior to analysis for pesticides).

4.5. Compounds extracted from the sample matrix to which the detector will respond, such as single-component chlorinated pesticides, including the DDT analogs (DDT, DDE, and DDD) will cause interference. A standard of the DDT analogs should be injected to determine which of the PCB or Aroclor peaks may be subject to interferences on the analytical columns used. There may be substantial DDT interference with the last major Aroclor 1254 peak in some soil and sediment samples. A System Performance Check (DDT/Endrin) is analyzed daily to assist in evaluation of potential DDT interference with this Aroclor 1254 peak.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas Chromatograph:

6.1.1 Agilent Technologies (Avondale, PA) model 5890/6890 Gas Chromatograph (GC), equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine pesticides and PCB's. All operations are as automated as possible with the equipment utilized.

6.1.2 Injection system: Sample injection is accomplished by a single auto injector. The auto injector is serviced by a robot arm that shuttles a single sample between the sample tray and the injector turret.

6.1.2.1 The sample is injected into a split/splitless injection port equipped with electronic pressure control (EPC). The injection port is normally operated in splitless mode during injection. The EPC is operated in the ramp pressure mode.

6.1.3 Liners: The injection port is each fitted with a replaceable, heavy-walled glass double gooseneck liner. The liner contains a plug of silanized glass wool approximately 1 cm in length. The glass wool is positioned in the liner between the double gooseneck. The liner is replaced on a regular maintenance schedule.

6.1.4 Oven and Columns: Temperature programmable gas chromatograph ovens are required, capable of integrated temperature control between 35^{O}C and 350^{O}C.

6.1.4.1 Two dissimilar columns are used for analysis. A Restek StxCLPesticides, 30m x 0.53mm ID x 0.5um film thickness column (or equivalent) is used for sample quantitation. The secondary column is a Restek StxCLPesticides II, 30m x 0.53mm ID x 0.42um film thickness column (or equivalent).

6.1.5 Detectors: Sample detection is by electron capture. The GC is equipped with dual Electron Capture Detectors (ECD), one for each column.

6.1.5.1 Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron plasma. This is in addition to the gas exiting the column. The make-up gas is fed from a supply other than the injection port.

6.2 Data System:

6.2.1 The data systems consist of Agilent Technologies GC Chemstation Revision A.08.02 and Agilent Technologies Enviroquant Chemstation G1701AA Version A.03.00 upgraded to A.03.02 which is used for acquisition and Eurofins Chrom (chromatography data processing software).

7. Reagents and Standards

7.1. Reagents

7.1.1. Gases: Ultra high purity (99.999%) Hydrogen is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. Ultra high purity (99.999%) Nitrogen is used as make-up gas. (Alternatively, ultra-high purity Helium with P-5 make-up gas may be used). Make-up gas is introduced to the GC via the make-up gas adapter at the end of the capillary column. Gases are supplied at tank pressures of 2000-2400 psig for a 300 cft tank. The tank pressure is regulated to an outlet pressure of 70 psig. Each tank is used until the tank pressure drops to less than 500 psig.

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the G.C. The traps are as follows:

- Hydrocarbon trap
- H_2O (moisture) trap
- O_2 scrubber

7.1.1.2. Both the moisture trap and the Oxygen scrubber are of the indicating type. They require either replacement or reconditioning upon color change of the active agents. Refer to the instructions for the individual traps to determine if it is still active. The hydrocarbon trap is a simple activated carbon trap. With high quality gas, it should last for an extended period of time (1-yr. minimum).

7.1.2. Solvents used in the extraction, clean up procedures and dilutions include Hexane, Methylene Chloride, and Acetone that are exchanged to Hexane prior to analysis. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis.

7.2. Standards

7.2.1. Standards are purchased as concentrated solutions. Standard compounds or mixtures for this analysis include an Aroclor 1016/1260 mix, Aroclor

1221, 1232, 1242, 1248, 1254, 1262, 1268 and the surrogate compound Decachlorobiphenyl (DCB) (packaged with the Tetrachloro-m-xylene (TCMX), a surrogate used in pesticide analysis).

NOTE: Two independent sources are used for quantitation standards and spiking standards

7.2.1.1. Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent.

7.2.2. Standards mixes and sources: *

Standard Name	Source	Concentration
TCMX/DCB Surrogate Calibration Mix	Restek 32000	200 ug/ml
TCMX/DCB Surrogate Spike Mix	Supelco 861275	10 ug/ml
Aroclor 1016 Calibration Standard	Supelco 48097	1000 ug/ml
Aroclor 1221 Calibration Standard	Restek 32007	1000 ug/ml
Aroclor 1232 Calibration Standard	Restek 32008	1000 ug/ml
Aroclor 1242 Calibration Standard	Restek 32009	1000 ug/ml
Aroclor 1248 Calibration Standard	Restek 32010	1000 ug/ml
Aroclor 1254 Calibration Standard	Restek 32011	1000 ug/ml
Aroclor 1260 Calibration Standard		1000 ug/ml
Aroclor 1262 Calibration Standard	Restek 32409	1000 ug/ml
Aroclor 1268 Calibration Standard	Restek 32410	1000 ug/ml
Aroclor 1660 Mix (Arolcors 1016 & 1260)	Restek 32039	1000 ug/ml
Aroclor 1016/1260 Calibration Standard (Second Source)	Restek 32039.sec	1000 ug/ml
1-Bromo-2-nitrobenzene (internal standard)	Restek 32279	1000 ug/ml

*Suppliers with equivalent standards may be used.

7.2.3. Aroclor 1016/1260 & Surrogate Calibration Standard Solution Preparation

Five levels of calibration standards are prepared using the above referenced Aroclor 1660 Mix (Restek – 23039) and TCMX/DCB Surrogate Calibration standard mix (Restek 32000). They are prepared as follows:

Final Concentration of Aroclor 1016/1260 (Concentration of DCB)	Volume (ul) of Aroclor 1660 Mix (1000 ug/ml)	Volume (ul) of TCMX/DCB Surrogate Calibration Mix (200 ug/ml)	Final Volume in hexane (ml)
50 ppb (25 ppb DCB) (1)	5	6.25	100
500 ppb (50 ppb DCB)	50	25	100
1000 ppb (100 ppb DCB)	1000	500	1000
1500 ppb (150 ppb DCB)	150	75	100
2500 ppb (200 ppb DCB)	250	100	100

(1): The low level Aroclor 1016/1260 standard is 50 ppb and 12.5 ppb for the surrogate DCB (prepare by making a 2x dilution of the 100 ppb standard in hexane).

7.2.4. Surrogate Spiking Solution (soil and water)

A TCMX/DCB Surrogate Spike Mix is prepared by diluting 10 ml of Restek 32000 (see Table 1 above) to 200 ml of Acetone. Final solution concentration is 10 ug/ml. For reduced volume LVI preps a secondary dilution of this mix is utilized. This is prepared by diluting 20 ml of the 10 ug/ml solution to 100 ml acetone with a final solution concentration of 2 ug/ml.

7.2.5. Aroclor 1016/1260 Spiking Solution (soil, water and wipe)

An Aroclor 1660 Mix is prepared by diluting 10 ml of Restek 32039 (See Tables 1 and 2 above) to 100 ml acetone. Final solution concentration is 100 ug/ml. For reduced volume LVI preps a secondary dilution of this mix is utilized. This is prepared by diluting 20 ml of the 100 ug/ml solution to 200 ml with a final solution concentration of 20 ug/ml. used spiking soils and waters as received from Supelco without further dilution.

7.2.6. Individual Aroclor Calibration Solutions (1221, 1232, 1242, 1248, 1254, 1262 & 1268)

A 1000 ppb calibration standard is prepared for each remaining Aroclor from the stock standards detailed in Section 7.2.1. 200ul of 1000 ug/ml individual Aroclor solution and 100ul of 200 ug/ml TCMX/DCB is diluted to 200ml with Acetone. The final concentration of surrogates is 100 ppb.

7.2.7. Aroclor 1016/1260 Initial Calibration Verification (ICV) Standard Solution Preparation

A mid-point Aroclor 1016/1260 ICV standard is prepared using the second source Aroclor 1016/1260 Calibration Standard (Restek 32039.sec) detailed in Section 7.2.2. 1000 ul of 1000 ug/ml standard along with 500 ul of 200 ug/ml TCMX/DCB surrogate standard (Restek 32000) is diluted to 1000 ml with acetone for a final ICV concentration of 1 ug/ml (1000 ppb).

7.2.8. PCB Internal Standard Spike Mix (1 ug/ml)

The PCB 1 ug/ml internal standard spike mix is prepared by dilution 500ul of 1000 ug/ml of the 1-Bromo-2-Nitrobenzene standard (Restek 32279) in to 500 ml of Hexane. 20 ul of this solution is added to all standards, QC samples and field sample extracts prior to analysis.

7.2.9. System Performance Solution (3,3-DDT and Endrin at 0.25 ug/ml):
The DDT/Endrin performance solution is prepared by taking 250 ul of 500 ug/ml DDT/Endrin Mix (Supelco Catalog No 48282) and bringing it up to a volume of 500 ml with hexane.

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 250 ml (SW3510), 40 or 60 ml (SW3511)	250 ml (SW3510), 40 or 60 ml (SW3511)	Cool 4 ± 2°C	7 days to extraction; Analyze within 40 days of extraction	SW846
Soils	Glass, 2 or 4 oz	100 g	Cool 4 ± 2°C	14 days to extraction; Analyze within 40 days of extraction	SW846

.

8.1. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

8.2. Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standard	every sample[3]	Response within -50% to +100% of most recent cal standard.

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. **Method Blanks** are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. **Laboratory Control Sample (LCS):** A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to assess method performance and serves to determine whether the methodology is in control at the time of preparation and analysis. The recoveries of the LCS must fall within lab generated acceptance criteria. If the LCS recovery results are outside QC limits, the extract is reanalyzed. If upon reanalysis the recoveries remain outside of recovery limits the following evaluations are made:

- If LCS results fall outside the laboratory generated limits with low recoveries (refer to the current TALS Method Limit Group database), the LCS and all associated samples should be re-extracted and re-analyzed.

- If LCS results fall outside the laboratory generated limits with high recoveries (refer to the current TALS Method Limit Group database), the LCS and associated sample results may be reported with an Non-Conformance Memo (NCM) detailing the issue.

9.1.3. **Matrix Spike/Matrix Spike Duplicate (MS/MSD):** A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to the current TALS Method Limit Group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated. If the LCS recoveries meet criteria the data is reported and a Non-Conformance Memo (NCM) is written.

9.1.4. **Surrogate Standards:** All samples, blanks and QC samples are spiked with a 2 component surrogate standard mix containing TCMX & DCB (see Section 7.2). The percent recovery of the DCB surrogate standard is calculated and compared to lab generated limits (refer to the current TALS Method Limit Group database). (Note: the surrogate must pass CCV criteria to be reportable and must be reported from the same column as sample target analyte results). If the DCB recovery is outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.1.5. Internal Standard: The internal standard (1-bromo-2-nitrobenzne) must elute within 30 seconds of and have an area response of 50 to 100% as compared to the most recent preceding calibration standard.

9.2. Instrument QC

9.2.1. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.1.1. *Initial Calibration Range*: Aroclors 1016/1260 and the surrogate (DCB) are calibrated using a five-point calibration range using a minimum of five (5) peaks per Aroclor. The reporting limit (RL) is equal to the low point of the calibration range. The initial calibration block must include at least one level with Aroclor 1016 analyzed separately for pattern recognition purposes (note: this run does not need to be part of the actual calibration). If the 1016/1260 calibration meets the required average RF criteria all other Aroclors are then calibrated at the anticipated midpoint of the calibration range with a single point calibration using a minimum of five (5) peaks for each Arolcor (minimum of 3 peaks for Aroclor 1221). All peaks selected must be at least 25% the peak height of the largest peak in the Aroclor (except for Aroclor 1268 where the requirement is 10%). The following Aroclors can be analyzed together: 1016/1260, 1221/1254, 1232/1262 and 1242/1268. Standards are prepared following the instructions in Section 7.2.

9.2.1.2. *Initial Calibration Verification (ICV):* An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.7 and must be from a source separate from the standards used in the Initial Calibration Range.

9.2.2. Continuing Calibration Verification (CCV): A mid-point Continuing Calibration Verification (CCV) standard (typically Aroclors 1016/1260) must be analyzed after every 20 samples at minimum. If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260. If an Aroclor other than Aroclors 1016/1260 is detected a CCV of the identified Arolcor must be analyzed within 12 hours of the sample. If that CCV does not meet criteria the sample must be reanalyzed under an acceptable CCV using the detected Arolcor. The response factors for the CCV must be within +/- 20 % of the initial calibration RF.

9.2.3. Calibration Acceptance Summary

9.2.3.1. Retention Time Windows: Retention time (RT) windows must be determined for all analytes.

9.2.3.1.1 Initial determination of RT windows.

9.2.4.1.1.1. The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration of the first CCV in the daily sequence, whichever is most recent.

9.2.3.1. **Initial Calibration Range**. The internal standard calibration technique is employed for this method. The response factor (defined as the ratio of the area to the standard concentration) is calculated for each characteristic peak in the Aroclor 1016/1260 standard at each calibration concentration. The percent relative standard deviation (% RSD) of the response factors for each individual peak in the Aroclor 1016/1260 mix on each column is then determined (both columns must pass calibration criteria).

9.2.3.1.1. Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

RF = Response Factor

9.2.3.1.2. Linear Calibration: If the % RSD is less than 20% over its working range for at least five peaks in the Aroclor 1016/1260 mix, the linearity of the range is assumed for all Aroclors over the same analytical range. Each individual peak's response factor is used for quantitation of all the samples and verification standards. The average of the value calculated for each individual peak is used to report the concentration in the samples.

9.2.3.1.3. Linear Calibration Using Least Squares Regression: If the % RSD is >20% for any given compound, a first order linear regression can be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r squared value must be > 0.99 for the calibration to be acceptable

9.2.3.1.4. **Relative Standard Error:** The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation. The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.

Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the

quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (*r*) and Coefficient of Determination (r_2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on *r.* As a result, a curve may have a very good correlation coefficient (>0.990) while also having > 100% error at the low point

9.2.3.2. Initial Calibration Verification (ICV):

9.2.3.2.1. After the initial calibration range has been analyzed, an Initial Calibration Verification standard is analyzed on each column to verify the validity of the initial calibration range. The ICV standard must be from a standard lot independent of the standards used in the initial calibration range. The verification standard for PCBs is the mid-range Aroclor 1016/1260 standard at 1000ppb. (See Section 7.2.7 for details on the preparation of the ICV).

9.2.3.2.2. At least five characteristic peaks of each Aroclor 1016/1260 plus surrogates in the ICV must be checked to verify the Initial Calibration Verification. The calculated concentration of the ICV must be within ±20%D of the expected concentration. Should the %D exceed 20%, the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 20% after a single ICV reinjection, a new Initial Calibration Range must be analyzed.

9.2.3.3. Continuing Calibration Verification (CCV) and System Performance Check:

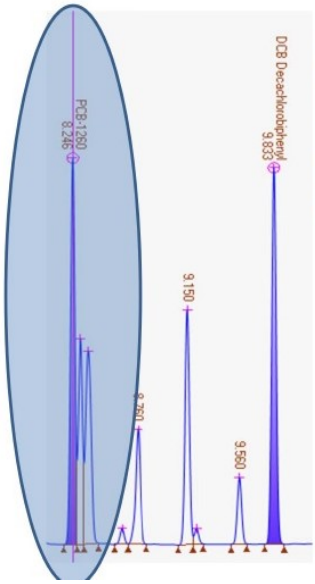
9.2.3.3.1. On each day of analysis the System Performance Check (see Section 7.2.9) is analyzed to assist in the evaluation of possible DDT interferences with the last major peak in Aroclor 1254.

9.2.3.3.2. A mid-point Continuing Calibration Verification (CCV) standard (typically Aroclors 1016/1260) must be analyzed after every 20 samples at minimum. If the samples being analyzed are being specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260.

9.2.3.3.3. If there are Aroclor hits in the associated samples then the calculated concentration of the CCV must be within

±20%D of the expected concentration on both columns. If there are no Aroclor hits in the associated samples one column may exceed the ±20%D criteria on the hight side (alternatively a low level Aroclor 1016/1260 standard may be analyzed to demonstrate adequate sensitivity. Should the foregoing criteria not be met the analyst must take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 20% after a single CCV reinjection, a new Initial Calibration Range must be analyzed. The surrogate (DCB) must also meet CCV criteria on both columns.

9.2.3.3.4. Resolution (Aroclor 1260): The CCV level Aroclor 1016/1260 standard must meet the following resolution critieria: Resolution (degree of overlap) for the triplet towards the end of the 1260 chromatogram must be <75% on <u>one</u> of the two columns used. The resolution requirement must be met for peak 1/2 and peak 2/3:



The circled triplet of peaks is observed towards the end of the 1260 pattern on columns such as CLP 1. Minimum resolution (degree of overlap) requirement between peak 1 / 2 and peak 2 / 3 is <75%. This chromatogram shows overlap of about 50% between peak 2 and 3, and 30% between peak 1 and 2.

Resolution (degree of overlap) is calculated as

[Height of the valley / (Sum of the two peak heights / 2)] x 100%

The acceptance criteria for the Initial Calibration Range, the ICV and the CCV are detailed in the table below.

Step	Standards	Type	Control Limit	Frequency
Method # 8082A				
Initial Calibration Range	50, 500, 1000, 1500 and 2500 ppb for Aroclor 1016/1260, 1000 ppb for all remaining Aroclors	*Average response factor or 1st order linear regression*	*For average RF: <20%RSD all analytes. For linear regression: r^2 ≥0.990*	*Initially and as required when ICV or CCV do not meet requirements*
ICV	*1000 ppb*	*Average*	*± 20%D*	*Once after each initial calibration*
CCV	*1000 ppb*	*Average*	*± 20%D (see Section 9.2.3.4.2 for exceptions); Acceptable Resolution*	*Every 20 samples*
System Performance Standard			*For evaluation of DDT intereference with Ar1254*	*Once daily*

10. Procedure

10.1. Gas Chromatograph (GC) Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. General Instrument Operating Conditions:

10.1.2.1. **Injection System**: A splitless injection port with electronic pressure control (EPC) is used. Seventy-five seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port.

10.1.2.2. The EPC is used in the pressure Ramp mode. The ramp pressure program is as follows:

Initial Pressure	InitialTime	Rate	Final Pressure	Hold
25 psi	0.50 min	20psi/min	15 psi	2.00 min
		8 psi/min	12 psi	6.60 min
		10.0 min	16 psi	2.00 min

10.1.2.3. For PCB analysis the normal operating conditions of the injection port are as follows:

Injection Temperature:	250ºC
Injection Port Pressure: Column flow:	25ml/min 33.2 ml/minute
Split vent flow:	60.0 ml/minute
Purge vent flow:	1.2 ml/minute
EPC:	Ramp pressure mode

10.1.2.4. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. This liner/glass wool combination provides many functions.

10.1.2.5. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.6. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.6.1. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.6.2. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.6.3. The septa should be changed each time the injection port is opened. Another routine maintenance operation

to improve column performance is the removal of the first 3 cm of the column.

10.1.2.6.4. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.2.7. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.2.7.1. The oven program and pressure ramping for PCB analysis is employed for all columns as follows:

Initial Temp	Hold Time 1	Rate1	Temp1
164°C	0.0min	12°/min	234°C
Hold Time2	**Rate2**	**FinalTemp**	**FinalTime**
2.4 min	40°/min	325°C	1.5min

10.1.2.8. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.2.9. Chemstation: The Chemstation is utilized for automation of runs and acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.2.10. Eurofins Chrom data processing software is used for the processing of the chromatography data files. Calibrations, verification standards and samples are processed and reviewed using this database. Chrom is integral to Eurofins LIMS (TALS) which is used to generate all reports.

10.2. Analytical Sequence

10.2.1. The instrument operating conditions should be set as detailed in Section 10.1.

10.2.2. Once instruments conditions have been established, the Initial Calibration Range, calibration verifications and retention time windows must be established Section 9.2.

10.2.3. The analytical sequence is established via the "SEQUENCE" macro of the Chemstation data system. The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle. It is common practice to run the calibration and/or calibration verification standards, evaluate the instrument status, and, finally, (if all meet criteria) complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro

10.2.4. An idealized analytical sequence including an Initial Calibration Range is presented in the table below.

.

Idealized Analytical Sequence with Initial Calibration Range	
Injection Number	**Identification**
1	Hexane
2	Instrument Blank
3	Aroclor-1660 Level 1 Cal Std (50 ppb)
4	Aroclor-1660 Level 2 Cal Std (500 ppb)
5	Aroclor-1660 Level 3 Cal Std (1000ppb)
6	Aroclor-1660 Level 4 Cal Std (1500 ppb)
7	Aroclor-1660 Level 5 Cal Std (2500 ppb)
8	Aroclor-1221 Level 3 Cal Std (1000 ppb)
9	Aroclor-1232 Level 3 Cal Std (1000 ppb)
10	Aroclor-1242 Level 3 Cal Std (1000 ppb)
11	Aroclor-1248 Level 3 Cal Std (1000 ppb)
12	Aroclor-1254 Level 3 Cal Std (1000 ppb)
13	Aroclor-1262 Level 3 Cal Std (1000 ppb)
14	Aroclor-1268 Level 3 Cal Std (1000 ppb)
15	Initial Calibration Verification (Aroclor 1660)
16	Hexane
17	Continuing Calibration Verification (Aroclor1660)
18 thru 37	Client samples and QC Samples (MS/MSD, LCS, Method Blank)
19	Continuing Calibration Verification (Aroclor1660) (*every 20 samples*)

10.2.5. After each 20 samples a CCV standard mix must be analyzed. If this standard fails the criteria listed in Section 9.2.3.4, all samples analyzed during the previous period must be re-analyzed with a passing CCV.

10.2.6. PCB Data Reporting: The Chrom data system calculates the concentrations of the selected Aroclor Peaks. The reporting limit is based

on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and % moisture (No unqualified analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).

10.2.7.1. The quantitative values for all confirmed analytes must agree within 40% between the primary column and the confirmation column.

10.2.7.2. If the quantitative values do not agree within 40%, the discrepancy must be noted in the report with a qualification

10.3 Dual Column Approach

NOTE: Data generated under the NJDEP DKQP requires the reporting of the higher concentration in all cases unless it can be demonstrated that interfering compounds are the cause of the higher results in which case the lower value can be reported with a narrative explanation. Other programs may also require a dual column reporting approach different than the one described below in which case the lab will report as required by that program.

10.3.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. Results are reported from the primary column unless the difference in concentration between the two columns results in ≥40% RPD in which case the lower concentration is reported (unless the client or program requirements dictate otherwise).

10.3.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows established from the most recent preceding calibration verification.

10.3.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P*.

10.3.4. If the surrogates on one column are very different (>40% RPD) compared to the other column, this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.3.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the compliant column. If the recovery falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.3.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.3.6.1. An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.

10.3.7. In some cases where the sample chromatography is complex and has largely varying peaks concentrations, the chromatographic separation may not be sufficient on the 0.53mm ID columns. In this case a confirmatory analysis on an instrument with 0.32 ID columns may be required. The supplemental data produced using analysis on the 0.32mm ID 'microbore' column may minimize overlapping and baseline interference difficulties, and better resolves potential positive identifications. Use of this alternative chromatographic technique shall be noted in the job summary and report case narrative.

10.3.8. In summary, the flow chart in Attachment 1 presents a recommended approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.4 Extract Cleanup

10.4.1. Cleanup methods are dictated by the original sample matrix and the parameters being determined.

10.4.2. Cleanup of all water samples, if needed, is performed using Sulfuric Acid Permanganate and/or TBA sulfite. Refer to Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision and Eurofins Edison SOP No. ED-ORP-022: *Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*, *SW846 Method 3665A,* most current revision. Blanks must also undergo cleanup following the same procedures as samples..

10.4.3. Cleanup of all soil samples is conducted using TBA sulfite and Sulfuric Acid Permanganate. Blanks must also undergo cleanup following the same procedures as samples.

10.4.4. Cleanup using Sulfuric Acid Permanganate effectively destroys the majority of organic material in the sample extract and should be used only when PCB is the only analysis to be performed on the sample extract.

10.5 Documentation

10.5.1 Before the analysis sequence is initiated the GC Performance and Repairs logbook must be filled out. It should contain the following information: date, injector temp, oven temp, detector temp, column A flow, column B flow, signal A, signal B, analysts initials, and notes for any necessary repairs.

10.5.2 After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor method, job number, QA number, extraction date, lab prep batch, target batch signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (O.K., dilution, non-inject, etc.).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculation of Sample Amounts (Internal Standard Procedure)

11.3.1 Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.2 Solid Samples

$$\text{Concentration (µg/kg)} = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml

11.3.3 Wipe Samples

$$\text{Concentration (µg/kg)} = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(W)\,(Vi)\,(1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
W	=	Wipe

The 1000 in the denominator represents the number of ul in 1 ml

11.4 Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of target analyte peak
Ais = Area of internal standard peak
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5 Percent Relative Standard Deviation (% RSD):

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6 Percent Difference (% D):

$$\overline{\% \text{ D}} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7 Relative Standard Error (RSE)

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level i

11.8 Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.9 Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted.

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL) The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification: The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.
  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then

removed from the hood and placed on secondary containment in the waste room.

Teris Profile Number: 50016624
Onyx Profile WIP Number: 545240

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method SW8000C: Determinative Chromatographic Separations,", Test Methods for Evaluating Solid Wastes, SW846, Revision 3, March 2003.

15.2. United States Environmental Protection Agency, "Method 8082A, Organochlorine Pesticide by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 1, February 2007.

15.3. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. Eurofins Edison SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C,* most current revision.

15.5. Eurofins Edison SOP No. ED-ORP-018*: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B,* most current revision.

15.6. Eurofins Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546*

15.7. Eurofins Edison SOP No. ED-ORP-019*: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A,* most current revision.

15.8. Eurofins Edison SOP No. ED-ORP-051: *Extraction of Semivolatile Organic Compounds in Water by Microextraction using SW846 Method 3511*

15.9. Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision.

15.10. Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision.

15.11. Eurofins Edison SOP No. ED-ORP-022: *Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*, *SW846 Method 3665A,* most current revision.

15.12. Eurofins Edison SOP No. ED-GEN-022, *Training,* most current revision.

15.13. Eurofins Corporate Work Instruction No. CA-T-WI-003, PCB Minimum Requirements, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Dual Column Approach

18.0. Revision History

Revision 8, dated 06/07/2024

- Updated to include SW846 3511 as aqueous prep method.

Revision 7, dated 21 Oct 2022

- Section 4.5 added which details possible DDT and analogs interference with the last major DDT peak.
- Section 7.2.9: Added System Performance Standard (DDT/Endrin) for use in evaluating potential interference of DDT with last major Aroclor 1254 peak.
- Section 9.2.3.3.1: Added requirement to analyze System Performance standard daily for evaluation of DDT interference with Aroclor 1254 quantitation.

Revision 6 dated 17 Oct 2022

- Updated to Eurofins branding, removed TestAmerica references
- Added Section 8.2 to include requirement to treat samples from chlorinated sources with sodium thiosulfate
- Section 9.2.3.1.4: Section 9.2.4.3: Included details of evaluation of initial calibration for Relative Standard Error (RSE).
- Section 11.7: added formula for calculation of RSE.
- Section 12.2 added: details the annual LLOQ verification requirement.

Revision 5 dated 01 Apr 2020

- Updated to Eurofins branding
- Section 7.2.5: added wipe matrix
- Added Section 11.3.3 – calculation for wipe samples

Revision 4 dated 15 Dec 2016

- Section 7.2.8: clarified text to more accurately describe the PCB Internal Standard Mix.

Revision 3 dated 16 May 2016

- Section 2.1: removed SW846 3550 as a prep method for solids and added it as a prep method for wipe samples.
- Section 2.1.1 and 15.0: removed reference to SW846 Method 3520C (SOP No. ED-ORP-003: *Extraction of Semi-Volatile Organic Compounds in Water by Continuous Liquid-Liquid Extraction, SW846 Method 3520C).*
- Section 2.2: expanded to include reference to internal standard calibration.
- Section 7.1.1: revised to clarify that the primary carrier:make-up gas combo is hydrogen and nitrogen.
- Section 7.2.2: added 1-Bromo-2-nitrobenzene (internal standard) to the list of standards (Restek 32279). Components and concentration added to the table
- Added Section 7.2.8 which describes the preparation of the internal standard solution.
- Section 9.1: added internal standard to 'Sample QC' table
- Section 9.1.2: completely re-written to clarify LCS evaluation and corrective action.
- Section 9.1.4: clarified that surrogates must pass CCV criteria in order to be reportable and must be reported from the same column as target analyte results.
- Added Section 9.1.5 which describes acceptance criteria for internal standards (retention time and response)
- Section 9.2.1.1: added following sentence for clarity and compliance with TestAmerica PCB Minimum Requirements document (WI No. CA-T-WI-003): "All other Aroclors are calibrated using a single point calibration up to 8 peaks at the anticipated midpoint of the calibration range if the 1016/1260 calibration meets the required average RF criteria." Added language detailing which Aroclor standards can be analyzed together.
- Section 9.2.1.1: added this sentence: "The initial calibration block must include at least one level with 1016 analyzed separately for pattern recognition purposes (note: this run does not need to be part of the actual calibration)."
- Section 9.2.1.1: clarified that a minimum of five peaks are required for quantitation of each Aroclor (except for Aroclor 1221 which requires a minimum of three peaks). Added requirement that selected peaks must be at least 25% of height of largest peak in Aroclor (except Aroclor 1268 where the requirement is 10%).
- Section 9.2.2: Revised first sentence to read: "A mid-point Continuing Calibration Verification (CCV) standard must be analyzed after every 20 samples at minimum," (i.e., removed the closing CCV requirement and 12 hour requirement).
- Section 9.2.2: clarified that the CCV typically consists of a 1016/1260 standard. Also added the following sentence: "If the samples being analyzed are being

specifically targeted for a different Aroclor, that Aroclor may be analyzed as the CCV instead of 1016/1260."

- Section 9.2.2: added requirement to analyze a CCV for each detected Aroclor within 12 hours of detection.
- Table 2: updated with the expected RT acceptance criteria from Minimum Requirements document (0.03 minutes).
- Section 9.2.3.2: Revised 'External standard' to 'Internal Standard'. Emphasized that both columns must pass calibration criteria.
- Section 9.2.3.2.2: revised to require that 5 peaks must be selected (was 3) and evaluated for initial calibration (per corporate Minimum Requirements).
- Section 9.3.3.3.1: corrected section reference for preparation of ICV (was Section 7.2.9; now Section 7.2.7);
- Section 9.2.3.3.2: revised first sentence to read: "At least five characteristic peaks of each Aroclor 1016/1260 plus surrogates in the ICV must be checked to verify the Initial Calibration Verification." (it had previously required three peaks).
- Section 9.2.4.3.1: updated text reflect corporate Minimum Requirements document.
- Added Section 9.2.3.4.2: more fully describes CCV acceptance criteria and corrective actions. Updated table with calibration criteria.
- Added Section 9.2.3.4.3: details resolution requirements per the corporate Minimum Requirements document.
- Section 10.2.5: revised to remove 12 hour requirement (replaced solely with 20 sample requirement) and bracketing requirement. Corrected section reference for CCV acceptance criteria.
- Section 11: completely re-written to include internal standard result calculations and other required QC calculations.
- Section 15 (References): added following document reference: TestAmerica Corporate Work Instruction No. CA-T-WI-003, PCB Minimum Requirements, most current revision.
- Attachment 1: revised completely.

- Revision 2, dated 08 Jun 2015

  - Section 1.1: Corrected Method Limit Group (MLG) reference to 8082 (was incorrectly listed as 8081B,
  - Table 2: updated RLs for leachates from 0.0050 mg/L to 0.00050 mg/L.
  - Throughout document: updated the default initial volumes for aqueous preps (250 ml) and leachate (TCLP/SPLP/ASTM) preps (250 ml).
  - Throughout document: removed any notes referencing option for 'reduced volume' extractions since this is now standard as defined in SOP.
  - Section 2.2.2: replaced SW846 3541 (Soxtherm) prep with SW846 3546 (Microwave) prep.
  - Section 6.2.1 (and throughout document): replaced Target software references with references to TestAmerica's Chrom chromatography data processing software.
  - Section 7.2.2 and throughout document: updated source of standards from Supelco to Restek.
  - Section 7.2.3 and throughout document revised concentration of low calibration standard to 50 ppb.
  - Section 7.29: updated ICV prep instructions using Restek standards.

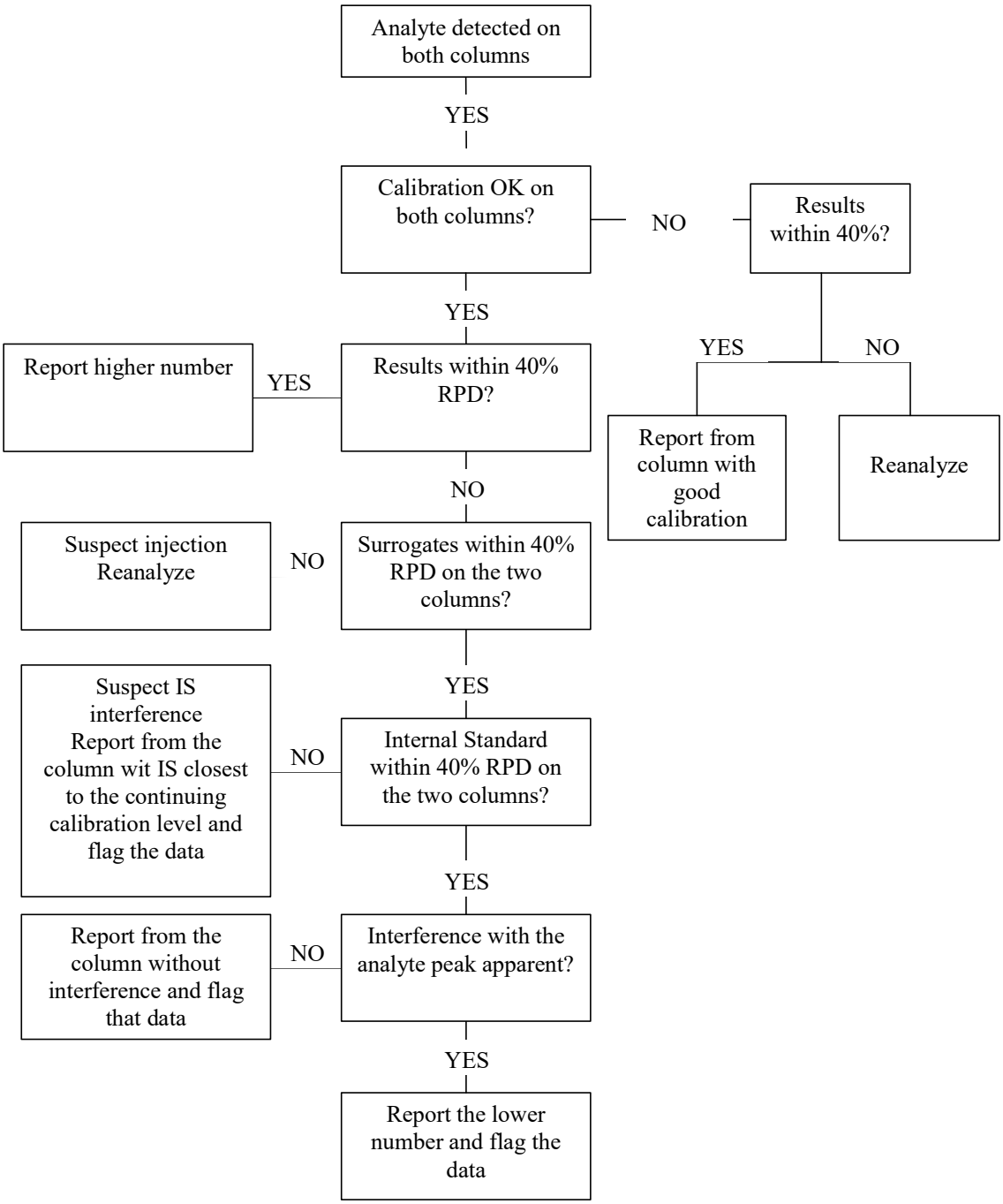
- Section 8: updated sample container from 1000ml to 250ml; updated minimum sample size to 250ml.
- Section 10.3: added note explaining NJDEP DKQP requirement to report the higher concentration in all cases.
- Section 10.3 and Attachment 1: revised to reflect current TestAmerica dual column reporting rules.
- Section 15: removed outdated references.

- Revision1, dated 11 October 2012

  - Throughout document: Revised LQM section references to reflect the most current LQM revision
  - Revised Table 2 to reflect RLs for reduced initial/final volume prep option.
  - Section 2.1.1: added description of reduced initial volume (125ml)/final volume (1ml) extraction option.
  - Section 2.1.2 and Section 15.0: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546.*
  - Section 7.2.3: added procedure for prep and analysis of a lower level Aroclor 1016/1260 standard (50 ppb) and for the surrogate DCB (12.5 ppb) when analyzing samples prepped using the reduced initial/final volume procedure.
  - Section 9.2.3.4.1: Added reference to 50 ppb 1016/1260 ICAL standard for the reduced initial/final volume method.
  - Section 10.2.4: Added 50 ppb 1016/1260 ICAL standard to the sequence for the reduced initial/final volume method

- Revision 0, dated 02/16/2011: NEW

Attachment 1
Dual Column Approach


Analyte detected on both columns
YES
Calibration OK on both columns?
NO
Results within 40%?
YES
NO
Report from column with good calibration
Reanalyze
YES
Report higher number
YES
Results within 40% RPD?
NO
Suspect injection Reanalyze
NO
Surrogates within 40% RPD on the two columns?
YES
Suspect IS interference Report from the column wit IS closest to the continuing calibration level and flag the data
NO
Internal Standard within 40% RPD on the two columns?
YES
Report from the column without interference and flag that data
NO
Interference with the analyte peak apparent?
YES
Report the lower number and flag the data

Title: Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW846 Methods 8270E

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Approvals (Signature/Date):

Sylvanus Klusey Organics Operations Manager	10/18/2022 Date	Dan Helfrich Health & Safety Manager	10/18/2022 Date
Carl Armbruster Quality Assurance Manager	10/18/2022 Date	Mark Acierno Laboratory Director	10/18/2022 Date

Diaa Nimer — 10/18/2022 Date
SVOA GC/MS Manager

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

USEPA Method 8270E is an analytical method which employs the use of GC/MS to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and water samples

Eurofins Edison has the capability to analyze and report the compounds listed in Table 1 via Method 8270E.

Table 1

Compound	CAS No.	Compound	CAS No.
1,1'-Biphenyl	92-52-4	Anthracene **(1)**	120-12-7
1,2,4,5-Tetrachlorobenzene	95-94-3	Atrazine	1912-24-9
1,2,4-Trichlorobenzene	120-82-1	Benzaldehyde	100-52-7
1,2-Dichlorobenzene	95-50-1	Benzidine	92-87-5
1,2-Diphenylhydrazine	122-66-7	Benzo[a]anthracene **(1)**	56-55-3
1,3-Dichlorobenzene	541-73-1	Benzo[a]pyrene **(1)**	50-32-8
1,3-Dimethylnaphthalene	575-41-7	Benzo[b]fluoranthene **(1)**	205-99-2
1,4-Dichlorobenzene	106-46-7	Benzo[g,h,i]perylene **(1)**	191-24-2
1,4-Dichlorobenzene-d4 **(ISTD)**	3855-82-1	Benzo[k]fluoranthene **(1)**	207-08-9
1,4-Dioxane **(1) (2)**	123-91-1	Benzoic acid	65-85-0
1-Methylnaphthalene	90-12-0	Benzyl alcohol	100-51-6
1-Naphthylamine	134-32-7	Bis(2-chloroethoxy)methane	111-91-1
2,2'-oxybis[1-chloropropane]	108-60-1	Bis(2-chloroethyl)ether **(1)**	111-44-4
2,3,4,6-Tetrachlorophenol	58-90-2	Bis(2-ethylhexyl) phthalate	117-81-7
2,3,7,8-TCDD	1746-01-6	Bisphenol-A	80-05-7
2,3-Dihydroindene	496-11-7	Butyl benzyl phthalate	85-68-7
2,3-Dimethylaniline	87-59-2	Caprolactam	105-60-2
2,4,5-Trichlorophenol	95-95-4	Carbamazepine	298-46-4
2,4,5-Trimethylaniline	137-17-7	Carbazole	86-74-8
2,4,6-Tribromophenol **(Surrogate)**	118-79-6	Chrysene **(1)**	218-01-9
2,4,6-Trichlorophenol	88-06-2	Chrysene-d12 **(ISTD)**	1719-03-5
2,4-Dichlorophenol	120-83-2	Coumarin	91-64-5
2,4-Dimethylphenol	105-67-9	Dibenz(a,h)anthracene **(1)**	53-70-3
2,4-Dinitrophenol	51-28-5	Dibenzofuran	132-64-9
2,4-Dinitrotoluene	121-14-2	Diethyl phthalate	84-66-2
2,4-Xylidine	95-68-1	Dimethyl phthalate	131-11-3
2,6-Dinitrotoluene	606-20-2	Di-n-butyl phthalate	84-74-2
2-Chloronaphthalene	91-58-7	Di-n-octyl phthalate	117-84-0
2-Chlorophenol	95-57-8	Fluoranthene **(1)**	206-44-0
2-Ethylaniline	578-54-1	Fluorene **(1)**	86-73-7
2-Fluorobiphenyl **(Surrogate)**	321-60-8	Hexachlorobenzene **(1)**	118-74-1
2-Fluorophenol **(Surrogate)**	367-12-4	Hexachlorobutadiene	87-68-3
2-Methylnaphthalene	91-57-6	Hexachlorocyclopentadiene	77-47-4
2-Methylphenol	95-48-7	Hexachloroethane	67-72-1
2-Naphthylamine	91-59-8	Indeno[1,2,3-cd]pyrene **(1)**	193-39-5
2-Nitroaniline	88-74-4	Isophorone	78-59-1
2-Nitrophenol	88-75-5	n,n'-Dimethylaniline	121-69-7
2-tertbutyl-4-methylphenol	2409-55-4	Naphthalene **(1)**	91-20-3
2-Toluidine	95-53-4	Naphthalene-d8 **(ISTD)**	1146-65-2

Table 1

Compound	CAS No.	Compound	CAS No.
3 & 4 Methylphenol	15831-10-4	n-Decane	124-18-5
3,3'-Dichlorobenzidine	91-94-1	Nitrobenzene	98-95-3
3,4-Dimethylaniline	95-64-7	Nitrobenzene-d5 **(Surrogate)**	4165-60-0
3,5-di-tert-butyl-4-hydroxytol	128-37-0	N-Nitrosodimethylamine **(1)**	62-75-9
3-Nitroaniline	99-09-2	N-Nitrosodi-n-propylamine	621-64-7
4,6-Dinitro-2-methylphenol **(1)**	534-52-1	N-Nitrosodiphenylamine	86-30-6
4-Bromophenyl phenyl ether	101-55-3	n-Octadecane	593-45-3
4-chloro-2-methylaniline	95-69-2	o-Toluidine-d9 **(Surrogate)**	194423-47-7
4-Chloro-3-methylphenol	59-50-7	Pentachloronitrobenzene	82-68-8
4-Chloroaniline	106-47-8	Pentachlorophenol **(1)**	87-86-5
4-Chloroaniline–d4 **(Surrogate)**	191656-33-4	Perylene-d12 **(ISTD)**	1520-96-3
4-Chlorophenyl phenyl ether	7005-72-3	Phenanthrene **(1)**	85-01-8
4-Methylphenol	106-44-5	Phenanthrene-d10 **(ISTD**)	1517-22-2
4-Nitroaniline	100-01-6	Phenol	108-95-2
4-Nitrophenol	100-02-7	Phenol-d5 **(Surrogate)**	4165-62-2
Acenaphthene **(1)**	83-32-9	Phenyl ether	101-84-8
Acenaphthene-d10 **(ISTD)**	15067-26-2	Pyrene **(1)**	129-00-0
Acenaphthylene **(1)**	208-96-8	Pyridine	110-86-1
Acetophenone	98-86-2	Terphenyl-d14 **(Surrogate)**	1718-51-0
Aniline	62-53-3	Total Cresols	STL00160
Aniline-d5 **(Surrogate)**	4165-61-1		

(1) Compound can be analyzed by full scan or Selected Ion Monitoring (SIM).
(2) Compound can also be analyzed by Isotope Dilution/SIM.

1.2 For a listing of method detection limits (MDLs) and Reporting Limits (RLs) please refer to the currently active Method 8270 Method Limit Groups in TALS (Eurofins LIMS).

1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work)*, and Section 19 (*Test Methods and Method Validation)* in Eurofins Edison's Quality Assurance Manual (Eurofins Edison Document No. ED-QA-LQM).

1.4 Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in SOP ED-GEN-003. The NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

2.0 Summary of Method

2.1 This method is used for the analysis of aqueous and solid matrices for semi-volatile base, neutral and acid organic compounds that are extracted from the sample matrix with an organic solvent.

2.2 An aliquot of sample containing surrogate spiking compounds is extracted with an organic solvent. The extract is concentrated on a steam bath to a suitable volume. Internal standards are added to the extract.

2.3 Sample extraction techniques are specified for each matrix in the following Eurofins Edison SOPs:

- ED-ORP-002 (*Extraction of Semivolatile Organic Compounds in Water by Separatory Funnel, SW846 Method 3510C*);
- ED-ORP-043 (*SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270);*
- ED-ORP-0044 (*Microwave Extraction for Solids, SW846 Method 3546*);

2.4 A small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds which were recovered during the extraction step by boiling point. The effluent of the gas chromatograph is interfaced to a mass spectrometer (MS) which is used to detect the compounds eluting from the GC. The detected compounds are fragmented with an electron beam to produce a mass spectrum which is characteristic of the compound introduced into the MS. Identification of target analytes is accomplished by comparing their mass spectra with the electron ionization spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion (quantitation ion) relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Method 8270E as applicable.

2.5 The standard preparation procedure for aqueous samples involves use of a Reduced Volume Extraction (250 ml) (RVE) followed by analysis using a Large Volume Injection (LVI). Optionally, a full volume (1000 ml nominal) may be employed. The details of the extractions are outlined in the applicable prep SOPs while the analytical details for 8270E is presented in this SOP.

2.6 These methods are also applicable to the analysis of samples by Selected Ion Monitoring (SIM) for the purpose of obtaining lower reporting limits for the following compounds:

Table 2 – SIM Analytes

SIM Analytes	CAS #
1,4-Dioxane	123-91-1
4,6-Dinitro-2-methylphenol	534-52-1
Acenaphthene	83-32-9

Table 2 – SIM Analytes	
SIM Analytes	**CAS #**
Acenaphthylene	208-96-8
Anthracene	120-12-7
Benzo[a]anthracene	56-55-3
Benzo[a]pyrene	50-32-8
Benzo[b]fluoranthene	205-99-2
Benzo[g,h,i]perylene	191-24-2
Benzo[k]fluoranthene	207-08-9
Bis(2-chloroethyl)ether	111-44-4
Chrysene	218-01-9
Dibenz(a,h)anthracene	53-70-3
Fluoranthene	206-44-0
Fluorene	86-73-7
Hexachlorobenzene	118-74-1
Indeno[1,2,3-cd]pyrene	193-39-5
Naphthalene	91-20-3
N-Nitrosodimethylamine	62-75-9
Pentachlorophenol	87-86-5
Phenanthrene	85-01-8
Pyrene	129-00-0

2.7 An isotope dilution selected ion monitoring (SIM) technique for the analysis of 1,4-dioxane in water at a reporting level of 0.2 ug/l is also described in this SOP. Using this technique 1,4-dioxane-d8 is added prior to sample extraction and is used as an internal standard to calculate the concentration of 1,4-dioxane present. Additionally, 1,4-dichorobenzene-d4 is added to the extract prior to analysis to monitor the recovery of 1,4-dioxane-d8.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of the Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Analysts must take steps to determine the source of the interference and take corrective action to eliminate the problem.

4.1.1 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce

carryover, the sample syringe is automatically rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of a solvent blank to check for cross-contamination. Alternately, verify that the sample analyzed after the high concentration sample does not show any carryover through inspection of chromatogram and target results.

4.1.2 Contaminants from the extraction process detected in the method blank should be evaluated to determine the impact on the analysis. Interferences from any target analyte must not be present in the method blank above the reporting limit for that compound. If these types of interferences occur, corrective action is required. The source should be identified and corrective action initiated to eliminate the interference from the extraction process. Affected samples must be re-extracted and re-analyzed.

4.1.3 The analyst must take precautions to make sure that contaminants do not enter the analytical system. These precautions include systematic procedures designed to eliminate interferences.

4.2 Some compounds analyzed by this method are unstable or sensitive to extraction and/or instrument conditions:

- Benzidine is easily oxidized during extraction. Neutral extraction may enhance the recovery of this compound.
- Hexachlorocyclopentadiene breaks down photochemically and can decompose from high temperatures, particularly in the injection port of the GC. This compound can also react with acetone in solution.
- 1,2-Diphenylhydrazine is unstable even at room temperature and readily converts to azobenzene.
- Phenols are sensitive to active sites and can give a low response or exhibit poor chromatography by tailing. Therefore, it is important the GC is maintained in the best possible condition. See Section 10.1 for proper daily maintenance.
- N-Nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be distinguished from diphenylamine.
- 3-Methylphenol cannot be separated from 4-methylphenol by the conditions specified in this method. They are reported as 3 and 4-methylphenol.
- Pyridine may perform poorly at the GC injection port temperatures listed in this SOP. Lowering the injection port temperature may reduce the amount of degradation.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Toluene	Flammable Poison Irritant	200 ppm-TWA 300 ppm-Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.
Dimethyl-dichloro-silane	Flammable	none	Can be corrosive to the respiratory tract causing severe irritation and tissue damage. Harmful if absorbed through the skin. May cause severe irritation and systemic damage. Severely irritating to the skin and eyes. Harmful if swallowed. Can cause abdominal discomfort, nausea, vomiting, diarrhea, and irritation to the mouth, throat and stomach.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Gas chromatograph/mass spectrometer system

6.1.1 Gas chromatograph: An Agilent/HP 6890/7890/900 Intuvo (or equivalent) houses the capillary column. The GC provides a splitless injection port and allows the column to be directly coupled to the mass spectrometer. The oven is temperature programmable to meet the requirements of the method. An HP/Agilent 7673/7683/7963 autosampler (or equivalent) with a 10 ul syringe provides automatic injection of sample extracts while the instrument is unattended.

6.1.2 Analytical Column: 30m x 0.25mm ID, 0.25 um film thickness, Restek Rxi-5Sil MS, Catalog #13623

6.1.3 Mass spectrometer: Agilent (HP) 5972, 5973, 5975 or 5977A Mass Selective Detector (MSD) Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts electron energy in the electron ionization mode. The mass spectrometer must be capable of producing a mass spectrum for 50 ng of decafluorotriphenylphosphine (DFTPP) which meets the criteria in Section 9.2.1 when 2 ul of the 25 ug/ml GC/MS tuning standard is injected through the GC.

6.1.4 GC/MS interface: Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.

6.1.5 Data system: The data system is interfaced to the mass spectrometer and accommodates continuous acquisition and storage of GC/MS data throughout the duration of the chromatographic program. The data system consists of a Hewlett-Packard Chemstation equipped with Mustang software used for instrument control and data acquisition. This, in turn, is interfaced to Eurofins's Chrom software for data processing. Data from sample extract analysis can be accessed in real-time, while sample data reports and library searches can be performed on data files from previously run samples. The software is also capable of searching any GC/MS data file for ions of a specific mass whose abundances can be plotted versus time or scan number which allows integration of abundances for any extracted ion between specified times or scan-number limits. Library searches utilize a NIST 02.1 Mass Spectral Library.

6.2 Bottles, glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.

6.3 Injection port liners, splitless

6.4 Injection port septa

6.5 Injection port graphite seals

6.6 Pre-silanized glass wool (Supelco 2-0411 or equivalent)

6.7 Syringes, Assorted sizes 10ul - 1000ul; gas-tight

6.8 Bottles, 10 and 5ml amber screw cap with Teflon liner

6.9 Vials, 2ml amber screw cap with Teflon liner

6.10 Wheaton microvials 100ul (or equivalent)

6.11 Volumetric Flasks, Class A with ground glass stoppers (2ml - 100ml)

6.12 Analytical balance, ASP Model SP-180 (or equivalent), capable of accurately weighing to 0.0001 gr.

7.0 Reagents and Standards

The following items are recommended for performing this procedure. Equivalent items should only be used when they result in an improvement in quality, efficiency, productivity, or cost. An item can be considered equivalent if with its use, the analytical and QA/QC requirements in this SOP can be met. Please refer to the MSDS prior to the use of any reagent or standard.

The preparation of standards, surrogates and spiking solutions is documented in the TALS Reagent Module. Formulary reports can be generated upon request.

7.1. Reagents:

7.1.1. Methylene Chloride: J.T.Baker Resi-Analyzed, used for Organic Residue Analysis (P/N 9266-V8 or equivalent).

7.1.2. Methanol: J.T.Baker Purge and Trap Grade (P/N 9077-02 or equivalent).

7.1.3. Sylon-CT: Supelco (P/N 33065-U or equivalent). Sylon-CT is a highly reactive silanizing reagent consisting of 95% Toluene and 5% Dimethyldichlorosilane (DMDCS).

7.1.4. Each lot of solvent is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2. Standards:

7.2.1. **Calibration Standards (Full Scan Analysis)**: Stock analytical standard solutions are purchased mainly from Restek Corporation. Other standards are prepared in the laboratory as needed using neat compounds or prepared solutions purchased from Agilent, SPEX CertiPrep, Chem Service,

Accustandard, Supelco or other suppliers. Standards prep instructions are detailed for the following full scan analyte list options:

- Full Volume Aqueous Prep; and,
- Reduced Volume Aqueous Prep and Soils

Secondary dilutions are either made from purchased stock solutions as listed below or from prepared solutions as listed in the following table:

NOTE: Second sources (from certified separate lots) are used for ICV standards.

Table 3 – Full Scan Stock Standards			
Target Analyte Standard Name	**Conc. (PPM)**	**Vendor**	**Catalog #**
1,2,3,4-TCDD	50	SPEX	SVO-TANJ-12
Agilent Mix (contains compounds listed in Table 4 below)	2000 *	Agilent	Cus 0456
8270 List 1/ Std #1 Megamix	Varied	Restek	571995
8270 List 1/ Std#9	2000	Restek	569730
8270 List 1/ Std#11	2000	Restek	569732
8270 Surrogate Standard	5000*	Restek	567685
8270 Internal Standard	2000	Restek	567684
8270 List 1/ Std#10	2000	Restek	569731
Bisphenol-A	1000	Agilent	Cus-0457

*Agilent Mix, 8270 list1/std#9 and 8270 Surrogate standard are diluted to 100ppm prior to the preparation of the 1.0ppm and 0.5ppm standards.

Table 4	
Agilent Mix Catalog No. Cus-0456	
Analyte	**Concentration (PPM)**
Pentachloronitrobenzene	2000
2 -tert-butyl-4-Methylphenol	2000
2,6-Di-tert-butyl-4-Methylphenol	2000
Coumarin	2000
Phenyl ether	2000
N,N'-Dimethylaniline	2000
N-Methylaniline	2000
Carbamazepine	2000
Benzonitrile	2000
1,3-Dimethylnaphthalene	2000

7.2.1.1. Individual calibration standards for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 5
Full Volume Aqueous Prep and Soils
Working Standards Preparation

Solution Name	120 PPM	80 PPM	50 PPM	20 PPM	10 PPM	5 PPM	2 PPM	1 PPM	0.5 PPM
8270 List 1/ Std #1 Megamix	3000 ul	2000 ul	2500 ul	500ul	250ul	250ul	100ul	50ul	25ul
8270 List 1/ Std #9	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	50ul	500ul*	250ul *
8270 List 1/ Std #10	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	-	-	-
Agilent custom Mix	1500 ul	1000 ul	1250 ul	250ul	125ul	125ul	50ul	500ul*	250ul *
1,2,3,4-TCDD	-	-	500ul	-	-	-	-	-	-
8270 Surrogate Standard	600ul	400ul	500ul	100ul	50ul	50ul	20ul	500ul*	250ul *
8270 Internal Standard	500ul	500ul	1000 ul	500ul	500ul	1000 ul	1000 ul	1000 ul	1000 ul
Bisphenol-A	3000 ul	2000 ul	2500 ul	500ul	250ul	250ul	100ul	-	-
8270 List 1/ Std #11	400ul	300ul	500ul	200ul	125ul	125ul	50ul	25ul	-
Final Volume (ml)	**25**	**25**	**50**	**25**	**25**	**50**	**50**	**50**	**50**

Note: The 1.0ppm and 0.5pmm standards (above) are prepared using the 100ug/ml standard for Agilent custom Mix, 8270 List1/std#9 and 8270 Surrogate Standard.

Table 6
Reduced Volume Extraction/LVI
Working Standards Preparation

Solution Name	24 PPM	16 PPM	10 PPM	4 PPM	2 PPM	1 PPM	0.4 PPM	0.2 PPM	0.1 PPM
120 ppm (see Table 5)	2.0mL								
80 ppm (see Table 5)		2.0 mL							
50 ppm (see Table 5)			2.0 mL						
20 ppm (see Table 5)				2.0 mL					

Table 6 Reduced Volume Extraction/LVI Working Standards Preparation									
Solution Name	**24 PPM**	**16 PPM**	**10 PPM**	**4 PPM**	**2 PPM**	**1 PPM**	**0.4 PPM**	**0.2 PPM**	**0.1 PPM**
10 ppm (see Table 5)					2.0 mL				
5.0 ppm (see Table 5)						2.0 mL			
2.0 ppm (see Table 5)							2.0mL		
1.0 ppm (see Table 5)								2.0 mL	
0.5 ppm (see Table 5)									2.0mL
Final Volume (ml)	**10**	**10**	**10**	**10**	**10**	**10**	**10**	**10**	**10**

7.2.1.2. **Initial Calibration Verification (full scan)**: Second source ICVs for full scan analysis are prepared in one of several ways depending upon the technique (full volume aqueous prep, soils prep, reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of ICVs for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 7 8270/625 ICV Working Standards Preparation	
Solution Name	**25 PPM**
8270 List 1/ Std #1 Megamix (2nd Lot)	250ul
8270 List 1/ Std #9 (2nd Lot)	125ul
8270 List 1/ Std #10 (2nd Lot)	125ul
Agilent custom Mix (2nd Lot)	125ul
8270 Internal Standard	200ul
8270 List 1/ Std#11	125ul
Bisphenol-A (2nd Lot)	250ul
Final Volume (ml)	**10**

7.2.1.3. **Surrogate Standards (Full Scan Analysis):** A 5000ppm Surrogate Standard is purchased from Restek for use in spiking blanks, samples and associated QC prior to extraction (reference the applicable sample prep SOPs for spiking instructions).

Table 8 Full Scan Surrogate Standards Solution Restek Catalog No. 567685	
Surrogate Standard Compounds	**Concentration (PPM)**
Nitrobenzene-d5	5000
p-Terphenyl-d14	5000
2,4,6-Tribromophenol	5000
Phenol-d5	5000
2-Fluorobiphenyl	5000
2-Fluorophenol	5000

7.2.1.4. **Internal Standards** (**Full Scan Analysis**): The Internal Standards Solution at 2000ppm is purchased from Restek (Catalog # 567684). The Internal Standard solution is stored in 10ml amber screw cap bottles with Teflon liners in the dark at 4°C. The Internal standard solution is used in preparing all analytical standards. Inject 20ul of this solution (2000ppm) per ml of sample extract prior to analysis resulting in a concentration of 40ppm (ug/ml) in the extract.

Table 9 Full Scan Internal Standards Solution Restek Catalog No. 567684	
Internal Standard Compounds	**Concentration (PPM)**
1,4-Dichlorobenzene-d4	2000
Phenanthrene-d10	2000
Naphthalene-d8	2000
Chrysene-d12	2000
Acenaphthene-d10	2000
Perylene-d12	2000

7.2.2. **Calibration Standards (SIM analysis)**: The Edison lab currently analyzes only a select list of compounds by 8270E SIM (see Sections 1.0 and 2.0). Stock analytical SIM standard solutions are purchased mainly from Agilent. Working standards are prepared from these solutions as listed in the tables in Section 7.2.2.1:

Table 10 Stock SIM Standards			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
Pentachlorophenol	1000ppm	AGILENT	PH-180-1
n-Nitrosodimethylamine	100ppm	AGILENT	NS-100-1
Hexachlorobenzene	100ppm*	AGILENT	CH-151-1
PAH Mix	100ppm	AGILENT	PAH-605-1
Bis(2-chloroethyl)ether	100ppm*	AGILENT	BEC-110-1
4,6-Dinitro-2-methylphenol	1000ppm**	AGILENT	PH-150
1,4-Dioxane	1000ppm**	AGILENT	NV-152-1

*Hexachlorobenzene and Bis(2-chloroethyl)ether are diluted to 10ppm prior to SIM Standards prep

** 4,6-Dinitro-2-methylphenol and 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

NOTE: Second sources (from separate lots are used for ICV standards).

7.2.2.1 Individual calibration standards for SIM analysis are prepared in one of two ways depending upon the technique (full volume aqueous prep or reduced volume prep with LVI) as well as the target analyte list. The following tables detail the preparation of calibration standard solutions for each of these techniques and analyte lists. Prepare by combining the indicated volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 11
Full Volume Aqueous Prep – SIM
Working Standards Preparation

	0.025 PPM	0.05 PPM	0.1 PPM	0.5 PPM	1.0 PPM	5.0 PPM
Pentachlorophenol	2.5uL	2.5uL	12.5uL	10uL	20uL	50uL
n-Nitrosodimethylamine	25uL	25uL	125uL	100uL	200uL	500uL
PAH mix	6.25uL	5uL	25uL	50uL	100uL	200uL
Hexachlorobenzene	25uL	25uL	250uL	1000uL	2000uL	500uL*
Bis(2-chloroethyl)ether	25uL	25uL	250uL	1000uL	2000uL	500uL*
4,6-dinitro-2-methylphenol	50ul	50ul	250ul	200ul	400ul	1000ul
1,4-Dioxane	25ul	50ul	250ul	200ul	400ul	1000ul
ISTD	500uL	200uL	500uL	200uL	200uL	200uL
Final Volume (ml)	**25**	**10**	**25**	**10**	**10**	**10**

*For Hexachlorobenzene and Bis(2-chloroethyl)ether the 5.0 ppm level is prepared using the 100ppm standard.

Table 12
Reduced Volume Extraction/LVI – SIM
Working Standards Preparation

	0.005 PPM	0.01 PPM	0.02 PPM	0.10 PPM	0.20 PPM	1.0 PPM
0.025 PPM Std (see Table 11)	1.0 mL					
0.05 PPM Std (see Table 11)		1.0 mL				
0.1 PPM Std (see Table 11)			1.0 mL			
0.5 PPM Std (see Table 11)				1.0 mL		
1.0 PPM Std (see Table 11)					1.0 mL	
5.0 PPM Std (see Table 11)						1.0 mL
Final Volume (ml)	**5**	**5**	**5**	**5**	**5**	**5**

7.2.2.2 Initial Calibration Verification (SIM): A 0.1 ppm separate lot SIM ICV is prepared as detailed in Table 13 using the stock standards detailed in Section 7.2.2 (above)

Table 13 0.1ppm SIM ICV preparation	
Pentachlorophenol	25uL
n-Nitrosodimethylamine	25uL
PAH mix	5uL
Hexachlorobenzene	5uL
1,4-Dioxane	5ul
4,6-Dinitro-2-methylphenol	100ul
ISTD	**100uL**
Final Volume	**5 ml**

7.2.2.3 Internal Standard solution (SIM): A 50 ppm Internal Standard solution for SIM analysis is prepared by adding 125ul of the 2000ppm stock ISTD (see Section 7.2.1.4) and bringing to volume with Methylene Chloride in a 5ml volumetric flask.

7.2.2.3.1 For SIM analysis inject 20ul of this solution (50ppm) per ml of sample extract prior to analysis resulting in a concentration of 1ppm (ug/ml) in the extract.

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7.2.3. Calibration Standards (Isotope Dilution SIM – 1,4-Dioxane): The Edison lab currently analyzes only for 1,4-dioxane by 8270E isotope dilution SIM (see Sections 1.0 and 2.0). Stock analytical isotope dilution SIM standard solutions are purchased mainly from Accustandard and Restek. Working standards are prepared from these solutions as listed in the tables below.

Table 14 - Stock 1,4-Dioxane Isotope Dilution SIM Standards			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dioxane	1000ppm*	Accustandard	APP-9-096

* 1,4-Dioxane is diluted (10x) to 100ppm prior to SIM Standards prep

Table 15 - Stock Labeled 1,4-Dioxane SIM Surrogate/Internal Standard (added at prep)			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dioxane-d8	2000ppm	Restek	30614

Table 16 - Stock 1,4-Dioxane Isotope Dilution SIM Internal Standard (added to extract)			
Standard Name	**Concentration**	**Vendor**	**Catalog #**
1,4-Dichlorobenzene-d4	2000ppm	Accustandard	AZ-014J-3

7.2.3.1 Individual calibration standards for 1,4-dioxane isotope dilution SIM analysis are prepared at the concentrations detailed in the following tables. Prepare by combining the appropriate volumes of each stock solution using volumetric flask. Dilute to the volume marker with methylene chloride.

Table 17 Reduced Volume Extraction/LVI – 1,4-Dioxane Isotope Dilution SIM ICAL Standard Concentrations (ug/ml)									
	Lev 1	**Lev 2**	**Lev 3**	**Lev 4**	**Lev 5**	**Lev 6**	**Lev 7**	**Lev 8**	**ICV***
1,4-Dioxane	0.02	0.04	0.1	0.2	0.5	1	2	10	0.2
1,4-Dioxane-d8	4	4	4	4	4	4	4	4	4
1,4-Dichlorobenzene-d4	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2	0.2

***:** The ICV is prepared from the second source stock in Table 13.

7.2.4. GC/MS Instrument Performance Check (DFTPP): The DFTPP standard is prepared by is prepared at 25 ppm by adding 2.5ml of EPA 8270 GC/MS Tuning Solution II (Restek Catalog # 31615) to a 100ml volumetric flask and bringing to volume with Methylene Chloride.

7.2.5. Information on prepared standard solutions must be recorded in the TALS Reagent Module. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Standards must be remade every 6 months, or sooner, if the standards expire or begin to show signs of unacceptable degradation. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

7.2.6. Please refer to Eurofins Edison SOP No. ED-GEN-008, Standard Operating Procedure for Preparation, Purity and storage of Reagents and Standards.

- Shelf Life of Standard: 1 year after preparation or stock standard manufacture expiration, whichever comes first;
- Storage Requirements: Stock standards are stored at 4°C and Working Standards stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources.

8.2 All samples must be stored at 4ºC (± 2ºC) upon receipt.

8.3 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

8.4 Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 1L	1000 ml or 250 ml (1)	Cool 4 ± 2ºC	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270E
Solids	Wide mouth glass, 8 or 16 oz.	50g	Cool 4 ± 2ºC	14 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270E

(1) : Reduced volume extraction (RVE) LVI option

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standards	Every sample	Response within -50% to +100% of CCV

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.

[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are determined annually and are updated into TALS limit group..

9.1.1. Method blanks are extracted with every sample batch on each day that samples are extracted. To be considered acceptable, the method blank must contain less than the reporting limit of all target compounds except for phthalates, which can be present at up to 5x the MDL. For method 8270E the method blank is generally acceptable if target analyte concentrations are less than the one half the reporting limit.

If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be re-extracted and re-analyzed. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed.

9.1.1.1. Surrogate recoveries for the method blank are compared to laboratory generated limits. If two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference.. If any surrogate is still outside limits, all samples and QC samples associated with that method blank must be re-extracted (volume permitting).

9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD): A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared and extracted concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria. See the current active TALS 8270 Method Limit Group for QC limits. The MS/MSD spiking solution should the same as used for the calibration standards.

9.1.2.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.2.2 An LCS/LCSD may be substituted for the MS/MSD if insufficient sample volume is available.

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (See the current active TALS 8270 Method Limit Group for QC limits). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.3.2 Spike recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.4. Surrogate Standards: All full scan samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.1.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (See the current active TALS 8270 Method Limit Group for QC limits).

If any two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix interference. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary.

9.1.4.1 Surrogate recovery limits are lab generated and are updated annually. Certain state regulatory programs have defined recovery limits which, where applicable, are used for spike recovery evaluations. The TALS Method Limit Groups detail these regulatory program criteria.

9.1.5. Internal Standards: The response (area count) of each internal standard in the sample must be within -50 +100% of its corresponding internal standard in the CCV or, the ICAL midpoint for samples analyzed under the initial calibration range. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

9.2. Instrument QC

9.2.1 GC/MS Instrument Performance Check (DFTPP): (**Note**: the DFTPP performance check applies only to full scan analyses and is not evaluated for SIM analysis). The GC/MS system is tuned using Perfluortributylamine

(PFTBA) such that an injection of 50ng of Decafluorotriphenylphosphine (DFTPP) meet the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all DFTPP key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples. Daily tune verification is not required for 8270E CCV.

DFTPP Key Ions and Abundance Criteria	
Mass	**Ion Abundance Criteria**
51	30-60% of mass 198
68	<2% of mass 69
69	reference only
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base Peak, 100% relative abundance
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	present but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

9.2.1.1. Evaluate DFTPP using three scan averaging and background subtraction techniques. Select the scan at the peak apex, add +1 scan from the apex and -1 scans from the apex.

9.2.1.2. The mass spectrum of DFTPP may be background subtracted to eliminate column bleed or instrument background ions. Background subtract DFTPP by selecting a scan for subtraction ≤20 scans before the apex scan of DFTPP.

9.2.1.3. Check column performance using pentachlorophenol and the benzidine peaks (these compounds are included in the DFTPP solution). Benzidine & Pentachlorophenol should respond normally without significant peak tailing (Tailing Factor should be <2 measured at 10% peak height). If responses are poor and excessive peak tailing is present, corrective action for the GC/MS instrument may be required. Corrective actions may include:

9.2.1.3.1 Retune the GC/MS;
9.2.1.3.2 Clip the injector end of the GC column;
9.2.1.3.3 Replace the septum and injection port liner;
9.2.1.3.4 Change the injection port seal;

9.2.1.3.5 Replace the GC column;
9.2.1.3.6 Clean the injection port with MeCl2
9.2.1.3.7 Clean the MS ion source;
9.2.1.3.8 Place a service call.

9.2.1.4. The breakdown of 4, 4-DDT into 4,4-DDD and 4,4'DDE may also be used to assess GC column performance and injection port inertness. If so evaluated the breakdown must be <20%.

9.2.1.5. DFTPP parameter settings are stored in a tune file, which will be used in all subsequent analysis of standards and sample extracts.

9.2.2 Initial Calibration Range and Initial Calibration Verification

9.2.2.1. Initial Calibration: The initial calibration range consists of a minimum of five concentration levels of analytical standards (six for second order regression) prepared as described in Section 7.2. and analyzed once the DFTPP instrument performance check has met the criteria in Section 9.2.1. .

9.2.2.2. Initial Calibration Verification (ICV): An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2. The ICV must be from a source (or lot) separate from the standards used in the Initial Calibration Range.

9.2.3 Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV): A mid-point Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the DFTPP instrument performance check (when applicable).. The CCV is prepared as detailed in Section 7.2. (typically, 50 ug/ml for full volume aqueous and soils, 10 ug/ml for LV, 0.02 ug/ml for LVI SIM) and 0.2 for isotope dilution SIM). Additionally a Low Level Continuing Calibration Verification (LLCCV) is analyzed after the CCV for full scan analysis. The LLCCV is the same as the lowest calibration level analyzed with the initial calibration range (See Section 7.2).

9.2.4 Calibration Acceptance Summary

9.2.4.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. Obtain the retention time for all compounds from the analysis of the midpoint standard for the calibration curve. Establish the center of the retention time window by using the absolute retention time for each analyte, internal standard and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the

same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. For qualitative identification to be acceptable the retention time of the relative retention time (automatically calculated in Chrom) must be within 0.8 - 1.2 RRT units of its assigned internal standard. The relative retention times of each compound in the five calibration standards must agree within .06 relative retention time units.

9.2.4.2. Initial Calibration Range: Internal standard calibration is employed for this method. After the initial calibration range has been analyzed the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
A_x = Area characteristic ion (see Table 21) for the compound
Ais = Area characteristic ion (see Table 21) of associated internal standard
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

9.2.4.2.1. Determine the mean RRF for each compound. Minimum response factors must be met for each of the compounds listed in Table 18 (below). Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity in the analytical batch to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met.

Table 18:
Minimum Response Factors

Compound	Minimum Response Factor
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

Table 18: Minimum Response Factors	
Compound	**Minimum Response Factor**
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 phthalene	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
4-chlorophenyl-phenyl ether	0.400
Fluorene	0.900
4-Nitroanailine	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 phthalene	0.010

Table 18: Minimum Response Factors	
Compound	**Minimum Response Factor**
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
Pentachloronitrobenzene	0.050

9.2.4.2.2. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

9.2.4.2.3. The % RSD of the RRF's must be ≤20% for each target analyte listed in Table 18. The % RSD of each target analytes must be ≤20% in order for the calibration range to be acceptable. Additionally for 8270E, the calculated concentration or amount of each analyte of interest in the CCV standard should fall within ±20%. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit or do not meet the minimum correlation coefficient (0.99) or Relative Standard Error (≤20%) for alternate curve fits (see below) then appropriate corrective maintenance action must be performed. If more than 10% of the compounds included with the initial calibration exceed the 20% RSD limit **AND** do not meet the minimum correlation coefficient (0.99)) or Relative Standard Error (≤20%) then recalibration is necessary.

9.2.4.2.4. If the above listed criteria is met, the system can be assumed to be linear and sample analysis may begin and the average RF from the initial calibration range is used to quantitate all samples.

9.2.4.2.4.1 Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.2.5. An alternative calibration technique may be employed for those any compounds exceeding the 20% RSD criteria:

9.2.4.2.5.4 Calculate the first order linear regression for any compound which did not meet the 20% criteria. First order linear regression calibration may be employed if alternative average response calibration procedures were not applicable. The r value (Correlation Coefficient) of the equation must be ≥0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.5.2 Second order regression calibration can be used for any compound that has an established history as a non-linear performer.

9.2.4.2.5.3 If second order regression calibration is used a minimum of six (6) calibration levels must be analyzed.

9.2.4.2.5.4 If second order regression calibration is used, the r^2 (Correlation Coefficient) value must be ≥ 0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.5.5 Any compound that fails to meet the 20% RSD or 0.99 correlation coefficient or RSE (≤20%) criteria must be flagged as estimated for detects (or must be noted in the narrative). If there are non-detects the compounds may be reported if there is adequate sensitivity to detect at the quantitation limit. To demonstrate adequate sensitivity analyze the low level point of the initial calibration in each analytical batch (LLCCV) The criteria for demonstrating adequate sensitivity is detection in the LLCCV using the standard qualitative identification criteria.

9.2.4.2.5.6. When calculating the calibration curve using the linear calibration model a minimum quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration back into the curve. The recalculated concentration of the low calibration point should be within ±50% of the

standard's concentration. This evaluation can be checked using the Initial Calibration %Drift Report in Chrom. Any detects for analytes calibrated using the linear model and failing this readback criterion must be flagged as estimated or detailed in the narrative.

9.2.4.3. **Calibration Point Read-back Criteria**: Whichever calibration model above is selected, it should be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount.

- CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD / Linear Response Factor.

- The absolute value of the % Error for each calibration point should be < 30%. For the lowest calibration point, the % Error may be <50%.

- See Section 11.8 for the Calculation of Percent (%) Error.

9.2.4.4. **Initial Calibration Verification (ICV):**.Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 7.2) must be analyzed and evaluated. The ICV must meet the criteria of 70-130% recovery for all compounds with the exception of the poor performing compounds listed in Attachment 1 which are allowed to be within 50-150% : An NCM must be initiated to denote any ICV non-conformances.

9.2.4.5. The ICV must meet the criteria of 70-130% recovery for all compounds however up to 10% of the compounds are allowed to exceed these criteria as long as their recoveries are within 65-135%. For the poor performers (see Attachment 1) the range is 50-150%. If the criterion is not met, a second ICV may be analyzed after corrective measures are taken. If a second ICV analysis fails to meet criteria proceed with corrective action and the analysis of a new initial calibration range. Flagging: If the ICV limits are outside of criteria (high) for an analyte and that analyte is undetected in the sample, no flagging or narration is required. If the ICV limits are outside of criteria (low) for an analyte and that analyte is undetected in a sample, narrate the non-conformance in an NCM. When that out of spec analyte is detected in a sample, describe the issue in the narrative, or flag as estimated.

9.2.4.6. Continuing Calibration Verification (CCV): A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a DFTPP instrument performance check (not required for 8270E), and analysis of a calibration verification standard. **Note:** Certain state regulatory programs have defined calibration acceptance limits which, where applicable, are used for calibration evaluations. The TALS ICAL Limit Groups detail these regulatory program criteria.

9.2.4.5.1 Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.1 using a 50 ng injection of DFTPP. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process. For 8270E analysis only, tune verification is required just prior to ICAL.

9.2.4.5.2 Calibration Verification: Analyze the calibration verification standard immediately after a DFTPP that meets criteria. Daily analysis of the DFTPP is not required as part of the CCV for 8270E analysis. .When samples are analyzed after an ICAL the last ICAL standard may be used as the starting time reference for evaluation. Use the mid point calibration standard (approximately 50ug/l). NOTE: The calibration standard contains internal standards; Dichlorobenzene d_4, Naphthalene d_8, Acenaphthene d_{10}, Phenanthrene d_{10}, Chrysene d_{12}, and Perylene d_{12} at 40ug/l (0.1ug/L for SIM). The calibration check standard must also include all the target analytes from the original calibration.

9.2.4.5.3 The RFs must meet the criteria for the compounds in Table 18. Any compound that fails the minimum response factor must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only but the standard qualitative identification criteria in the method must be met

9.2.4.5.4 The percent difference (when using average response factor) or percent drift (when using linear regression) of the compounds in Table 18 must be ≤20% for at least 80% of the total analyte list. If more than 20% of the compound list fail to 20% difference or drift criterion then appropriate corrective action must be taken prior to the analysis of the samples. Any individual compound that fails must be reported as estimated for detects and must have a demonstration of sensitivity to report non-detects. To demonstrate adequate sensitivity for out of criterion compounds analyze the low level point of the calibration (LLCCV) in the analytical sequence. The criterion for the LLCCV is detection only (%D criteria are not applied) but the standard qualitative identification criteria in the method must be met.

9.2.4.5.5 **CCV Poor Performers**: Refer to Attachment 1 for the identification of poor and/or erratic performing analytes. These analytes are allowed a %D >20% but must be <50 %D to be acceptable. If there are poor performers that exceed 50%D, the data may be reported provided results are noted as estimated. An NCM must be initiated to denote this situation.

9.2.4.5.6 The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.

9.2.4.5.7 The response (area count) of each internal standard in the calibration verification standard must be within 50 - 100% of its corresponding internal standard in the mid-level calibration standard of the active calibration curve. If the EICP area for any internal standard changes by more than a factor of two (-50% +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

9.2.4.5.8 The relative retention times of each compound in the calibration verification standard must agree within .06 relative retention time units of its value in the initial calibration.

9.2.4.5.9 Use the average response factors from the original five-point calibration for quantitative analysis of target analytes identified in field samples.

9.2.4.5.10 Prepare a calibration summary or list indicating which compounds did not meet the 20% average percent difference criteria. Record this information in that run log.

9.2.4.7. Low Level Continuing Calibration Verification (LLCCV): An LLCCV consisting of the low level standard from the initial calibration range is analyzed every 12 hours of instrument operation after the CCV. The purpose and evaluation of the LLCCV is described in Section 9.2.4.4.4.

10.0 Procedure

10.1. Gas Chromatograph/Mass Spectrometer Operation

10.1.1. The sequence of events for GC/MS analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed.

10.1.2. Preparation of the Injection Port Liner and Installation Procedure: Prior to the start of initial calibration and each daily analysis of sample extracts, a new liner for the injection port must be prepared. Once a liner has been used it is no longer inert and will cause serious chromatography problems with phenols and other compounds. When preparing the liner, proper laboratory protection must be worn and the liner must be prepared in a well-ventilated hood. When the procedure is completed all traces of toluene, Sylon-Ct and methanol will be removed immediately so that extraction solvents and preparation of sample extracts will not come into contact with these solvents and become contaminated.

10.1.2.1 Remove one liner from a 40ml VOA bottle containing other liners immersed in Sylon-Ct solution. Rinse off the liner with Toluene and wipe dry. Insert 1cm of pre-silanized glass wool partially into one end of the liner and trim neatly. Push the glass wool into the center of the liner so that it is 1 1/4" from the bottom. Do not use glass wool or solvents that are dirty (i.e. suspended particles) or use liners which are chipped on the ends, deformed or fractured. Inspect the glass wool for cleanliness after it has been inserted.

10.1.2.2 Using a Pasteur pipette flush out the interior of the liner containing the glass wool with Sylon-Ct. Rest the liner horizontally on a small beaker and allow the Sylon-Ct to re-deactivate the interior surfaces and the glass wool. There should be no air bubbles caught in the glass wool. After several

minutes flush out the Sylon-Ct with toluene and finally with methanol. Dry the outer surface of the liner and rest it on the injection port housing until the remaining methanol is boiled off

10.1.2.3 Insert the liner with the newly silanized glass wool plug into the injection port. Verify that the column extends up into the injection port and is perpendicular. Inspect the graphite seal and replace it if the edges are knife-shaped.

10.1.2.4 The septum is always replaced daily. Bake out the column at 300^{O}C for 15 minutes after the vacuum in the analyzer has returned to normal.

10.1.2.5 Performance may enhanced by clipping a small portion of the column at the injection port end. Document this activity in the maintenance record.

10.1.3. Prior to calibration or sample analysis always verify that the analyzer is under sufficient vacuum and that the column has proper carrier gas flow.

10.1.4. Establish the following GC/MS operating conditions:

10.1.4.1 Full Scan Operating Mode

Full Scan Mode – Standard Injection Volume
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300^{O}C
Source Temperature: Preset by H.P. at 280^{O}C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45^{O}C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310^{O}C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275^{O}C
Injector: Grob-type, pulse, splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

Full Scan Mode – Large Volume Injection (LVI)
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300^{O}C
Source Temperature: Preset by H.P. at 280^{O}C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time: 45^{O}C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310^{O}C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275^{O}C
Injector: Grob-type, pulse, splitless
Injection Volume: 5ul
Splitless Valve Time: 0.3 minutes

10.1.4.2 SIM Operating Mode

SIM Mode
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300^{O}C
Source Temperature: Preset by H.P. at 280^{O}C
Scan start time: 1.5 minutes
Initial Column Temperature and Hold Time: 40^{O}C for 0.5 minutes
Column Temperature Program: 20°C /min to 100°C 25°C/min to 270°C 10° C/min to 310°C
Final Column Temperature Hold: 310^{O}C for 3 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275^{O}C
Injector: Grob-type, pulse splitless
Injection Volume: 1ul
Splitless Valve Time: 0.3 minutes

10.1.4.3 Isotope Dilution Selected Ion Monitoring Mode :

SIM Parameters

Group 1
Plot 1 Ion: 74.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	42.0 50	43. 0 50	68.0 50
	74.0 50	128.0 50	129.0 50
	136.0 50	150.0 50	152.0 50
	93.0 50	66.0 50	
	58.0 50		
	88.0 50		

Group 2
Group Start Time: 6.00
Plot 1 Ion: 152.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	151.0 50	152.0 50	153.0 50
	154.0 50	162.0 50	164.0 50
	165.0 50	166.0 50	

Group 3
Group Start Time: 7.80
Plot 1 Ion: 188.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	94.0 50	101.0 50	142.0 50
	178.0 50	179.0 50	188.0 50
	202.0 50	264.0 50	266.0 50
	284.0 50		

Group 4
Group Start Time: 10.50
Plot 1 Ion: 228

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	120.0 50	228.0 50	229.0 50
	240.0 50		

Group 5
Group Start Time: 12.00
Plot 1 Ion: 252.0

Ions/Dwell in Group	(Mass Dwell)	(Mass Dwell)	(Mass Dwell)
	138.0 50	139.0 50	252.0 50
	253.0 50	260.0 50	264.0 50
	267.0 50	276.0 50	278.0 50

Table 19: Target Compound - Primary and Monitoring Ions

Compound	1	2	3

Compound	1	2	3
1,4-Dioxane-d8	96	64	62
1,4-Dioxane	88	58	57
1,4-Dichlorobenzene-d4	152	150	

10.1.5. The above listed instrument conditions are used for all analytical standards for calibration and for all sample extracts analyzed by this method.

10.1.5.1 The column conditions, scan start time, and splitless valve time for analysis of DFTPP only are as follows are as follows:

Initial Column Temperature and Hold Time: 140^{o}C for 0.5 minutes
Column Temperature Program: 140^{o} to 320^{o}C at 22^{o}C/minute
Final Column Temperature Hold: 320C for 0.5 minutes
Scan Start Time: approx. 5 minutes
Splitless Valve Time: 0.3 minutes
Injection Volume: 2 ul

10.2. Analytical Sequence

10.2.1. Dilutions are made based on initial GC/MS analysis. Dilutions are made in 1-ml vials using microsyringes. Calculate the dilution factor using the equation below:

DF= Ph / 5 x Is
Where:
DF = Dilution Factor
Ph = Sample Peak Height
Is = Internal Standard Peak Height

When DF >1 but <2, combine 500ul of sample extract with 500ul methylene chloride in a 1 ml amber vial, add20 ul internal standard and crimp seal

Use **Table 20** to determine dilution and internal standard amount.

Table 20 Dilution Factor Calculations			
DF Value	**Volume of Sample (ul)**	**Volume of Methylene Chloride (ul)**	**Volume of ISTD (ul)**
<1	1,000	None	None
>1, <2	500	500	10
>4, <5	200	800	16
>10, <20	100	900	36
>20	500*	500	10
*Prepare this dilution by serially diluting the >10, <20 dilution			

10.2.2. Instrument Performance and Calibration Sequence

10.2.2.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.2.2.2. Analyze the Instrument Performance Check Standard (DFTPP) as discussed in Section 9.2.1.

10.2.2.3. Initially and as required, analyze the Initial Calibration Range (minimum 5 points, six points for second order regression) as detailed in Sections 7.2.1 and 9.2.4.2. Evaluate the acceptability of the Initial Calibration Range as detailed in Section 9.2.4.2.

10.2.2.4. Immediately after the Initial Calibration Range only, analyze the Initial Calibration Verification (ICV) as detailed in Sections 7.2. and 9.2.4.3. Evaluate the acceptability of the ICV as detailed in Section 9.2.4.3.

10.2.2.5. Every 12 hours, reanalyze and evaluate the Instrument Performance Check Standard (DFTPP), not required for 8270E followed by the Continuing Calibration Verification (CCV) and Low Level Continuing Calibration Verification (LLCCV) as detailed in Section 9.2.3, 9.2.4.4 and 9.2.4.5. Evaluate the acceptability of the CCV and LLCCV as detailed in Section 9.2.4.4

10.2.2.6. Client samples and QC samples are analyzed (as detailed in Section 10.2.3) after acceptable Instrument Performance and Calibration Checks and until the 12 hour clock expires. Repeat the sequence as required. The automation of GC/MS runs is accomplished via the “SEQUENCE” macro of the ChemStation.

10.2.3. Sample Analysis Sequence

10.2.3.1. Sample extracts are normally prepared on the same day as analysis. The GC/MS operator will prepare the extracts that will be run on his or her instrument. Volume adjustments to the extracts will be made at the discretion of the supervisor.

10.2.3.2. Prior to the start of sample analysis the GC/MS operator will generate a sequence program containing the list of the sample extracts to be analyzed, the position on the autosampler tray, and the proper acquisition and tune methods that are to be used. This sequence program contains all the necessary information on the samples to be analyzed and how the GC/MS system is to analyze them. The sample extracts are loaded onto the autosampler (ALS) tray. Their position is verified by checking them against the ALS number on the sequence. This batch analysis will be performed automatically over the 12-hour period.

10.2.3.3. The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

10.3. Data Processing

10.3.1. Prior to processing any standards or samples, target compound lists and sublists must be assembled. Chrom's auto-processing system queries TALS (LIMS) for each sample's processing parameters (including target compounds lists) and downloads the required processing methods from LIMS to analyze data. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

10.3.2. Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.

10.3.3. The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW 8270E are listed in Table 21.

10.4. Interpretation and Qualitative Identification: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The

characteristic ions are 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:

10.4.1 Target Analytes: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are 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:

10.4.1.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.4.1.2. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

10.4.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.4.1.4. The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.

10.4.1.5. All other ions that are greater than 10% in the standard target spectra should also be present in the sample.

10.4.1.6. 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%).

10.4.1.7. If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound will be positively identified and reported with documentation of the identification noted in the raw data record.

10.4.2 Non-Target Analytes: Upon client request a library search to identify non-target Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:

10.4.2.1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.4.2.2. 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%).

10.4.2.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.4.2.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

10.4.2.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.4.2.6. If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid' , etc.).

10.5. Data Reporting

10.5.1. Final Report. The Chom data system automatically produces a data report consisting of hardcopy reports corresponding to specific data reporting requirements, which is uploaded to the TALS LIMS System for the report production group.

10.5.1.1. Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.

10.5.1.2. Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.

10.5.1.3. The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.

10.5.1.4. Data summaries for each method blank indicating which samples were extracted with the indicated blank.

10.5.1.5. A copy of the initial calibration range together with the calibration verification report, and tune report.

10.5.1.6. Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.

10.6. The low-level calibration standard establishes the reporting limit. All reported data must be at a concentration at or above the low concentration standard. Any quantitative values below the report limit must be qualified as estimated.

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11.0. Calculations/Data Reduction

11.1. Target Compounds: are quantitated using the internal standard method (see the formula in Section 11.3).

11.1.1. Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).

11.1.2. The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3. See Section 9.2.4 for discussion of RRF.

11.1.3. Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.

11.2. Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method (see formula in Section 11.3). For quantiation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 11.1) with the following revisions:

11.2.1. The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).

11.2.2. The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).

11.2.3. A RF on 1.0 is assumed.

11.2.4. The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.

11.3. Internal Standard Calculation:

11.3.1. Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.2. Solid Samples

$$\text{Concentration } (\mu g/KG) = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

11.4. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area characteristic ion for the compound (see Table 21)
Ais = Area characteristic ion of associated internal std (See Table 21)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5. Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.4 (Initial calibration):

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6. Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

$$\% D = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.7. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.8. Calculation of Relative Standard Error (RSE)

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level

11.9. Calculation of Percent (%) Error

$$\%Error = \frac{x_i - x_i'}{x_i} x\,100$$

Where:

x_i ' = Measured amount of analyte at calibration level *i*, in mass or concentration units

x_i = True amount

11.10. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. NOTE: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) study, as described in the Eurofins corporate Detection and Quantitation Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change affecting sensitivity is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Lower Limit of Quantitation Verification

The lowest calibration standard analyzed establishes the LLOQ or Reporting Limit. The capability to reliably detect this concentration through the preparation, clean-

up and analytical procedure is verified through the annual analysis of a standard at the LLOQ/RL. The LLOQ verification shall also be performed whenever significant changes are made to the preparation and/or analytical procedure.

12.3.1 The LLOQ verification standard shall be prepared at a concentration 0.5-2 times the LLOQ/RL, and be taken through all of the same preparation and clean-up methods as client samples.

12.3.2 The LLOQ verification standard for aqueous matrix shall be prepared using laboratory deionized water and for the solid matrix using clean Ottawa sand. Other clean matrices may be used in addition, for project specific requirements.

12.3.3 The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3.4 Recovery of each analyte must meet the laboratory established LCS recovery limits + 20%. (For example, if the LCS recovery limits are 70-130%, the LLOQ verification must meet recovery limits of 50-150%.) Once sufficient points have been generated, LLOQ based statistical limits may be used in place of limits based on LCS recovery.

NOTE: The lower recovery limit for the LLOQ can be no lower than 10%.

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, (*Training*), for the laboratory's training program.

13.0. Pollution Control

13.1 It is Eurofins's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

14.1. Pollution Prevention

14.2.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

14.2.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method SW8270D, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Laboratory Manual, Physical/Chemical Methods, Revision 5, July 2014..

15.2. United States Environmental Protection Agency, “Method SW8270E, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Update IV, Laboratory Manual, Physical/Chemical Methods, Revision 6, June 2018.

15.3. United States Environmental Protection Agency, "Method SW8000D: Determinative Chromatographic Separations", Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Update V, Revision 4, October 2012..

15.4. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.5. Eurofins Edison SOP No. ED-ORP-002, *SW846 Method 3510C-Extraction of Semi-Volatile Organic Compounds in Water by Separatory Funnel,* current revision.

15.6. Eurofins Edison SOP No. ED-ORP-043, *SW846 Method 3580A - Waste Dilution Prep for Analysis of BNAs by SW846 Method 8270,* current revision.

15.7. Eurofins Edison SOP No. ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW3546, current revision.*

15.8. Eurofins Document No. CW-E-M-001, Corporate Environmental Health and *Safety Manual,* current revision.

15.9. Eurofins Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.10. Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), current revision.

15.11. Eurofins Edison SOP No. ED-GCS-001, *Preparation and Screening of Semivolatile Organic Extracts for GC/MS Analysis*, current revision.

15.12. Eurofins Edison Work Instruction Document No. EDS-WI-012, *Client Complaint/Corrective Action Form,* current revision.

15.13. Eurofins Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision.

15.14. Eurofins Edison SOP No. ED-ORP-001, *Extraction of Semivolatile Organic Compounds in Water, EPA Method 625.1,* current revision.

15.15. Eurofins Edison SOP No. ED-GEN-022, *Training,* current revision.

15.16. Eurofins Corporate Quality Memorandum, CA-Q-QM-002, *GC/MS Tuning Policy*, current revision.

15.17. Eurofins Corporate Quality SOP No. CA-Q-S-006, *Detection and Quantitation Limits,* current revision.

16.0. Method Modifications:

Method 8270E requires the DFTPP tune standard to be analyzed once prior to an ICAL

and not daily prior to sample analysis. Until such time as 8270D is removed from lab capabilities and in order to satisfy both 8270D and 8270E The laboratory will analyze the DFTPP tune daily, prior to QC and sample analysis. The laboratory will use the tighter criteria from Methods 8270C/8270D for tune evaluation, rather than the criteria suggested in Table 3 of Method 8270E.

17.0. Attachments

Attachment 1 Poor Performing Analytes

18.0. Revision History

- Revision 10, date 10/18/2022
    - Updated to Eurofins branding throughout.
    - Section 8.1: updated to include procedure for sampling chlorinated water sources.
    - Section 9 updated throughout to include ICAL evaluation with Relative Standard Error (RSE).
    - Section 11.8: added formula for calculation of Relative Standard Error (RSE)

- Revision 9, date 03/15/2021
    - Updated as needed to reflect 1,4-dioxane RL of 0.2 ug/l.
    - Updated Tables 11 and 12 to reflect new low ICAL standard concentration of 1,4-dioxane..

- Revision 8, date 06/29/2020
    - Updated to Eurofins branding.
    - Updated throughout to include 8270E requirements.
    - Removed references to SW846 3550B/C prep methods (no longer in use for this method at Edison lab).
    - Update equipment listed in Section 6.0.Updated analytical column in Section 6.1.2.
    - Updated, deleted and renumbered tables as required.
    - Made extensive updates to Standards (sources and preparation) in Section 7.2.
    - Removed all references to Aromatic Amines. Deleted all tables specific to Aromatic Amine analysis. Renumbered remaining tables in document and updated text references.
    - Throughout document clarified tune requirements for 8270E.
    - Following added to Section 9.1.1: For method 8270E the method blank is generally acceptable if target analyte concentrations are less than the one half the reporting limit.
    - Calibration Point Read-back Criteria was added to Section 9.2.4.3. The calculation for percent error was added to Section 11.8.
    - Section 9.2.4.2.3: added following for 8270E: the calculated concentration or amount of each analyte of interest in the CCV standard should fall within ±20%.
    - Section 9.2.4.2.5.6: added ‘The recalculated concentration of the low calibration point should be within ±50% of the standard’s concentration.”

- Section 12.1 revised to reflect the updated MDL procedure.
- Added Section 12.3: annual Lower Limit of Quantitation Verification
- Added Corporate SOP CA-Q-S-006, Detection and Quantitation Limits to references.
- Section 16.0: added a Method Modification regarding tuning check requirements.

- Revision 7, date 06/08/2018

  - Section 2.3: revised to clarify that RVE/LVI is lab standard procedure.
  - Section 9.1.3: removed statement regarding allowance for up to five analytes to recover outside of lab acceptance limits in LCS/LCSD.
  - Section 9.2.4.3: Replace table 'ICV Poor Performers (50-150% Recovery) with expanded list of 'Poor Performing Analytes' in Attachment 1.
  - Added Section 9.2.4.4.5: CCV Poor Performers
  - Corrected number in section 9.2.4.5
  - Added Attachment 1 – Poor Performing Analytes

- Revision 6, date 01/12/2018:

  - Section 7.2.5 included to specify reagent and standard storage conditions.
  - Revised Section 9.1.3 to clarify requirements for specific LCS/LCSD evaluation criteria regarding the # of out of criteria analytes.
  - Revised Section 9.2.4.3 to add 2,4-Dimethylphenol as a poor performing analyte, increased the range for the poor performers to 50-150 and also expanded the guidelines for flagging the ICV outliers.

- Revision 5, dated 09/29/2017:
  - Revised Section 9.1.1 to clarify requirements for surrogate recovery in method blanks.

- Revision 4, dated 08/21/2017:
  - Updated throughout to add a procedure for the analysis of 1,4-dioxane by isotope dilution selected ion monitoring (SIM)
  - Added tables for isotope dilution SIM standards. Renumbered all tables as necessary.
  - Section 7.2.1: added a list of full scan calibration list options.
  - Table 3: Renamed 'Full Scan Stock Standards'.
  - Section 9.2.1: noted that DFTTP applies only to full scan analysis.
  - Section 9.2.3: updated CCV concentrations
  - Added reference to GC/MS Tuning Policy in Section 15.16.

- Revision 3, dated 01/07/2016:
  - Tables 1 and 2: added SIM as option for 1,4-Dioxane.
  - Section 2.3: removed SW3541 (Soxtherm) as option for soils prep (lab has discontinued use of this method). Also removed SW3541 SOP reference from Section 15.0.
  - Tables 19 and 20: added source and prep instructions for 1,4-Dioxane SIM standard. Updated source and prep instructions for 4,6-Dinitro-2-methylphenol.
  - Table 22: added prep instructions for 1,4-Dioxane and 4,6-Dinitro-2-methylphenol

SIM ICV standard.
- Corrected the information in the 'DFTPP Key Ions and Abundance Criteria' table in Section 9.2.1 to match the info found in SW846 8270C.
- Section 10.1.4.2: updated "SIM Parameters" to included ion masses/dwell times for 1,4-Dioxane.

- Revision 2, dated 01/28/2015:
  - Extensively reformatted the SOP. Placed tables that had been in rear of document into the body of the text. Renumbered tables as applicable and fixed text references to tables.
  - Section 1.1, Table 1: Revised table to include all current analytes. Also footnoted those compounds which are currently analyzed by SIM.
  - Section 2.3: added options for extraction of solids by SW846 3456 (Microwave Extraction) and by SW3580A (Waste Dilution) and added SOP references. Deleted reference to SOP ED-ORP-005 (SW3550B – Low Level); Updated Section 15 (References).
  - Section 2.5: added text detailing the RVE/LVI options.
  - Section 2.6: added table which includes all analytes routinely analyzed by SIM.
  - Section 6: updated to include newer GC, MS and autosampler models currently in use.
  - Section 6.1.3: added Zebron ZB column as an option.
  - Section 7.2: extensively revised standards information to reflect switch to Restek standards.
  - Table 3:Added Custom Aromatic Amine Surrogate Standard and revised Table 8 to include initial calibration prep instructions for the Aromatic Amine surrogates.
  - Throughout document: removed references to Target and replaced with Chrom.
  - Section 7.2.1: Added reference to section 10.2.1.2 for LVI.
  - Added Section 7.2.1.3.1 and Table 17A both of which discuss use of Aromatic Amine surrogates.
  - Section 7.2.1.2: Added reference to Tables 9,10 and 11 (ICV Preparation)
  - Section 8.0: Added Sample container and minimum sample size (250 ml) for Reduced volume extraction.
  - Sections 9.1.2, 9.1.3, 9.1.4 and 9.2.4: added statement that certain state regulatory programs have defined recovery limits which, where applicable, are used for spike and calibration evaluations.
  - Section 9.1.2: Deleted sentence "A minimum of 16 spiked analytes are reported to in client reports (the full list is reported at least once during each 2 year period because we employ full spiking list.
  - Section 9.1.4: Added note regarding use of Aromatic Amine Surrogates.
  - Section 9.2.2.2: Added reference to ICV Preparation tables in Section 7.2.
  - Section 9.2.3: added more specific info as to the concentration of the CCVs for all techniques.
  - Section 9.2.4.2.1: Changed to reflect that each analyte should meet minimum RF's, not the average across the calibration. Added LLCCV requirement.
  - Section 10.3.1: added explanation of Chrom's interaction with TALS. Removed references to Target.
  - Section 9.2.4.2.5.5: Added: (or can be noted in the narrative)
  - Section 9.2.4.2.5.6: Revised last sentence to read: "This evaluation can be checked using the Initial Calibration %Drift Report in Chrom."
  - Section 9.2.4.3: Removed 65-135% criteria and added "poor performing" analyte list and associated criteria of 60-140%.

- Section 9.2.4.4.3: Added LLCCV criterion for RFs
- Section 9.2.4.4.4: Added LLCCV criterion for %D
- Section 10.1.4: Updated GC/MS operating conditions for full scan, SIM and DFTPP.
- Section 10.1.4.1: added a table detailing operating conditions for LVI option.
- Table 2: Added 2-ethylaniline, 2,4-dimethylaniline, 3,4-dimethylaniline, 2,3-dimethylaniline, 2,4,5-trimethylaniline and 4-chloro-o-toluidine to Working Standards preparation information.
- Table 25: updated to include all current analytis/surrogates/internal standards and associated ions.
- Throughout document: updated LQM section references as appropriate as some have changed with the latest LQM revision.

- Revision 1, dated 11/07/2011

  - Section 1.1, Table 1: Added Pentachloronitrobenzene and associated CAS# to the analyte list.
  - Section 7.2.1: Added Pentachloronitrobenzene standard information.
  - Table 2: Added Pentachloronitrobenzene to Working Standards preparation information.
  - Table 4: Added Pentachloronitrobenzene and associated minimum RF.
  - Table 8: Added Pentachloronitrobenzene and associated ions.

- Revision 0, dated 02/22/2011: NEW

Table 21
Characteristic Ions Of Semi-Volatile Organic Compounds

Compound	Primary Ion	Secondary Ion(s)
1,1'-Biphenyl	154	153,76
1,2,4,5-Tetrachlorobenzene	216	214, 179
1,2,4-Trichlorobenzene	180	182, 145
1,2-Dichlorobenzene	146	148, 111
1,2-Diphenylhydrazine	77	105, 182
1,3-Dichlorobenzene	146	148, 111
1,3-Dimethylnaphthalene	156	141, 115
1,4-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene d4 **(ISTD)**	152	150, 115
1,4-Dioxane	88	58, 43
1-Methylnaphthalene	142	141, 115
1-Naphthylamine	143	115, 116
2,2'-oxybis[1-chloropropane]	45	77, 121
2,3,4,6-Tetrachlorophenol	232	131, 230
2,3,7,8-TCDD (screen)	320	322, 324
2,3-Dihydroindene		
2,3-Dimethylaniline	106	129

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
2,4,5-Trichlorophenol	196	198, 200
2,4,5-Trimethylaniline	102	55, 56
2,4,6-Tribromophenol **(Surrogate)**	330	132, 141
2,4,6-Trichlorophenol	196	198, 200
2,4-Dichlorophenol	162	164, 98
2,4-Xylidine	121	120, 106
2,4-Dimethylphenol	122	107, 121
2,4-Dinitrophenol	184	63, 154
2,4-Dinitrotoluene	165	63, 89
2,6-Dinitrotoluene	165	63, 89
2-Chloronaphthalene	162	127, 164
2-Chlorophenol	128	64, 130
2-Ethylaniline	106	122,104
2-Fluorobiphenyl **(Surrogate)**	172	171
2-Fluorophenol **(Surrogate)**	112	64
2-Methylnaphthalene	142	141
2-Methylphenol	108	107
2-Naphthylamine	143	115, 116
2-Nitroaniline	65	108, 138
2-Nitrophenol	139	109, 65
2-tert-butyl-4-Methylphenol	149	121, 91
2-Toluidine	107	106, 77
3,3'-Dichlorobenzidine	252	254, 126
3,4-Dimethylaniline	106	129, 127
3,5-Di-tert-butyl-4-Hydroxytol	205	220, 145
3-Nitroaniline	138	108, 65
4,6-Dinitro-2-methylphenol	198	51, 105
4-Bromophenyl phenyl ether	248	250, 141
4-chloro-2-methylaniline	106	144, 142
4-Chloro-3-methylphenol	107	144, 142
4-Chloroaniline	127	129
4-Chloroaniline-d4 **(Surrogate)**	131	133
4-Chlorophenyl phenyl ether	204	206, 141
4-Methylphenol	108	107
4-Nitroaniline	138	108, 65
4-Nitrophenol	139	109, 65
Acenaphthene	154	153, 152
Acenaphthene d10 **(ISTD)**	164	162, 160
Acenaphthylene	152	151, 153
Acetophenone	105	77, 51
Aniline	93	66
Aniline-d5 (**Surrogate**)	98	71,42
Anthracene	178	176, 179
Atrazine	200	173,215
Benzaldehyde	77	105,106
Benzidine	184	92, 185

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
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	122	105, 77
Benzyl Alcohol	108	79, 77
Bis(2-chloroethoxy)methane	93	95, 123
Bis(2-chloroethyl)ether	93	63, 95
Bis(2-ethylhexyl)phthalate	149	167, 279
Bisphenol-A	213	228, 119
Butyl benzyl phthalate	149	91, 206
Caprolactam	113	55,56
Carbamazepine	193	236, 135
Carbazole	167	166, 139
Chrysene	228	226, 229
Chrysene d12 **(ISTD)**	240	120, 136
Coumarin	146	118, 63
Dibenz(a,h)anthracene	278	139, 279
Dibenzofuran	168	139
Diethylphthalate	149	177, 150
Dimethylphthalate	163	194, 164
Di-n-butylphthalate	149	150, 104
Di-n-octylphthalate	149	167, 43
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
Kepone	272	237, 355
N,N-Dimethylaniline	120	122, 104
Naphthalene	128	129, 127
Naphthalene d8 **(ISTD)**	136	68
n-decane	43	57
Nitrobenzene	77	123, 65
Nitrobenzene-d5 **(Surrogate)**	82	128, 54
N-Nitrosodimethylamine	42	74, 44
N-Nitroso-di-n-propylamine	170	42,101,130
N-Nitrosodiphenylamine	169	168, 167
n-Octadecane	57	43, 85
o-Toluidine-d9 **(Surrogate)**	114	112, 42
Pentachloronitrobenzene	237	214,295
Pentachlorophenol	266	264, 268

Table 21 Characteristic Ions Of Semi-Volatile Organic Compounds		
Compound	**Primary Ion**	**Secondary Ion(s)**
Perylene d12 **(ISTD)**	264	260, 265
Phenanthrene	178	179, 176
Phenanthrene d10 **(ISTD)**	188	94, 80
Phenol	94	65, 66
Phenol-d5 **(Surrogate)**	99	42, 71
Phenyl ether	170	77, 115
Pyrene	202	200, 203
Pyridine	79	52, 51
Terphenyl-d14 **(Surrogate)**	244	122, 212

Attachment 1
Poor Performing Compounds

1,2,4,5-Tetrachlorobenzene
1,4-Dioxane
1-Naphthylamine
2,3,4,6-Tetrachlorophenol
2,4-Dimethylphenol
2,4-Dinitrophenol
2-Chloroaniline
2-Naphthylamine
3&4-Methylphenol
3'3-Dichlorobenzidine
4,6-Dinitro-2-methyl- phenol
4-Chloroaniline
4-Nitrophenol
Aniline
Atrazine
Benzaldehyde
Benzidine
Benzoic Acid
Benzyl Alcohol
Biphenyl
Caprolactam
Diphenylamine
Hexachlorocyclopentadiene
Hexachloroethane
n-Decane
n-Nitrosodimethylamine
o,o,o-Triethylphosphorothioate
o-Toluidine
Pentachloronitrobenzene
Pentachlorophenol
Phenol
Pyridine

These analytes are exempt from the ICV and CCV criteria as detailed in this SOP

Title: Closed System Purge and Trap and Extraction for Volatile Organics in Soil , SW846 Method 5035A

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This method describes a closed system purge and trap procedure for the analysis of volatile organic compounds in soils, sediments and solid wastes.

1.1.2 This method is designed for use on samples containing low levels of volatile compounds (0.5 to 200ug/kg), and requires the use of hermetically sealed sample vials, which limits a samples exposure to the atmosphere, thereby minimizing the loss of volatile compounds.

1.1.3 Method 5035A can be used for most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Water-soluble compounds can be included in this technique, but will have higher quantitation limits due to poor purging efficiency.

1.1.4 Method 5035A also includes sample collection and preparation procedures for medium and high concentration soil samples (i.e., >200 ug/kg). Medium and high level soil samples are prepared either by field preservation of a soil sample in methanol or by creating a methanol extract from a soil sample that was collected as bulk soil.

1.1.5 The preparation steps described for medium and high level samples will be followed by the aqueous purge and trap procedure detailed in Eurofins Edison SOP No. ED-MSV-001(*Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*).

1.1.6 The preparation and sample introduction steps described for low-level soils will be followed by analysis using Eurofins Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current revision*) or ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*) as applicable.

1.1.7 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 **Low Level Soils:** Volatile organic compounds are determined by collecting a 5g-soil sample with a specially designed sampling device (ex., En Core® Sampler) in which the sample is stored without headspace. Upon receipt and within 48 hours of sampling, the soil is placed in a pre-weighed vial containing 5 mL of reagent water and a stir bar. The vial containing the soil is then frozen extending the hold time to 14 days. The same samples may be received from the field in a 40 mL vial with 5 mL water (ex., Terra Core® Sampler). Immediately prior to analysis, the soil is thawed, reagent water, surrogate spiking solution and/or internal standard

spiking solution are added to the vial by the automatic sampler, without opening the vial to the atmosphere. The sample vial is heated and purged with helium while being magnetically stirred. The volatile components travel through a transfer line to a sorbent trap. When purging is complete, the trap is heated and backflushed with helium to desorb the sample components on to a GC column for separation and analysis by the appropriate determinative method.

2.2 **Medium Level Soils:** Medium level soils are soils that were originally intended to be analyzed by a low level soil method, but show contaminants >200ug/kg when screened. A methanol extract is prepared by extracting 5g of soil with 10mL of methanol. The volatiles are effectively transferred from the soil to the methanol. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

2.2.1 Samples may also arrive from the field as 5 mL or 10 mL methanol preserved samples. These are considered to be medium level prepared samples.

2.3 **High Level Soils:** The high level soil method acts as a combined preservation and extraction technique. 25mL of methanol /surrogate solution is placed in each VOA vial and weighed prior to field sampling. Approximately 10g of soil is added to each vial in the field. Upon returning to the lab, the vial is weighed again, and the difference between the two weights is recorded as the initial weight of sample. As with the medium level method, the volatiles are effectively transferred to the methanol. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

2.4 **Waste Dilution:** Organic waste samples are prepared by extracting 1g of sample with 10mL of methanol / surrogate solution. A portion of the methanol extract is introduced into the GC or GC/MS system by using the purge and trap Method SW846 5030 followed by the appropriate determinative method.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method and the determinative methods that follow are susceptible to contamination from a number of sources. Potential sources of contamination include organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatile laboratory is located in a separate building, away from the organic extraction area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatile laboratory.

4.3 The nitrogen used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 A trip blank prepared from organic-free reagent water is carried through the sampling, storage, and analysis of each group of samples to check for such contamination.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Purge vessels are removed from the autosampler units after each use, rinsed, baked, returned to the units and pre-purged before the next use.

4.7 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.8 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

4.9 Potential cross-contamination of samples stored in lab refrigerators is monitored by preparation, analysis and evaluation of storage blanks. Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After one week, the storage blanks are removed and analyzed. Additional details can be found in Eurofins Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

5.1.1 Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.1.2 Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required. A purge and trap unit consists of three parts: the sample purge unit, the trap, and the concentrator. Unit configurations currently in use are:

- OI Analytical 4551 Automatic Sampler/4560 concentrator;
- Archon 5100A Automatic sampler/ OI Analytical 4560 concentrator;
- EST Centurion Autosampler/ EST Encon concentrator;
- Archon Autosampler/EST Encon concentrator.

6.1.2 A VOCARB 3000 trap from Supelco is used in the Encon concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001

6.1.3 An OI analytical purge trap #10 is used for the OI 4560 concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed to contain the following absorbents: Tenax/silica gel/carbon molecular sieve.

6.1.4 Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 Both the Encon and OI concentrators are capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.6 Gas chromatograph: HP 5890/Agilent 6890/7890 equipped with temperature programming capability.

6.1.7 GC column: reference the applicable determinative method for column specifics.

6.1.8 Detector: Flame Ionization Detector or Mass Spectrometer (HP5971/5972/Agilent 5973). Reference the applicable determinative method for detector specifics.

6.1.9 Data system: HP Chemstation II for data acquisition and HP UNIX based TARGET software for data processing.

6.2 Equipment and Supplies

6.2.1 Freezer: Capable of holding a temperature of -7°C to -20°C.

6.2.2 Refrigerator: Capable of holding a temperature of 4°C ±2°C

6.2.3 Top loading analytical balance.

6.2.4 Portable field balance. Capable of weighing to 0.01g.

6.2.5 Microsyringes: 10 uL to 1000 uL.

6.2.6 Syringes: 5mL, 10mL, and 30mL gas-tight.

6.2.7 Volumetric flasks: Class “A” glassware, 10mL , 50mL , and 100 mL.

6.2.8 VOA vials: 40 mL glass with PTFE –faced septum.

6.2.9 En Core® Sampler. Designed to take a 5g-soil sample. Sealed to prevent loss of volatiles.

6.2.10 Vials: 2mL amber glass with screw cap with Teflon-faced septa.

6.2.11 Spatula: Narrow, stainless steel.

6.2.12 Stir bars: PTFE coated, small enough to spin freely inside a VOA vial.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Organic free reagent water. Distilled water purchased from Poland Spring.

7.1.2 Methanol. Purge and trap grade, purchased from JT Baker. (Cat # 9077-02)

7.1.2.1 Each lot of methanol and hydrochloric acid is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2 Standards

7.2.1 Calibration standard preparation and use is described in detail in each of the determinative methods.

7.2.2 Method 8260 Methanol/Surrogate solution (2.5 ppm) is prepared using purge and trap grade methanol and neat a,a,a-Triflurotoluene solution. Initially a primary solution is prepared at 2500 ppm:

Standard Name	Vendor	Catalog #	Volume added	Concentration of Stock Std.	Concentration of Standard	Total Volume Volume of MeOH
1°Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4	Sigma Aldrich	 B67201 151998 396540	 1585 ul 2678 ul 1932 ul	Neat	2500ppm	1000 ml

The 2500 ppm solution is used to prepare a 2.5 ppm Method 8260 Methanol/Surrogate working solution by diluting 1.0 ml of 2500 ppm stock to 1000ml with purge and trap grade methanol.

7.2.3 Method 8015 (GRO) Methanol/Surrogate solution (1.25 ppm) is prepared by diluting 0.5ml of the 2500 ppm stock (see Section 7.1.4) to 1000 ml with purge and trap grade methanol.

8.0 Sample Collection, Preservation, Shipment and Storage

8.1 Low Level Soils

Sample containers used to collect low-level soils are 40 mL VOA vials with PTFE-faced septa (closed system purge and trap), En Core® Samplers, Terra Core® kits or bulk soil.

8.1.1 For the 40 mL VOA vial (closed system purge and trap) collection procedure, add 1 clean magnetic stir bar and 5 mL of reagent water to each vial.

8.1.1.1. Cap the vial and weigh to the nearest 0.01g. Record this as the tare weight.

8.1.1.2. Collect approximately 5g of soil using an appropriate sampling device and add 5g to each of 3 vials for low level analysis.

8.1.1.3. Collect a fourth aliquot for field methanol preservation into a pre-weighed vial containing 10 mL of methanol.

8.1.1.4. Closed system purge and trap collection vials are re-weighed to determine actual sample weight. Record the sample weight on the sample vial.

8.1.1.5. Within 48 hours of sampling the collection vials are placed into a freezer (on their side to prevent breakage) at -7°C to -20°C. Freezing in this manner extends the holding time to 14 days from sampling.

8.1.2 For En Core® Sampler collection, collect up to 3 En Core® Sampler for low level preservation via freezing and a fourth for medium level preservation with methanol (Section 8.2 details the medium level preservation procedures for En Core® samplers). The low level preservation procedures are:

8.1.2.1. The En Core® device is received at the lab sealed without headspace with a cap and viton o-rings.

8.1.2.2. For each En Core® requiring low level preservation, add 5ml of reagent water and a magnetic stir bar to a 40 ml vial. Cap the vial.

8.1.2.3. Weigh each vial to the nearest 0.01g. Record this as the tare weight.

8.1.2.4. Transfer the contents of each En Core® to separate preweighed vials within 48 hours of sampling.

8.1.2.5. Reweigh the vial. Record the weight on each sample vial.

8.1.2.6. The vials are placed into a freezer (on their side to prevent breakage) at -7°C to -20°C. Freezing in this manner extends the holding time to 14 days from sampling.

8.1.2.7. A bulk soil sample must also be collected for the purposes of percent moisture analysis for dry weight determination.

8.1.2.8. Vials are removed from the freezer the day of analysis for thawing.

8.1.2.9. Stir bars may be reused after the vial is analyzed. Retrieve the stir bar from each vial using the magnetic stir bar retriever, rinse the stir bar with methanol and bake at 105°C for 2 hours.

8.1.3 Terra Core® Kits. This option requires samples to be field preserved:

8.1.3.1. The sample kit typically includes two pre-weighed 40 mL vials containing 5 mL of water with a small magnet stir bar and one or two pre-weighed 40 mL vials containing pre-measured and recorded amount of methanol.

8.1.3.2. Approximately 5g of sample is placed into each vial by the field sampler.

8.1.3.3. The vials containing the samples are re-weighed in the lab and the net sample weight is recorded for each vial. The vials containing the sample and water are frozen up until the time of analysis.

8.1.3.4. The methanol preserved samples are considered to be medium level prepared samples and do not require freezing but they must be refrigerated at 4°C until time of analysis.

8.1.3.5. A bulk soil sample must also be collected for the purposes of percent moisture analysis for dry weight determination.

8.1.3.6. Stir bars may be reused after the vial is analyzed. Retrieve the stir bar from each vial using the magnetic stir bar retriever, rinse the stir bar with methanol and bake at 105°C for 2 hours.

8.1.4 Bulk soil. Typically consists of soil collected in 8-oz or 16-oz glass jars. At client request bulk soil samples can be sub-sampled, preserved and analyzed for volatile organics. Soils collected for low level VOA analysis as bulk soil in jars will be preserved as described above in Section 8.1.1. Documentation of preservation is recorded in the Soil Preservation Logbook.

8.2 Medium Level Soils

8.2.1 Medium level refers to any soil sample extracted in methanol not preserved in the field. The fourth Encore sample collected (as described in Section 8.1.2 above) will be prepared as a medium level soil in case the analysis of a low level preserved aliquot exhibits concentrations of target compounds exceeding 200 ug/kg. All medium level extracts must be refrigerated at 4°C until time of analysis.

8.2.1.1. Prepare the medium level soil by adding 5 g of soil or Encore to 10 mL of Methanol / Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5. depending upon the method to be analyzed). All medium level extracts shall be prepared within 48 hours of collection. The weight of soil used to prepare the extract is recorded on the sample vial.

8.2.1.2. Since methanol extract results tend to increase with time as the sample contact time increases, a minimum contact time of one day is allowed ***or*** the soil is sonicated for 20 minutes.

8.2.1.3. Any soil requiring medium level preservation but collected out of compliance with method 5035A (bulk soil without preservative or not in an Encore device) will be prepared in the same manner as in section 8.2.1.1. Documentation will be noted in the soil preservation log.

8.3 High Level Soils

8.3.1 High level soils are field collected using laboratory prepared 40 ml VOA vials (with PTFE-faced septa) containing 25 mL of the Methanol/Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5. depending upon the method to be analyzed).

8.3.2 The vials containing the Methanol/Surrogate solution are weighed using a barcode reader and an analytical balance. The bottle ID and weight (Weight 1) are recorded in the methanol-tracking program.

8.3.3 The containers are issued to the field as required.

8.3.4 Field samplers are directed to weigh 10 g of sample using a field balance. They must carefully add the 10 g sample to the sample vial containing the Methanol/Surrogate solution, taking care not to spill any of the solution from the vial.

8.3.5 Field samplers are directed to wipe the neck of the vial carefully to remove any soil around the threads. This helps to ensure a good seal once the vial is capped. Field samplers are further directed to cap the vial, shake gently, dispersing the soil into the methanol. An additional aliquot of soil without any methanol or preservative must be collected for use in determining percent moisture (i.e., for dry weight determination).

8.3.6 Upon the return to the laboratory, the sample vials (now containing both the Methanol/Surrogate solution and approximately 10g of soil) are reweighed using the barcode reader and analytical balance. Record this weight (Weight 2) in the methanol tracking system. Calculate the initial weight of soil by subtracting Weight 1 from Weight 2.

8.3.7 All samples for volatile analysis are protected from light and stored at 4°C±2°C in an area free of organic solvents. No standards or solvents are stored in the sample refrigerators or in the surrounding area.

8.3.8 All samples preserved in this manner must be analyzed within 14 days of collection.

9.0 Quality Control

9.1 Specific quality control procedures are outlined in each of the determinative methods that follow this purge and trap technique of sample introduction. Standard quality assurance practices are used with all methods.

9.2 Reagent water blanks are analyzed to ensure that reagents and/or sample dilutions are free of interferences.

9.3 Method blanks are prepared and analyzed in the same manner as samples, and are carried through the entire analytical procedure to show that each system is in control.

9.4 Potential cross-contamination of samples stored in lab refrigerators is monitored by preparation, analysis and evaluation of storage blanks. Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After one week, the storage blanks are removed and analyzed. Additional details can be found in Eurofins Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

9.5 An initial demonstration of accuracy and precision is performed for each determinative method. This demonstration is done for each sample introduction technique.

10.0 Procedure

10.1 This section provides guidance for the analysis of low level soils using a closed system purge and trap procedure. Reference the SOPs for the determinative methods for additional details as applicable: Eurofins Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current revision*) or ED-MSV-014 (*SW846 Method 8260D Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*).

10.2 Instrument operating parameters are set at the beginning of a method of analysis and remain constant throughout the entire analytical procedure. Again, reference the SOP for the applicable determinative method for specifics. Sample operating conditions are listed here:

Example Instrument Operating Parameters

Purge and trap unit	
Purge Time	11 minutes
Dry Purge	1 Minutes
Purge Gas	Helium
Purge Flow	40-45 ml/min
Purge Temp	Water - Ambient; Solids - 40^{0}C
Trapping Temp	Ambient, <30^{0}C
Desorb Time	1-2 Minutes
Desorb Temp	Vocarb - 260^{0}C
Gas chromatograph	
Injector	180^{0}C
Carrier Gas	Helium
Carrier Flow	6 ml/min
Oven Program	35 - 250^{0}C
Run Time	30 Minutes
Mass Spectrometer	
Electron Energy	70 volts (nominal)
Mass range	35-260 AMU
Scan time	0.9 sec./scan
Source Temp	200^{0}C
Separator Temp	180^{0}C

10.3 All samples are screened prior to analysis, using the procedure outlined in Eurofins Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID SW846 Method 5021(modified),* current revision. In addition to reducing excessive instrument contamination and/or extensive instrument clean up, screen data provides the analyst with an approximate dilution factor and is used to determine which sample preparation technique should be used for a particular sample. If the estimated concentration from the screening procedure shows concentrations below 200 ug/kg, follow the low concentration closed system purge and trap method. If the screen result shows concentrations >200 ug/kg, follow the medium level procedure.

10.4 Low Level Soil Procedure

10.4.1 Low level soils are field collected as described in Section 8.1 using 40 mL VOA vials with PTFE-faced septa (closed system purge and trap), En Core® Samplers, Terra Core® kits or bulk soil.

10.4.2 Set up the purge and trap autosampler (Archon 5100A or EST Centurion, see Section 6.1) for the closed system method for low-level soils. The purge and trap autosampler will add 5 mL reagent water, and 1uL of the internal standard and/or surrogate spiking solution to the sample vials. The heating block will heat each sample to 40° C and hold for 0.5 minutes before purging begins.

10.4.3 Tuning, calibration, and method blank analysis:

- Tuning and calibration is accomplished as detailed in the SOP for the applicable determinative method.

- The volume of water used when purging a calibration standard must be the same as the volume of water purged during a sample analysis. Since the low-level soils have 5 mL water added at vial preparation plus the 5 mL of reagent water added by the purge and trap autosampler prior to purging, a total volume of 10 mL is required for calibration standards.

- Internal standard (if required by the determinative method) is added to each calibration standard by the purge and trap autosampler, in the same manner it will be added to the samples.

- After successful calibration, analyze a method blank. The method blank must meet all criteria in the determinative method. A successful method blank shows that the system is free of interferences and sample analysis may begin.

10.5 Medium Level Soil Procedure

10.5.1 Medium level soils are typically field collected using laboratory prepared 40 ml VOA vials (with PTFE-faced septa) containing 25 mL of the Methanol/Surrogate solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed).

10.5.2 When preparing a medium level soil from a bulk soil sample, do not decant any supernatant liquids. Quickly mix the contents of the sample container with a narrow spatula. Weigh out 5g of soil into a 40ml vial, add 10ml of Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) and cap the container and proceed with Section 10.5.6.

10.5.3 For samples collected in the Encore sampling device, break open one end and push the aliquot of soil out into a vial. Add 10ml of the Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) and cap the vial and proceed with Section 10.5.6.

10.5.4 For Method 8260 samples collected as Terra Cores® in 5 mL or 10 mL methanol add 5 uL and 10 uL of the 2500 ppm 8260 Methanol/Surrogate mix (see Section 7.1.4) respectively to extract and proceed with Section 10.5.6.

10.5.5 For Method 8015 (GRO) samples collected as Terra Cores® in 5 mL or 10 mL methanol add 2.5 uL and 5 uL of the Methanol/Surrogate Mix surrogate solution (see Section 7.1.5) respectively and proceed with Section 10.5.6.

10.5.6 Cap and shake the extract for 2 minutes. Allow soil to settle.

10.5.7 Transfer 2 mL of the extract to a 2 mL amber glass vial for storage with minimal headspace.

10.5.8 Refrigerate at 4°C until the time of analysis.

10.5.9 Analyze a portion of this extract using Method 5030 purge and trap for aqueous samples (Eurofins Edison SOP No. ED-MSV-001, *Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*). The appropriate dilution factor is calculated from the low-level analysis.

10.6 High Level Soil Procedure

10.6.1 High level soils have been preserved in the field by sampling either 10 g of soil into vials containing 25 mL Methanol/Surrogate mix solution (either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed) or 5 g of soil into vials containing 10 mL Methanol/Surrogate mix solution (again, either the 8260 solution as described in Section 7.1.4 ***or*** the 8015 solution as described in Section 7.1.5 depending upon the method to be analyzed).

10.6.2 Screen high level soils by Method 5021 to determine the proper dilution factor prior to analysis (see Eurofins Edison SOP No. ED-GCV-001, *Screening for Volatile Organics,Static Headspace with GC FID SW846 Method 5021(modified),* current revision.

10.6.3 Transfer 2 ml of the extract to a 2 mL amber glass vial for storage with minimal headspace.

10.6.4 Analyze a portion of this extract using Method 5030 purge and trap for aqueous samples.

10.7 Sample Analysis

10.7.1 Remove sample vials from storage and allow thawing to ambient temperature before analysis. Shake the vial gently to ensure that the contents move freely within the vial. Place the sample vials in the autosampler tray.

10.7.2 Schedule the autosamplers to run the samples using the low level soil method. 5 mL of reagent water plus 1 uL of internal standard/ surrogate spiking solution is added to the sample vial directly through the septum.

10.7.3 Prior to purging, the sample is heated to 40°C for 0.5 minutes.

10.7.4 Purge the sample with helium for 11 minutes while agitating with magnetic stirring.

10.7.5 The purgeable compounds will flow out of the vial through a glass lined transfer line to a sorbent trap.

10.7.6 Desorb the contents of the trap on to the GC column by rapidly heating the trap to 260 °C and backflushing it with helium.

10.7.7 Begin the GC temperature program and data acquisition.

10.7.8 Re-condition the trap after desorb by baking at 260° C for 8-12 minutes.

10.7.9 Perform quantitative and qualitative analysis using TARGET software. Evaluate data according to each determinative method.

10.7.10 If a re-analysis is required, perform the re-analysis using the second frozen sample vial.

10.7.11 If a dilution is required, analyze the methanol preserved aliquot by Method 5030.

10.7.12 Technical acceptance criteria for sample analysis. (Note: these are general guidelines; please see the SOP for the determinative method for more specific acceptance criteria)

- The samples must be analyzed on a GC or GC/MS system meeting the initial calibration, continuing calibration and blank technical acceptance criteria.
- The sample must be analyzed within the required holding time.
- The sample must have an associated method blank meeting the blank technical acceptance criteria.
- The percent recovery of each of the system monitoring compounds in the sample must be within the acceptance windows.
- After analyzing a sample that exceeds the initial calibration range the analyst must either analyze an instrument blank (using the same purge inlet if using an auto sampler) which must meet technical acceptance criteria for blank analysis or monitor the sample analyzed immediately after the contaminated sample for all compounds that were in the contaminated sample that exceeded the calibration range.

10.7.13 Corrective Action for Sample Analysis (Note: these are general guidelines; please see the SOP for the determinative method for more specific corrective actions):

- Samples must meet technical acceptance criteria before reporting data.

- Corrective action for failure to meet instrument performance checks, initial, continuing calibration and method blanks must be completed prior to sample analysis.

- Corrective action for system monitoring compounds and internal standard compounds that fail to meet acceptance criteria must be completed prior to sample analysis.

- If any of the system monitoring compounds and internal standard compounds fail to meet acceptance criteria, check all calculations, instrument logs, the system monitoring compound and internal standard compound spiking solutions and the instrument operation. If the calculations were incorrect, correct calculations and verify that the system monitoring compound recoveries and internal standard compound responses meet acceptance criteria.

- Check the preparation of the internal standards and system monitoring compounds for concentration and expiration.

- Verify that the instrument is operating correctly.

- Data that fails to meet minimum acceptance criteria will be annotated (flagged) with qualifiers and/or appropriate narrative comments defining the nature of the outage. If applicable, a Corrective Action Reports will be initiated in order to provide for investigation and follow-up.

11.0 Calculations / Data Reduction

11.1 Methanol volume correction for soil moisture content (medium/high level methods):

$$Vt\ (\mu L\ solvent/water) = [\ ml\ of\ solvent + \frac{(\%moisture \times g\ of\ sample)}{100}] \times 1000\ uL/mL$$

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed.

12.2 Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3 Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, *Training* (latest revision) for the laboratory's training program.

13.0 Pollution Control

13.1 It is Eurofins's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out.

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system**.**

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These

boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

Teris Profile Number (incineration): 50016710
Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

- Returned Methanol Preservative - Methanol preserved vials are collected in satellite accumulation and then transferred to 55 gallon open top steel waste drums in the waste room. These waste drums are then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, *Method 5035A: Closed System Purge and Trap and Extraction for Volatile Organics in Soil and Waste Samples*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 0, December 1996.

15.2 Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3 Eurofins Edison SOP No. ED-GCV-001, *Screening for Volatile Organics,Static Headspace with GC FID SW846 Method 5021(modified),* current revision.

15.4 Eurofins Edison SOP No. ED-MSV-001, *Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*.

15.5 Eurofins Edison SOP Nos. ED-GCV-008, *Gasoline Range Organics using GC FID Method SW846 8015D, current revision.*

15.6 Eurofins Edison SOP No ED-MSV-014, *SW846 Method 8260D Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision.*

15.7 United States Environmental Protection Agency, *Method 8000C: Determinative Chromatographic Determinations*, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, March 2003

15.8 Eurofins Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

15.9 Eurofins Corporate Environmental Health and Safety Manual (CW-E-M-001)

15.10 Eurofins SOP No. ED-GEN-022, *Training*, latest revision.

15.11 Eurofins Edison SOPs Nos. ED-SPM-007, *Disposal of Samples and Associated Laboratory Waste, current revision.*

15.12 Eurofins Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures,* current revision.

16.0 Method Modifications:

16.1 N/A

17.0 Attachments

17.1 N/A

18.0 Revision History

- Revision 11, effective date: 08/01/2024
  - Throughout document: updated to Eurofins branding.
  - Updated references to reflect current revisions
  - Updated list of signatories to reflect recent organizational change.

- Revision 10, effective date: 4/11/2019
  - Section 8.2.1: Added temperature preservation requirement for Medium level extracts.

- Revision 9, effective date: 11/27/2012
  - Updated referenced Lab Quality Manual section and appendix numbers as appropriate

- Revision 8, effective date: 12/6/2010
  - Section 3: revised to reference new location for definitions.
  - Sections 8.2.1.4 and 8.3.9 referencing methanol solvent adjustment for solid samples with significant moisture content (>10%) was removed.

- Revision 7, effective date: 11/4/2008:
  - Revised SOP format in accordance with Eurofins Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Updated the SOP to comply with SW846 Method 5035A (was 5035). Retitled SOP accordingly.

- Section 1.1.5: Added reference to Eurofins Edison SOP No. ED-MSV-001(*Purge and Trap for Aqueous Samples, SW846 Method 5030, current revision*).
- Section 1.1.6: Added references to Eurofins Edison SOP Nos. ED-GCV-006 (*Gasoline Range Organics using GC FID Method SW846 8015, current revision*) and ED-MSV-005 (*SW846 Method 8260B Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry, current revision*)
- Section 1.1.7: Added reference to Quality Assurance Manual for method modifications.
- Section 3: revised to reference new location for definitions.
- Section 4: revised to include a reference to storage blanks and the applicable SOP: Eurofins Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination*, current revision.
- Section 5 (Safety): Revised to include most up to date corporate health and safety references and information.
- Section 6: Updated with current instrumentation and configurations.
- Section 7.0: added details of the solvent testing and approval program.
- Section 7.2: Updated standards sources and catalog numbers.
- Section 8: Updated with additional details for each type of preservation/analysis (low level, medium level, high level). Added used of Terra Core® Sampling Kits (Section 8.1.3).
- Section 8: Updated to include requirement to adjust the methanol volume (medium/high level) for soil moisture content.
- Section 9.0: Added details of storage blanks and reference to Eurofins Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision..
- Section 10: Revised and expanded to include instrument operating conditions and specific sample prep details for low level, medium and high level preps.
- Section 11: Deleted reference to SOP for Organic Calculations. Added statement that calculations can now be found in the applicable determinative methods.
- Section12.0: added reference to Training SOP.
- Section 13.0: Revised to include most up to date company policy on Pollution Control as well as to include corporate health and safety references and information.
- Section 14.0: Revised to include most up to date company policy on Waste Management as well as to include corporate health and safety references and information.
- References: Expanded to include more specific SOP references.
- Revision history: updated.

Title: Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) by SW846 Method 8260D

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Approvals (Signature/Date):

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Carl Armbruster Quality Assurance Manager	08/08/23 Date	Mark Acierno Laboratory Director	08/08/23

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 USEPA SW846 Method 8260D is used for the determination of volatile organic compounds in a variety of aqueous and solid matrices by purge and trap gas chromatography (GC)/mass spectrometery (MS). The methods are applicable to the compounds listed in Table 1 (below). Actual target compound lists are determined through regulatory or project specifications. Method performance criteria for each target analyte will be determined prior to sample analysis.

1.1.2 This SOP also describes the optional procedure for analyses of compounds using 8260D Selected Ion Monitoring (SIM). SIM analyses is specific to target compounds: 1,2-dibromoethane, 1,2-dibromo-3-chloropropane, 1,2,3-Trichloropropane and 1,4-Dioxane. Benzene and Chloroform if needed.

Table 1: Method Analytes

Compound	CAS #	Compound	CAS #
1,1,1,2-Tetrachloroethane	630-20-6	cis-1,2-Dichloroethene	156-59-2
1,1,1-Trichloroethane	71-55-6	cis-1,3-Dichloropropene	10061-01-5
1,1,1-Trifluoro-2,2-dichloroethane	306-83-2	Cyclohexane	110-82-7
1,1,2,2-Tetrachloroethane	79-34-5	Cyclopentene	142-29-0
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	Dibromomethane	74-95-3
1,1,2-Trichloroethane	79-00-5	Dichlorobromomethane	75-27-4
1,1-Dichloroethane	75-34-3	Dichlorodifluoromethane	75-71-8
1,1-Dichloroethene	75-35-4	Dichlorofluoromethane	75-43-4
1,1-Dichloropropene	563-58-6	Dimethylnaphthalene (total)	28804-88-8
1,1-Difluoroethane	75-37-6	Epichlorohydrin	106-89-8
1,2,3-Trichlorobenzene	87-61-6	Ethanol	64-17-5
1,2,3-Trichloropropane **(1)**	96-18-4	Ethyl acetate	141-78-6
1,2,3-Trimethylbenzene	526-73-8	Ethyl acrylate	140-88-5
1,2,4,5-Tetramethylbenzene	95-93-2	Ethyl ether	60-29-7
1,2,4-Trichlorobenzene	120-82-1	Ethyl methacrylate	97-63-2
1,2,4-Trimethylbenzene	95-63-6	Ethylbenzene	100-41-4
1,2-Dibromo-3-Chloropropane **(1)**	96-12-8	Ethylene Dibromide **(1)**	106-93-4
1,2-Dichloro-1,1,2,2-tetrafluoroethane	76-14-2	Hexachlorobutadiene	87-68-3
1,2-Dichloro-1,1,2-trifluoroethane	354-23-4	Hexane	110-54-3
1,2-Dichlorobenzene	95-50-1	Indan	496-11-7
1,2-Dichloroethane	107-06-2	Iodomethane	74-88-4
1,2-Dichloroethene, Total	540-59-0	Isobutyl alcohol	78-83-1
1,2-Dichloropropane	78-87-5	Isopropyl acetate	108-21-4
1,3,5-Trichlorobenzene	108-70-3	Isopropyl alcohol	67-63-0
1,3,5-Trimethylbenzene	108-67-8	Isopropyl ether	108-20-3
1,3-Dichlorobenzene	541-73-1	Isopropylbenzene	98-82-8
1,3-Dichloropropane	142-28-9	Methacrylonitrile	126-98-7
1,3-Dichloropropene, Total	542-75-6	Methyl acetate	79-20-9

Compound	CAS #	Compound	CAS #
1,4-Dichlorobenzene	106-46-7	Methyl acrylate	96-33-3
1,4-Dioxane **(1)**	123-91-1	Methyl methacrylate	80-62-6
1-Chloropropane	540-54-5	Methyl tert-butyl ether	1634-04-4
2,2,4-Trimethylpentane	540-84-1	Methylcyclohexane	108-87-2
2,2-Dichloropropane	594-20-7	Methylene Chloride	75-09-2
2,4,4-Trimethyl-1-pentene	107-39-1	Methylnaphthalene (total)	1321-94-4
2-Butanone (MEK)	78-93-3	Monochloropentafluoroethane	76-15-3
2-Chloro-1,3-butadiene	126-99-8	m-Xylene & p-Xylene	179601-23-1
2-Chloroethyl vinyl ether	110-75-8	Naphthalene	91-20-3
2-Chloropropane	75-29-6	n-Butanol	71-36-3
2-Chlorotoluene	95-49-8	n-Butyl acetate	123-86-4
2-Hexanone	591-78-6	n-Butyl acrylate	141-32-2
2-Methyl-1,3-butadiene	78-79-5	n-Butylbenzene	104-51-8
2-Methyl-2-propanol	75-65-0	n-Heptane	142-82-5
2-Nitropropane	79-46-9	n-Propyl acetate	109-60-4
2-Octanol	123-96-6	N-Propylbenzene	103-65-1
2-Octanone	111-13-7	o-Xylene	95-47-6
4-Chlorotoluene	106-43-4	p-Diethylbenzene	105-05-5
4-Ethyltoluene	622-96-8	Pentane	109-66-0
4-Isopropyltoluene	99-87-6	Propene	115-07-1
4-Methyl-2-pentanone (MIBK)	108-10-1	Propionitrile	107-12-0
Acetaldehyde	75-07-0	sec-Butylbenzene	135-98-8
Acetone	67-64-1	Styrene	100-42-5
Acetonitrile	75-05-8	Tert-amyl methyl ether	994-05-8
Acrolein	107-02-8	Tert-butyl ethyl ether	637-92-3
Acrylonitrile	107-13-1	tert-Butylbenzene	98-06-6
Allyl chloride	107-05-1	Tetrachloroethene	127-18-4
Amyl acetate (mixed isomers)	628-63-7	Tetrahydrofuran	109-99-9
Benzene **(1)**	71-43-2	Toluene	108-88-3
Benzyl chloride	100-44-7	Total BTEX	STL00431
Bromobenzene	108-86-1	trans-1,2-Dichloroethene	156-60-5
Bromoform	75-25-2	trans-1,3-Dichloropropene	10061-02-6
Bromomethane	74-83-9	trans-1,4-Dichloro-2-butene	110-57-6
Butadiene	106-99-0	Trichloroethene	79-01-6
Butyl Methacrylate	97-88-1	Trichlorofluoromethane	75-69-4
Camphene	79-92-5	Vinyl acetate	108-05-4
Camphor	76-22-2	Vinyl chloride	75-01-4
Carbon disulfide	75-15-0	Xylenes, Total	1330-20-7
Carbon tetrachloride	56-23-5	1,4-Dichlorobenzene-d4 **(ISTD)**	3855-82-1
Chlorobenzene	108-90-7	1,4-Dioxane-d8 **(ISTD)**	17647-74-4
Chlorobromomethane	74-97-5	2-Butanone-d5 **(ISTD)**	24313-50-6
Chlorodibromomethane	124-48-1	Chlorobenzene-d5 **(ISTD)**	3114-55-4
Chlorodifluoromethane	75-45-6	Fluorobenzene **(ISTD)**	462-06-6
Chloroethane	75-00-3	TBA-d9 **(ISTD)**	25725-11-5
Chloroform **(1)**	67-66-3	1,2-Dichloroethane-d4 **(Surrogate)**	17060-07-0
Chloromethane	74-87-3	4-Bromofluorobenzene **(Surrogate)**	460-00-4

Compound	CAS #	Compound	CAS #
Chlorotrifluoroethene	79-38-9	Dibromofluoromethane **(Surrogate)**	1868-53-7
Chlorotrifluoromethane	75-72-9	Toluene-d8 **(Surrogate)**	2037-26-5
2-ethylhexyl acrylate	103-11-7		

(1) Compound can be analyzed by full scan or Selected Ion Monitoring (SIM).

1.1.3 Method 8260D can be used to quantitate most volatile organic compounds that have boiling points below 200°C, and that are insoluble or slightly soluble in water. Water-soluble compounds can be included in this method, but quantitation limits will be higher due to poor purging efficiency.

1.1.4 The standard reporting limit (RL) is established at or above the low-level standard in the calibration curve (1 ug/l for most compounds). For a complete list of method detection limits (MDLs) and RLs, please see reference the current TALS (LIMS) active Method Limit Group database.

1.1.5 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (*Review of Work Request*) and 20 (*Test Methods and Method Validation*) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

1.1.6 Any variation in procedure shall be completely documented using an NCM. The NCM is approved by the supervisor and then automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA department also receives NCMs by e-mail for tracking and trending purposes. The NCM process is described in more detail in Eurofins Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision NCM shall be filed in the project file and addressed in the case narrative. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

2.0 Summary of Method

2.1 Method 8260D is used to determine volatile organic compounds in aqueous, non-aqueous and solid matrices. Sample preparation techniques vary, depending on the matrix and the level of contamination expected. Purge and trap techniques are used to introduce the sample to the GC/MS system. Refer to Eurofins Edison SOP Nos. ED-MSV-001*, Purge and Trap for Aqueous Samples, SW846 Method 5030*, current revision and ED-MSV-002, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, SW846 Method 5035A,* current revision.

2.2 All soil samples are screened by GC/FID static headspace or MS analysis to provide the analyst with appropriate initial dilution factors. Aqueous samples without historical results are also screened. For additional details see Eurofins Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

2.3 An aliquot of sample containing internal standard and surrogate spiking solution is purged with nitrogen in a closed sparging vessel. The volatile compounds are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent column where the volatiles are trapped. After purging is complete, the sorbent column is heated and backflushed with helium to desorb the volatiles onto a gas chromatograph column.

2.4 Analytes eluted from the capillary chromatograhy 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 spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a minimum of a five-point calibration curve.

2.5 For aqueous VOA samples submitted for New Jersey Groundwater Quality Standard (NJ GWQS) evaluation, a full scan analysis is initially performed using the 8260 methodology No further analysis by SIM is required if all of the following compounds are present above the full scan RL: 1,2-dibromoethane, 1,2-dibromo-3-chloropropane, 1,2,3-Trichloropropane and 1,4-dioxane, chloroform, vinyl chloride and benzene. If any of these compounds are undetected in the undiluted, full scan analysis, the sample must be analyzed via 8260D SIM for those compounds.

2.6 In order to meet lower reporting limits of 0.5ug/L for most analytes, 2.5 ug/L for ketones and generally lower limits for other non-routine analytical compounds, samples must be analyzed against an initial calibration with a low point at those levels. The corresponding TALS login method for low level aqueous analysis is 8260_LL. See Table 3b for initial calibration levels and spike amounts.

3.0 Definitions

3.1 For a complete list of definitions refer to Appendix 2 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 This method is susceptible to contamination from a number of sources, including organic solvents used in other laboratory procedures, impurities in the purge gas, improper cleaning of syringes or purge vessels, and carryover from high level samples. Samples can be contaminated by the diffusion of volatile organics through the septum during shipment or storage. Steps have been taken to ensure that these potential problems are eliminated from the laboratory.

4.2 The volatiles analytical laboratory is housed in a separate building, away from the organic extraction lab area where large quantities of organic solvents are used. No organic solvents are used or stored in the volatiles laboratory.

4.3 The nitrogen used as purge gas passes through a solvent trap prior to its inlet into the purge and trap units.

4.4 Trip Blanks are shipped to clients with aqueous bottle ware as requested. The purpose of the trip blank is to detect and identify any VOC contamination of the samples while in transit to and from the lab. The blank is created at the laboratory by completely filling the volatile vial container with lab grade organic free deionized water and sealing the container Trip Blanks accompany bottle ware and samples through the sampling, storage and analysis stages as a check on contamination that may occur at these points.

4.5 Individual samples are each handled with a unique syringe that has been baked in a drying oven at 105°C to ensure the absence of volatile compounds.

4.6 Carryover can occur anytime a high level sample is analyzed. Screening procedures are employed to ensure that a sample is analyzed at an appropriate dilution to minimize potential carryover. When a high level sample is analyzed, it is followed by the analysis of a reagent water blank. If another sample was analyzed after the high level sample, this sample is inspected carefully for signs of carryover. If this sample does not contain any of the compounds found in the high level sample, the system can be considered contamination free.

4.7 The analytical system is checked daily with the analysis of a method blank. This blank must meet all quality control criteria for the method before sample analysis may take place.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

Any questions pertaining to safety issues or procedures should be brought to the department manager or Edison Safety Officer.

5.1 Specific Safety Concerns or Requirements

5.1.1 Latex, nitrile and vinyl gloves all provide adequate protection against the methanol used in this method.

5.1.2 Purge vessels on purge-and-trap instruments can be pressurized by the time analysis is completed. Vent the pressure prior to removal of these vessels to prevent the contents from spraying out.

5.1.3 The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

5.1.4 The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.1.5 There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1 Purge and trap units from several different manufacturers are used, depending upon the sample matrix and preparatory technique required. A purge and trap unit consists of three parts: the sample purge unit, the trap, and the concentrator. Unit configurations currently in use are:

- OI Analytical 4551, 4100 Automatic Sampler/4660,4760 concentrator;
- Archon 5100A Automatic sampler/ OI Analytical 4660,4760 concentrator;
- EST Centurion Autosampler/ EST Encon concentrator;
- Archon Autosampler/EST Encon concentrator.
- Archon/EST Evolution

6.1.2 A VOCARB 3000 trap from Supelco is used in the Encon concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed with 10.0cm Carbopack B, 6.0 cm Carboxin 1000, and 1cm Carboxin 1001.

6.1.3 An OI analytical purge trap #10 is used for the OI 4560,4660 and 4760 concentrator. The trap is 25cm long with an inside diameter of 0.105 inches. The trap is packed to contain the following absorbents: Tenax/silica gel/carbon molecular sieve.

6.1.4 Alternate traps may be used provided the adsorption and desorption characteristics are equivalent to those of the trap recommended by the method.

6.1.5 Both the Encon and OI concentrators are capable of rapidly heating the trap to 260°C and holding at that temperature for the duration of the desorb time.

6.1.6 Gas chromatograph: HP Agilent 6890/7890 equipped with temperature programming capability.

6.1.7 GC column: 30M long x 0.25mm ID, 1.4um film thickness, 20M x 0.18mm x 1um DB-624 and 20M long x 0.18 mm ID Restek Rtx-VMS capillary column with 1um film thickness or similar phase.

6.1.8 Mass Spectrometer (Agilent 5973/5975/5977): scanning from 35-260 amu every 0.9 seconds, utilizing 70 volts (nominal) electron energy in the electron ionization mode and producing a mass spectrum which meets all EPA performance criteria when 50 ng of 4-Bromofluorobenzene (BFB) is injected through the gas chromatograph inlet.

6.1.9 GC/MS Interface: transfer lines heated to 180°C .

6.1.10 Data system: HP Chemstation II for data acquisition and Eurofins Chrom for data processing.

6.2 Supplies

- Microsyringes: 10 ul to 1000 ul.
- Syringes: 5 ml to 25 ml gas-tight.

- Injection port liners: HP 18740-80200 or equivalent
- Volumetric flasks: Class "A" glassware, 5 ml to 500 ml.
- VOA vials: 20-ml and 40-ml glass with PTFE – faced septum.
- Vials: 2-ml amber glass with screw cap with Teflon-faced septa.
- Top loading analytical balance.
- Spatula: Narrow, stainless steel.
- Stir bars: PTFE coated, small enough to spin freely inside a VOA vial.

7.0 Reagents and Standards

7.1 Reagents

7.1.1 Organic free reagent water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.1.1) Reagent water is obtained from Millipore system. Other methods of preparing reagent water are acceptable, provided that the water produced meets method blank criteria.

7.1.2 Methanol: Ultra Resi-Analyzed, purge and trap grade, purchased from JT Baker or equivalent. (Cat # 9077-02)

7.1.2.1 Each lot of methanol is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2 Standards

7.2.1 **Calibration Standards** Stock target compound analytical standard solutions are purchased mainly from Restek, Supelco, Inc, Absolute Standards and Spex although standards of similar quality from other suppliers may be substituted as required. Standards noted with an asterisk (*) are custom mixes made especially for Eurofins Edison.

Target Analyte Standard Name	Concentration	Vendor	Catalog #
8260 List 1 / Std #3 Gases*	2500 ppm	Restek	569722
8260 List 1 / Std #3 Gases – (SS)*	2500 ppm	Restek	569722 sec
8260 List 1 / Std #1 MegaMix*	1250-62500 ppm	Restek	569720
8260 List 1 / Std #1 MegaMix (SS)*	1250-62500 ppm	Restek	569720 sec
8260 List 1 / Std #2 Ketones *	12500 ppm	Restek	569721
8260 List 1 / Std #2 Ketones * (SS)	12500 ppm	Restek	569721 sec
8260 List 1 / Std #5 Acrolein *	20,000 ppm	Restek	568720
8260 List 1 / Std #5 Acrolein (SS)	20,000 ppm	Restek	568720 sec
8260 List 1 /Std #4 2 CEVE *	2500 ppm	Restek	569723

Target Analyte Standard Name	Concentration	Vendor	Catalog #
8260 List 1 /Std #4 2 CEVE (SS) *	2500 ppm	Restek	569723 sec
8260 List 1 /Std #6 Vinyl Acetate *	5000 ppm	Restek	569724
8260 List 1 /Std #6 Vinyl Acetate (SS) *	5000 ppm	Restek	569724 sec
8260 List 2 / Std #1 Additions *	2500-62500ppm	Restek	568725
8260 List 2 / Std #1 Additions (SS) *	2500-62500 ppm	Restek	568725 sec
8260 List 3 / Std #1 Polar Additions *	2500-100000ppm	Restek	568728
8260 List 3 / Std #1 Polar Additions (SS) *	2500-100000 ppm	Restek	568728 sec
VOC Extra Standard 2015 *	2500-5000 ppm	Absolute	98593
VOC Extra Standard 2015 * (SS)	2500-5000 ppm	Absolute	98593
Epichlorohydrin	1000 ppm	Absolute	70377
Acrolein	5000 ppm	Restek	91980
Acrolein *	Neat	Sigma	110221
2-Freon Mix quote # 12258 *	2500ppm	Absolute	12258
2-Freon Mix quote # 12258 * (SS)	2500ppm	Absolute	12258
1,4-Dioxane	Neat	Sigma	360481
Epichlorohydrin	Neat	Sigma	45340
2-Chloroethylvinyl ether	Neat	Sigma	109983
1,4-Dioxane	1000 ppm	Absolute	70373
1,4-Dioxane	10000 ppm	Absolute	92785
Benzene	1000 ppm	Absolute	70025
Chloroform	1000 ppm	Absolute	70076

(1): The separate source for this material is not available as a distinct catalog number. Analyst must ensure that a separate lot of the material is selected and used as required.

An asterisk (*) indicates a custom standard mix.

7.2.1.1. Prepare stock solutions at volumes and concentrations indicated in Table 2 (Working Standards Preparation) by combining the indicated volumes of each stock solution into a volumetric flask corresponding to the total final volume. Dilute to the volume marker with methanol.

7.2.1.2. Prepare individual calibration standards as applicable per Section 9.2.2.1, Table 3, Initial Calibration Standards Preparation, Low Level Soil, Table 3a, Initial Calibration Standards Preparation (Low Level), Aqueous or Table 3B Initial Calibration Standards Preparation, Aqueous.

7.2.1.3. The 'Second Source' standards listed are used in the preparation of the Initial Calibration Verification (ICV) standard (see Tables 4 and 4a for ICV preparation instructions) and the Laboratory Control Standard (LCS) (see Section 9.1.3 and Tables 4 and 4a).

7.2.2 **Surrogate Standards:** Surrogate standard solutions are prepared from the stock solution (2500ppm)

Surrogate Standard Name	Concentration	Vendor	Catalog #
4-Bromofluorobenzene	2500ppm	Restek	567650
Toluene-d8			
1,2-Dichloroethane-d4			
Dibromofluoromethane			

7.2.2.1 A primary surrogate stock solution (2500 ppm each) is prepared from the neat standards as follows:

7.2.2.2 Secondary surrogate standard solutions are prepared at two (2) levels using the 2500 ppm primary stock solution as detailed in the table below:

Standard Name	Vendor	Catalog #	Volume added	Concentration of Stock Std.	Concentration of Standard	Total Volume Volume in MeOH/Total volume of MeOH
8260 Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4 Dibromofluoromethane	Restek	567650	1ml	2500ppm	250ppm	10mL 9.0mL TV/M
8260 Surrogate Mix: 4-Bromofluorobenzene Toluene-d8 1,2-Dichloroethane-d4 Dibromofluoromethane	Restek	567650	1ml	2500ppm	50ppm	50mL 9.0mL TV/M

7.2.2.3 Methanol/Surrogate solution (2.5ug/mL): For methanol sampling field kits. Prepared by adding 1mL of 2500 ug/ml primary surrogate stock solution (see Section 7.2.2.1) to 1 L purge and trap grade methanol.

7.2.3 **Internal Standards:** Internal Standards Solutions are purchased from Restek:

Standard Name	Concentration	Vendor	Catalog #
8260 Internal Standard Mix: *Chlorobenzene-d5 *1,4-Dichlorobenzene-d4 *Fluorobenzene *1,4-Dioxane-d8 *TBA-d9	250-5000ppm	Restek	567649

7.2.4 **Internal Standard/Surrogate Mix (125 ppm each):** A solution containing both Internal Standards and Surrogates at 125 ppm is prepared in a 10ml volumetric flask as detailed below using the 2500

ppm surrogate stock solution prepared in Section 7.2.2.1 and the 2500 ppm internal standard mix detailed in Section 7.2.3:

Standard Name	Concentration of Stock Std.	Volume added to final volume of 20ml MeOH	Final Concentration of Standard
8260 Internal Standard/Surrogate Mix (125 ppm) For Aquatek Autosampler	2500 ppm Surrogate Mix	1.0ml	125 ppm each component
	250 Internal Std Mix	10 ml	

7.2.5 **Internal Standard/Surrogate Mix (SIM) (2.5/50 ppm each):** A solution containing both Internal Standards and Surrogates at 25 ppm is prepared in a 10ml volumetric flask as detailed below using the 2500 ppm surrogate stock solution prepared in Section 7.2.2.1 and the 250 ppm internal standard mix detailed in Section 7.2.3:

Standard Name	Concentration of Stock Std.	Volume added to final volume of 10ml MeOH	Final Concentration of Standard
8260 Internal Standard/Surrogate Mix (25 ppm) (SIM)	2500 ppm Surrogate Mix	10ul	2.5/50 ppm each component
	250 Internal Std Mix (Restek)	10ul	
1,4-Dioxane-d8	10000 ppm	50ul	

7.2.6 **GC/MS Instrument Performance Check (BFB):** The instrument performance check solution consists of 4-Bromofluorobenzene in addition to the other three surrogates in methanol. Prepare the solution at **50ppm as specified in section 7.2.2.2**. Assign an expiration date of 6 months.

7.2.7 All standards preparation information must be logged into the TALS Reagent Module. All pertinent information must be entered: Date prepared, Lot #'s, Expiration dates, Solvents used, Lab Lot # (expiration date), Manufacturer and Verification signature. Additionally, all prepped standards are typically given a unique Lot# and all information pertaining to standard preparation is entered into the GC/MS VOA Standard Preparation Log Book. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

7.2.8 Please refer to Eurofins Edison SOP No. ED-GEN-008, *Standard Operating Procedure for Preparation, Purity and Storage of Reagents and Standards*, current revision. For Method 8260D:

- Shelf Life of Standard:
  - Stock standards (Non-gases) - 6 months after opening vendor stock of up to 2000ppm, 3 years for 10,000ppm, 5 years for over 50,000ppm, or manufacturer's expiration date whichever comes first.
  - Stock Standards (Gases) - 2 months after opening vendor stock, or manufacturer's expiration date whichever comes first.
  - Working/Secondary dilution Standards (Non-gases) – 6 months after preparation, or manufacturer's expiration date whichever comes first.
  - Working/Secondary dilution Standards (Gases) – 1 week from the date of preparation for 50ppm and 2 weeks for 500ppm, or manufacturer's expiration date whichever comes first.
  - Daily Calibration Standards – 24 hours after preparation.

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- Storage Requirements:
  Aqueous standards are stored at 4°C and Methanol standards are stored at -10°C to -20°C.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass 40 ml vials	40 mLs	HCl, pH < 2; Cool 4 °C ± 2°C	14 Days / preserved 7 Days / unpreserved	SW846 Method 5030
Waters	Glass 40 ml vials	40mLs	TSP,pH>11 Cool 4 °C ± 2°C	14 Days / preserved	SW846 Method 5030
Soils (Low)	Encore or Terracore (40 ml vials)	5 grams in 5 mls DI H_2O	Frozen Stored -7°C to -20°C	14 Days	SW846 Method 5035A

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soils (Med)	Encore or Terracore (40 ml vials)	5 grams in 10 mls MeOH	Cool 4 °C ± 2°C	14 Days	SW846 Method 5030
Soils (High)	Glass (Lab Prepared Kits)	10 grams in 25 mls MeOH	Cool 4 °C ± 2°C	14 Days	SW846 Method 5030

8.1.1 There are several methods of sampling soil. The recommended method is to take samples using an EnCore™ sampler or using a Terra Core™ sampling kit. At specific client request, unpreserved soil samples in 4oz jars may be accepted. For EnCore and Terra Core sampling, a separate jar is required for percent solids/moisture determination, unless one is supplied for another analysis.

8.1.2 For EnCore™ samplers, the 5g sample is extruded into a pre-weighed 40mL vial containing 5mL of methanol (medium level analysis) or reagent water (for low level, <50 µg/kg, analysis). The exact samples weight is determined as the difference between the vial + preservative weight and weight after the sample is added.

- Samples must be transferred (extruded from the sampler) and preserved within 48 hours of sampling.
- Water preserved samples are then frozen at <10°C. Methanol preserved samples may be stored at > 0.0 °C but < 6° °C or frozen.
- Methanol preserved samples are shaken for at least 2 minutes, and a portion of the methanol extract after settling may be transferred to a smaller Teflon-lined capped vial for storage below 6 °C
- Normally one (1) medium level and two (2) low-level samples are taken and preserved.
- One vial with a clean matrix of each preservation type is prepared at the same time as samples, to be used for LCS analysis. Spikes are not added until the time of analysis.
- Samples are spiked with internal standards and surrogates at the time of analysis.

8.1.3 Terra Core™ sampling kits are pre-preserved for use and immediate samples preservation in the field. Kits are shipped that include one (1) methanol preserved and two (2) reagent water preserved vials, along with a 4oz jar for solids/moisture analysis volume.

- Terra Core™ vials are immediately placed in the freezer (<-10°C) upon receipt at the lab. Methanol preserved vials are shaken for at least two (2) minutes to break up the solid and create the methanol extract.
- Terra Core™ vials are labeled with the weight of the vial and preservative. The vials are re-weighed prior to analysis to determine the weight of the solid sample added. It is important that labels NOT

be added to these vials prior to weighing, because the weight of the label will add to the sample weight. Vials may be marked with indelible marker, or placed in a labeled, sectioned box until ID labels can be added after weighing.

8.2 Unpreserved soils - At client request, unpreserved soils packed into glass jars or brass tubes may be accepted and subsampled in the lab. A 5g portion of the sample is transferred to a 40mL vial and mixed with reagent water and/or methanol for analysis. Since this procedure is not compliant with SW5035A an NCM and case narrative statement describing the non-conformance must be included with any resulting data reported to the client.

8.3 Aqueous samples are stored in 40mL glass vials with Teflon lined septa at >0 and ≤ 6.0°C. Vials are required to have no headspace larger than a small pea.

8.3.1 Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources

8.3.2 Regulatory requirements for 2-Chloroethyl vinyl ether:

- 2-Chloroethyl vinyl ether: The stability of this compound is reduced when subjected to low pH, therefore samples for analysis to include 2-CEVE must be taken without acid preservation. Unpreserved samples must be analyzed within 7 days.

- SW846 Update V removed special preservation requirements for Acolein and Acylonitrile. These compounds may be analyzed for using a preserved sample vial.

8.4 Soil samples and water samples preserved to pH <2 with HCl have a maximum holding time is 14 days from sampling until the sample is analyzed. If water samples are known to be unpreserved, the holding time is 7 days from sampling to analysis.

8.4.1 Preserved water samples are checked to confirm the preservation pH AFTER analysis because the vials must not be opened prior to analysis. If the pH is found to be >2, this must be addressed in the case narrative.

8.5 Medium level solid methanol extracts, if taken at the time of preservation, are aliquoted into 4 mL glass vials with Teflon lined caps and stored at > 0.0 °C but ≤6.0°C or frozen. The extracts are stored with minimum headspace.

8.6 Storage blanks are prepared by filling 40 mL VOA vials with reagent water and placing one in each refrigerator. After 1-2 weeks, the storage blanks are removed and analyzed. Additional details can be found in Eurofins Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision.

9.0 Quality Control

9.1 **Sample QC** - The following quality control samples are prepared with each batch of samples:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standards	Every samples	Response within -50% to +100% of CCV

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. **Method blanks** are analyzed every 12 hours immediately after successful calibration verification (ICV and CCV) and before any samples are analyzed during the 12 hour clock. Analyze the blank in the same manner as the associated samples.

9.1.1.1. Prepare an aqueous blank by filling a 40 mL vial with reagent water and placing it in the autosampler. The autosampler will add the internal standard and/or surrogate standard.

9.1.1.2. Prepare a medium or high level blank in a 50 mL volumetric flask by adding 1.0 mL of purge and trap grade methanol to reagent water and bringing up to volume with the reagent water. The appropriate volume of this mix is added to the purge vessel. The autosampler will automatically internal standard and/or surrogate standard.

9.1.1.3. Prepare a low- level soil blank in a 40 ml VOA vial by adding a magnetic stir bar and 5 ml of reagent water and placing the vial in the autosampler tray. An additional 5mL of reagent water plus 1uL of 250ppm Internal Standard/Surrogate Mix (see Section 7.2.4) will be added by the Archon prior to purging.

9.1.1.4. To be considered acceptable, the method blank must not have any target analytes above the reporting limit. For method 8260D method blank is acceptable when target analyte concentrations are less than one-half of the reporting limit. Method blanks may contain analyte concentrations greater than acceptance limits if the associated samples in the batch are unaffected or sample

concentrations/responses are >10x the blank If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be re-extracted and re-analyzed. Re-analysis 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. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed. Method blanks, trip blanks and other field blanks must be carried out through all stages of sample preparation and analysis.

9.1.1.5. Surrogate recoveries for the method blank must be within the laboratory generated limits. (Method 8260D requires the use of a minimum of three (3) surrogates. Since we are spiking with four (4) surrogates, either 1,2-Dichloroethane-d4 or dibromofluoromethane can be recovered outside of control limits without corrective action) .Internal standard area counts in the method blank must be within method specified limits. If any surrogate or internal standard is outside the limits, the method blank must re-analyzed.

9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD): A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch which may contain up to 20 samples, and additional samples can be added to the batch for 14 days after the first sample was analyzed). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared (as described in Section 9.1.2.1) concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria which are updated annually. For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database.

9.1.2.1. Prepare the MS/MSD as follows:

9.1.2.1.1 Low Level Soil: The low level soil MS/MSD is prepared as detailed in the following table. This is prepared in duplicate (one for the MS, the other for the MSD) in a 5 ml syringe filled with reagent water. Once prepped the solution is added to separate 40 ml vials each containing 5 gram aliquots of the sample to be spiked :

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 5.0 ml of Reagent Water	Final Concentration (ug/kg)
Gas Mix Li	50ppm	2	20

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 5.0 ml of Reagent Water	Final Concentration (ug/kg)
8260 combined	50ppm	2	20
Acrolein	500 ppm	3	300
Propenes	50ppm (varied)	2	20 (varied)
Freons	50 ppm	2	20

9.1.2.1.2 Aqueous Samples: The MS/MSD for aqueous samples is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 50 ml volumetric flasks filled with an aliquot of sample to be spiked. Once prepped the solution is poured into a 40 ml VOA vial and loaded onto the purge and trap autosampler:

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 50 ml of Sample	Final Concentration(ug/L)
Gas Mix Li	50ppm	20	20
8260 combined	50ppm	20	20
Acrolein	500 ppm	4	40
Propenes	50ppm (varied)	20	20 (varied)
Freons	50	20	20

9.1.2.1.3 Medium & High Level Soils: The MS/MSD for medium/high level soils is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 50 ml volumetric flasks filled with reagent water which has been previously spiked with the methanol sample extract. Once prepped the solution is poured into a 40 ml VOA vial, the and loaded onto the purge and trap autosampler:

Standard Solution (Reference Table 2, Lab Names)	Concentration	Volume of Standard (ul) Added to 50 ml of Reagent Water containing sample methanol extract	Final Concentration (ug/L)
Gas Mix Li	50ppm	20	20
8260 conbined	50ppm	20	20
Acrolein	500ppm	4	40
Propenes	50ppm (varied)	20	20 (varied)
Freons	50 ppm	20	20

9.1.2.1.4 SIM: The MS/MSD for SIM samples is prepared as detailed in the following table. This is prepared in duplicate (one for MS, the other for MSD) in 100 ml

volumetric flasks filled with an aliquot of sample to be spiked. Once prepped, two separate 10ml solution is poured into 40 ml VOA vials, 2ul of SIM IS/S is then added to each vial and loaded onto the purge and trap autosampler:

Standard Solution	Concentration	Volume of Standard Added to 100 ml of Sample (ul)	Final Concentration (ug/L)
8260SIM Mix1	10ppm	0.5	0.05
1,4-Dioxane	50ppm (varied)	10	5
Benzene/Chloroform	10ppm	0.5	0.05

9.1.2.2. An Laboratory Control Sample (LCS) /Laboratory Control Sample Duplicate (LCSD) may be substituted for the MS/MSD if insufficient sample volume is available (see Section 9.1.3).

9.1.3. Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be prepared analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (see For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference. If the LCS recovery results are outside the method specified, the LCS is reanalyzed. If, upon reanalysis, the LCS is it is still outside of limits the entire batch must be reanalyzed. For 8260D, when an LCS is prepared in the same manner as CCV, the same standard can be used as both the LCS and CCV.

9.1.3.1 For LCS preparation instructions please refer to Section 9.1.2.1 for low level soil introduction technique (note: use reagent water only, no solid matrix is used when preparing the LCS) and Sections 9.1.2.1.2 and 9.1.2.1.3 as applicable for aqueous/medium or high level solids introduction (note: use reagent water only, no sample or sample extract is used when preparing the LCS).

9.1.3.2 The LCS for SIM samples is prepared as detailed in the following table. This is prepared in a 200 ml volumetric flasks filled with organic free reagent water. Once prepped, 10ml of the solution is poured into a 40 ml VOA vial and 2ul IS/SS added manually and loaded onto the purge and trap autosampler

Standard Solution	Concentration	Volume of Standard Added to 200 ml of Reagent Water (ul)	Final Concentration (ug/L)
8260 Mix1	10ppm	1	0.05
1,4-Dioxane	50ppm	20	5
Benzene/Chloroform	10ppm	1	0.05

9.1.3.3 A Laboratory Control Sample Duplicate (LCSD) is analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a four (4) component surrogate standard mix (see Section 7.2.2). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (For acceptance limits, reference the current TALS (LIMS) active Method Limit Group database).

9.1.4.1. Surrogate recovery limits are lab generated and are updated annually.

9.1.4.2. Surrogate recoveries are calculated for the blank, samples, and QC samples. Surrogate recovery is calculated as:

$$\frac{\text{Concentration found}}{\text{Concentration added}} \times 100 = \%\ \text{RECOVERY}$$

9.1.4.3. If the surrogate recoveries of any blank, sample, or QC sample fails to meet the current recovery criteria, the sample must be re-analyzed. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary. Method 8260D requires the use of a minimum of three (3) surrogates. As we spike with four (4) surrogates, one can be recovered outside of control limits without corrective action.

9.1.5. Internal Standards: All samples, blanks, standards and QC samples are spiked with a five (5) component internal standard mix (See Section 7.2.3). The response (area count) and retention time of each internal standard in all samples, standards, blanks and QC samples are monitored.

9.1.5.1. The internal standard responses must be within -50 +100% of its corresponding internal standard in the mid-level calibration standard or the active calibration curve. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

9.1.5.2. Internal standard retention time is evaluated immediately after acquisition. The retention times of the internal standards must be within ±30 seconds of the internal standards from the mid point standard of the initial calibration or the calibration verification standard. Any blank, sample, or QC sample that fails to meet these criteria must be re-analyzed.

9.2 Instrument QC

9.2.1 GC/MS Instrument Performance Check (BFB): The GC/MS system is tuned using Perfluortributylamine (PFTBA) such that an injection or purging of 50ng of 4-Bromofluorobenzene (BFB) meets the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all BFB key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples. For method 8260D tune checks are only required prior to initial calibration. (**NOTE**: see Method Modifications in Section 16.0).

BFB Key Ions and Abundance Criteria	
Mass	**Ion Abundance Criteria**
50	15.0-40.0 percent of the base peak
75	30.0-60.0 percent of the base peak
95	Base peak, 100% relative abundance
96	5.0-9.0 percent of the base peak
173	Less than 2.0% of mass 174
174	Greater than 50% of the base peak
175	5.0-9.0 percent of mass 174
176	Greater than 95.0% but less than 101% of mass174
177	5.0-9.0 percent of mass 176

9.2.1.1. The BFB mass spectrum may be evaluated using one of the procedures listed below. The spectrum may be background subtracted using a single peak no more than 20 scans before the peak apex. The BFB spectrum must meet the technical acceptance criteria listed in the table above:

- A single scan on the peak;
- An average of the peak;
- Use of three scan averaging and background subtraction techniques. Select the scan at the BFB peak apex, add +1 scan from the apex and -1 scans from the apex;

9.2.1.2. BFB parameter settings are stored in a tune file, which ill be used in all subsequent analysis of standards and samples.

9.2.2 Initial Calibration Range and Initial Calibration Verification

9.2.2.1. **Initial Calibration:** The initial calibration range consists of a five-point concentrations (six points for second order regression) of analytical standards prepared as described in Tables 3, 3A and 3B as applicable (attached). The initial calibration range must be analyzed only after the BFB instrument performance check has met the criteria in Section 9.2.1. A separate initial calibration range is analyzed for each sample introduction technique. The last initial calibration standard may be used to be the start of the 12 hour clock for samples analyzed after initial calibration. Verify closely eluting isomers resolution in the mid-point concentration of the ICAL. Isomers are considered resolved if the peaks are at least 50% resolved (i.e., the height of the valley between two isomer peaks is less than or equal to 50% of the average of the two peak heights. This should also be checked in the daily CC's.

9.2.2.2. If analysis by the SIM technique is required, prepare calibration standards for, Vinyl Chloride, Chloroform, Benzene 1,2-dibromoethane, 1,2,3-Trichloropropane and 1,2-dibromo-3-chloropropane at concentrations of 0.02, 0.04, .05, 0.10, 0.20, 0.50, 1.0 and 2.0 ppb; 1,4-Dioxane at 0.4, 1, 5, 10, 20, 30, 40 and 50ppb. See Table 5 that summarizes the preparation information.

9.2.2.3. **Initial Calibration Verification (ICV):** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.1.3 and Tables 4 and 4a (full scan) and Table 6 (SIM) (attached). The ICV must be from a source separate from the standards used in the Initial Calibration Range.

9.2.3 **Continuing Calibration Verification (CCV):** A approximately mid-point (20ug/ml and 0.050/5ug/ml for SIM) Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the BFB instrument performance check. BFB is not a requirement for 8260D CCV verification. The CCV is prepared as detailed in Section 7.2.1.1 and Table 3 (attached).

9.2.4 **Calibration Acceptance Summary**

9.2.4.1. **Retention Time:** The relative retention times of each compound in the five calibration standards must agree within 0.06 relative retention time units.

9.2.4.2. **Initial Calibration Range:** Internal standard calibration is employed for this method. After the initial calibration range has been analyzed as detailed in Section 10.3.3 the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:
A_x = Area characteristic ion for the compound (see attached Table 7)
Ais = Area characteristic ion of internal standard (see attached Table 7)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

9.2.4.2.1. Determine the mean RRF for each compound using the five or six RFs from the initial calibration range.

9.2.4.2.2. The average RFs of the target analytes listed in the table below must meet the indicated minimum RF criteria:

Minimum Relative Response Factor	
Common Target Analytes	**Minimum RF**
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl Chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone *	0.050
Carbon disulfide	0.100
Methyl Acetate *	0.005
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone *	0.050
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichloropropene	0.200
trans-1,3-Dichloropropene	0.100
4-Methyl-2-pentanone *	0.050
Toluene	0.400

Minimum Relative Response Factor	
Common Target Analytes	**Minimum RF**
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone*	0.050
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100
Chlorobenzene	0.500
Ethylbenzene	0.100
meta-/para-Xylene	0.100
ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorobenzene	0.200

Note: Alternate ions chosen for the analytes in the table above may result in lower than recommended value

* These values are lower than method recommended values.

9.2.4.2.3. Any individual analyte that fails the minimum response factor above must have a demonstration of sensitivity in the analytical batch to report non-detects. The demonstration of sensitivity is analysis of a low level CCV (at or below the reporting limit). The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. The low level CCV would normally be analyzed immediately after the mid-level CCV

9.2.4.2.4. Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

The % RSD of the common target compounds listed above must be ≤20% for average RF in order for the calibration range to be acceptable. If more than 10% of the compounds exceed the 20%RSD limit and do not meet the minimum correlation coefficient (0.99) or Relative Standard Error (RSE) of ≤20% for alternative curve fits, appropriate instrument maintenance like source cleaning should be performed. Any compound that do not meet the 20%

RSD or 0.99 correlation coefficient/RSE criteria must be flagged as estimated for detects.

9.2.4.2.5. For all compounds (including those analyzed by SIM): in order to assume linearity, the % RSD of the RRF's for each target analyte must be ≤20%.

9.2.4.2.6. If the above listed criteria is met, the system can be assumed to be linear, sample analysis may begin and the average RF from the initial calibration range may be used to quantitate all samples.

9.2.4.2.7. An alternative calibration technique may be employed for those any compounds exceeding the 20% RSD criteria:

9.2.4.2.5.4 Linear regression: Calculate the first order linear regression for any compound which did not meet the 20% RSD criteria the r^2 (Correlation Coefficient) value must be $\geq$ 0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.5.4 Quadratic (or second order) regression: may be used if the linear regression correlation coefficient exceeds criteria. Quadratic regression requires the use of a minimum six calibration points. If second order regression calibration is used, , the r^2 (Correlation Coefficient) value must be $\geq$ 0.99 or the Relative Standard Error (RSE) (≤20%) for the calibration to be employed.

9.2.4.2.8. If neither of the alternative calibration techniques meets acceptance criteria i.e for more than 10% of the analytes fail both 20%RSE and 0.990 the calibration is not valid. Corrective action must be taken and the initial calibration range reanalyzed.

9.2.4.2.9. Non-detect results for any analyte that fails both 20%RSD/RSE and 0.990 correlation coefficient may be reported without flagging if (and only if) there has been a successful analysis of a LLCCV (CCV at the reporting limit) in the same analytical batch. The criterion for the LLCCV is detection only (%D criteria are not applied) but the standard qualitative criteria in the method must be met. Flagging of detected analytes results as estimated is discouraged when the 20%RSD/RSE and 0.990 criteria fails. In general no more than one or two of the poorest performing analytes should fail both criteria.

9.2.4.2.10. Due to significant bias to the lower portion of a calibration curve using the linear regression fit model a quantitation check on the viability of the lowest calibration point should be performed by re-fitting the response from the low concentration calibration standard back into the curve as if it were an unknown sample (rename the lower point calibration file as a separate data file before re-processing). The results should be within ±30% of the standard's true concentration. This is not required for average RF or quadratic fits. Additionally forcing a linear regression through zero will meet the requirement of not re-fitting. Analytes which do not meet the minimum quantitation calibration re-fitting criteria should be considered 'out of control'. Report those target analyte outliers as estimated when the concentration is at or near the lowest calibration point and/or report to the next reporting level (i.e., the next higher calibration point for the analyte).

9.2.4.2.11. For additional detail refer to Eurofins Edison Work Instruction No. EDS-WI-096, *8260C ICAL Procedure*, latest revision.

9.2.4.3. Calibration Point Read-back Criteria: Whichever calibration model above is selected, it should be subjected to an additional check to establish the representativeness of the data that were used to produce it. This check is the refitting of each calibration point response back to the calibration model, or the comparison of the calculated amount of the standard against the expected amount.

- CHROM software provides an Initial Calibration %Drift report which shows the % Error for each calibration point. This report must be reviewed in addition to the %RSD Linear Response Factor.

- The absolute value of the % Error for each calibration point should be < 30%. For the lowest calibration point, the % Error may be < 50%. Relative standard error (RSE) can also be used and must be ≤ 20% for each calibration point. See section 11.10 for the Calculation of the %Error.

9.2.4.4. Initial Calibration Verification (ICV): Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 9.2.2.2) must be analyzed and evaluated. The ICV must meet the criteria of 70-130% recovery for all compounds however up to 10% of the compounds are allowed to exceed

this criteria as long as their recoveries are within 65-135%. For the poor performers the range is 50-150%. If the criterion is not met, a second ICV may be analyzed after corrective measures are taken. If a second ICV analysis fails to meet criteria proceed with corrective action and the analysis of a new initial calibration range. Flagging: If the ICV limits are outside of criteria (high) for an analyte and that analyte is undetected in the sample, no flagging or narration is required. If the ICV limits are outside of criteria (low) for an analyte and that analyte is undetected in a sample, narrate the non-conformance in an NCM. When that out of spec analyte is detected in a sample, describe the issue in the narrative, or flag as estimated.

9.2.4.5. Continuing Calibration Verification (CCV): A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a BFB instrument performance check, and analysis of a calibration verification standard.

9.2.4.4.1 Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.1 using a 50 ng injection of BFB. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process. For method 8260D, tune verification is not required for daily CCV.

9.2.4.4.1.1 Calibration Verification: Analyze the calibration verification standard immediately after a BFB that meets criteria. For method 8260D, BFB is not needed. Use the mid point calibration standard (20ug/L). **NOTE:** The same sample introduction technique employed for the initial six-point calibration must be used for the calibration verification.

9.2.4.4.1.2 Calculate response factors (RF) for each compound using the internal standard method.

9.2.4.4.1.3 The RFs must meet the minimum RF criteria listed in the table in Section 9.2.4.2.2.

9.2.4.4.1.4 Calculate the % Difference for each response factor in the calibration check standard vs. the response factors from the initial calibration.

9.2.4.4.1.5 If the percent difference/drift (%D) for the compounds listed in the table in Section 9.2.4.2.2 is ≤20%, the initial calibration is assumed to be valid. If the ≤20% D criteria is not met for more than 20% of the compounds in the initial calibration, corrective action/ investigation may be taken. After corrective action, another calibration verification standard may be injected. If the response for the analyte is still not ≤20%, a new initial calibration range must be generated.

9.2.4.4.1.6 For the poor performing compounds listed below that fail the 20%D or 50%D criteria adequate sensitivity may be demonstrated by including a low level standard (LLCCV) in the analytical batch.

Poor Performers	
Acetone	Acrolein
Carbon disulfide	1,4-Dioxane
2-Butanone	Cyclohexane
2-Hexanone	Methyl cyclohexane
4-Methyl-2-pentanone	Benzyl chloride
Chlorodibromomethane	Naphthalene
1,2-Dibromo-3-chloropropane	Cis-Dichloropropene
Bromomethane	Trans-Dichloropropene
Chloroethane	All Alcohols

When samples have non-detects for an analyte that fails the SOP criteria with low recovery a low level CCV must be analyzed in the batch as a demonstration of adequate sensitivity. The criterion for a passing LLCCV is detection only, and a passing LLCCV allows non-detects to be reported without flagging. Any sample detects for an analyte that fails the SOP criteria must be flagged as estimated, or detailed in the case narrative. In all cases every effort should be made to re-analyze on an instrument with a passing CCV.

9.2.4.4.1.7 Percent drift is used instead of percent difference in calibrations employing either the linear or second order regression modes.

9.2.4.4.1.8 For the compounds not listed in the table in Section 9.2.4.2.2: No one individual compound of interest may exceed 50%D. For SIM analysis the %D is 20%.

9.2.4.4.1.9 The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.

9.2.4.4.1.10 Internal standard area response is also evaluated immediately after acquisition. The response (area count) of each internal standard in the calibration verification standard must be within 50% - 100% of its corresponding internal standard in the mid-level calibration standard of the initial calibration curve. If the EICP area for any internal standard changes by more than a factor of two (-50% to +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

10.0 Procedure

10.1. Gas Chromatograph/Mass Spectrometer Operation

10.1.1. The instrument operating parameters are set as follows at the beginning of a method of analysis and remain constant throughout the entire analytical procedure

10.1.1.1 Full Scan Operating Mode

Purge and trap unit

Purge Time:	11 minutes
Dry Purge:	1 Minutes
Purge Gas:	Nitrogen
Purge Flow:	40-45 ml/min
Purge Temp:	Water: Ambient; Solids: 40°C
Trapping Temp:	Ambient, <30°C

Desorb Time:	1 Minute
Desorb Temp:	VOCARB: 260°C, #10: 190°C

Gas chromatograph

Injector:	180°C
Carrier Gas:	Helium
Carrier Flow:	6 ml/min, 6890: 0.8 ml/min
Oven Program:	40°C for 1 min, 8°C/min to 90°C, 20°C/ min to 250°C for 3 min; 6890: 40°C for 1 min, 8°C/min to 100°C, 24°C/min to 220°C for 2 min
Run Time:	15 - 20 Minutes

Mass Spectrometer

Electron Energy:	70 volts (nominal)
Mass range:	35-260 AMU
Scan time:	0.9 sec./scan
Source Temp:	200°C
Separator Temp:	180°C

10.1.1.2 SIM Operating Mode

Purge and trap unit

Purge Time:	11 minutes
Dry Purge:	1 Minutes
Purge Gas:	Nitrogen
Purge Flow:	40-45 ml/min
Purge Temp:	Water: Ambient; Solids: 40°C
Trapping Temp:	Ambient, <30°C
Desorb Time:	1 Minute
Desorb Temp:	VOCARB: 260°C, #10: 190°C

Gas chromatograph

Injector:	180°C
Carrier Gas:	Helium
Carrier Flow:	6 ml/min, 6890: 0.8 ml/min
Oven Program:	40°C for 1 min, 8°C/min to 90°C, 20°C/ min to 250°C for 3 min; 6890: 40°C for 1 min, 8°C/min to 100°C, 24°C/min to 220°C for 2 min
Run Time:	15 - 20 Minutes

Mass Spectrometer

Electron Energy:	70 volts (nominal)
Mass range:	35-260 AMU
Scan time:	0.9 sec./scan
Source Temp:	200°C
Separator Temp:	180°C

SIM Parameters:

Group 1
Plot 1 Ion: 51.0/96

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	51.0	100	58.0	100	65.0	100
	67.0	100	70.0	100	88.0	100
	96.0	100	78.0	100	83.0	100
	85.0	100	62.0	100	64.0	100

Group 2
Group Start Time: 6.20
Plot 1 Ion: 82/117

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	82.0	100	107.0	100	109.0	100
	117.0	100				

Group 3
Group Start Time: 8.50
Plot 1 Ion: 75/157

Ions/Dwell in Group	(Mass	Dwell)	(Mass	Dwell)	(Mass	Dwell)
	75.0	100	95.0	100	150.0	100
	152.0	100	152.0	100	157.0	100
	174.0	100				

10.2. Sample Preparation

10.2.1. Screening: All soil samples extracts must be screened by GC/FID static headspace or MS analysis to provide the analyst with appropriate initial dilution factors. Aqueous samples without historical results are also screened. For additional details see Eurofins Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

10.2.2. Aqueous Samples: Unopened 40 mls vials with aqueous samples are placed in an Archon autosampler. 1 uL of Internal Standard/Surrogate Mix (see Section 7.2.4) is added by the Archon as the 5 mL of the sample passes through the sample loop.

10.2.3. **Medium or high level soils:** Medium or high level extracts that will be run on an Archon autosampler are prepared in 50mL volumetric flasks. The Archon can be set up to add 1uL of 250ppm Internal Standard/Surrogate separately (see Section 7.2.3 and 7.2.2.2) to each sample as the 5mL portion passes through the sample loop.

10.2.4. **Low level soils:** Low level soils must be run on an Archon autosampler. 1uL of 250ppm Internal Standard/Surrogate separately (see Section 7.2.3 and 7.2.2.2) and 5mL reagent water is added to each sample vial by the Archon immediately before the sample is purged.

10.2.5. **SIM analysis:** Aliquot 10ml of sample and manually add 2ul of 2.5/50ppm of internal standard/surrogate mix. Load to soil section of the autosampler for heated purge.

10.3. Instrument Performance and Calibration Sequence

10.3.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.3.2. Analyze the Instrument Performance Check Standard (BFB) as discussed in Section 9.2.1.

10.3.3. A unique initial calibration is then prepared for each sample introduction technique.:

10.3.3.1 **40 ml VOA Vial (Aqueous/Medium-High Level Soils):** Prepare aqueous calibration standards at six concentration levels for each parameter by adding the volumes of working standards listed in Table 3 to a 50mL volumetric flask of reagent water. Pour the calibration standards into 40mL VOA vials and load into the autosampler tray. If the internal standard is to be added by the Archon/OI autosamplers the addition of internal standard into the 50ml volumetric flaks may be omitted.

10.3.3.2 **40 ml VOA Vial (Low Level Soils):** If the calibration is for low-level soils prepared according to Method 5035AA, the calibration standards must be prepared by adding the volumes of working standards listed in Table 3 into a 5 mL syringe filled with reagent water and pouring the prepared standards into 40 mL VOA vials containing a magnetic stir bar.

10.3.4. Purge the standard for 11 minutes.

10.3.5. After purging is complete, desorb the sample onto the GC column by rapidly heating the trap to 260°C for VOCARB, 190°C for #10 and backflushing it with helium.

10.3.6. Begin the GC temperature program and data acquisition.

10.3.7. Re-condition the trap by baking for 12 minutes at 260°C for VOCARB, 210°C for #10.

10.3.8. Cool the trap to (<31°C). The trap is now ready for the next sample.

10.3.9. Transfer data to network, and process using CHROM software.

10.4. Sample Analysis Sequence

10.4.1. Once the initial calibration has been verified by successful analysis of an ICV and Method Blank, analysis of samples may begin.

10.4.2. Samples must be analyzed under the same instrument conditions and using the same injection volume as the calibration standards.

10.4.3. Equilibrate all samples to room temperature prior to analysis.

10.4.4. If the sample concentration exceeds that of the range, the sample must be diluted and re-analyzed.

10.4.5. The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

10.5. Data Processing

10.5.1. Prior to processing any standards or samples, target compound lists and sublists must be assembled in the Chrom system. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

10.5.2. Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.

10.5.3. Data is transferred from the acquisition PC to the network for auto-processing with CHROM software.

10.5.4. Each data file is checked for correct information including sample number, job number, QA batch, dilution factor, initial volume, final volume, and % moisture.

10.5.5. The data processing service from Chrom queries LIMS for the sample processing parameters.

10.5.6. Each data file is processed using calibration factors from the most recent initial calibration, quantitation from the daily calibration verification standard is not permitted.

10.5.7. The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW8260D are listed in Table 7.

10.6. Interpretation and Qualitative Identification:

10.6.1 Target Analytes: Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are 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:

10.6.1.1. Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.

10.6.1.2. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.

10.6.1.3. The relative retention time (RRT) of the sample component is within ± 0.06 RRT units of the RRT of the standard component.

10.6.1.4. The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.

10.6.1.5. All other ions that are greater than 10% in the standard target spectra should also be present in the sample.

10.6.1.6. 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%).

10.6.1.7. Structural isomers that produce very similar mass spectra should be identified as individual isomers if they have sufficiently different GC retention times. Otherwise, structural isomers are identified as isomeric pairs.

10.6.1.8. If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound will be positively identified and reported with documentation of the identification noted in the raw data record.

10.6.2 Non-Target Analytes: Upon client request a library search to identify non-target Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:

10.6.2.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.

10.6.2.2 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%).

10.6.2.3 Molecular ions present in the reference spectrum should be present in the sample spectrum.

10.6.2.4 Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.

10.6.2.5 Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.

10.6.2.6 If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid' , etc..).

10.7. Data Reporting

10.7.1. Final Report. LIMS TALS system automatically produces a data report consisting of key, hardcopy reports corresponding to specific data reporting requirements.

10.7.1.1. Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.

10.7.1.2. Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.

10.7.1.3. The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.

10.7.1.4. Data summaries for each method blank indicating which samples were extracted with the indicated blank.

10.7.1.5. A copy of the initial calibration range together with the calibration verification report, and tune report.

10.7.1.6. Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.

11.0. Calculations / Data Reduction

11.1. Target Compounds: are quantitated using the internal standard method.

11.1.1. Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).

11.1.2. The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3.. See Section 9.2.4.2 for discussion of RRF.

11.1.3. Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.

11.1.4. Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RRF)(Vs)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.

Ais = Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Vs = Volume of sample purged (ml)

11.1.5. Low Level Solid Samples

$$\text{Concentration (µg/Kg) (dry wt)} = \frac{(As)(Cis)}{(Ais)(RRF)(Ws)\,(DW)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

DW = Dry wt correction = $\frac{100 - \%\ \text{moisture}}{100}$

Ais = Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Ws = Weight of sample purged (g)

11.1.6. Medium Level Solid Samples

$$\text{Concentration (µg/Kg) (dry wt)} = \frac{(As)(Cis)(Vt)(1000)(D)}{(Ais)(RRF)(Va)(Ws)(DW)}$$

Where:

As = Area of the characteristic ion for the target analyte in the sample

Cis = Concentration of the internal standard (ug/L)

D = Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1

DW = Dry wt correction = $\frac{100 - \% \text{ moisture}}{100}$

Ais = Area of the characteristic for the associated internal standard

RRF = Average relative response factor from the initial calibration.

Va = Volume of the aliquot of sample methanol extract added to reagent water for purging in ul

Vt = Total volume of methanol extract in milliliters

Ws = Weight of sample purged (g)

11.2. Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method . For quantiation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 11.1) with the following revisions:

11.2.1. The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).

11.2.2. The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).

11.2.3. A RF on 1.0 is assumed.

11.2.4. The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.

11.3. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area characteristic ion for the compound (see Table 7)
Ais = Area characteristic ion of associated internal standard (See Table 7)
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.4. Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.2. (Initial calibration):

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.5. Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

$$\% \text{ D} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.6. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.7. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. NOTE: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

11.8. Accuracy:

$$\text{ICV , CCV and LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.9. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.10. Calculation of Percent (%) Error:

$$\%Error = \frac{x_i - x_i'}{x_i} x\,100$$

Where:

x_i ' = Measured amount of analyte at calibration level *i*, in mass or concentration units
x_i = True amount

11.11. Relative Standard Error (RSE):

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) study, as described in the Eurofins corporate Detection and Quantitation Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change affecting sensitivity is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Lower Limit of Quantitation Verification

The lowest calibration standard analyzed establishes the LLOQ or Reporting Limit. The capability to reliably detect this concentration through the preparation, clean-

up and analytical procedure is verified through the annual analysis of a standard at the LLOQ/RL. The LLOQ verification shall also be performed whenever significant changes are made to the preparation and/or analytical procedure. The annual LLOQ verification is completed and documented with the required annual MDL evaluation

12.3.1 The LLOQ verification standard shall be prepared at a concentration 0.5-2 times the LLOQ/RL, and be taken through all of the same preparation and clean-up methods as client samples.

12.3.2 The LLOQ verification standard for aqueous matrix shall be prepared using laboratory deionized water and for the solid matrix using clean Ottawa sand. Other clean matrices may be used in addition, for project specific requirements.

12.3.3 The LLOQ shall be verified annually on each instrument used for client sample analysis.

12.3.4 Recovery of each analyte must meet the laboratory established LCS recovery limits + 20%. (For example, if the LCS recovery limits are 70-130%, the LLOQ verification must meet recovery limits of 50-150%.) Once sufficient points have been generated, LLOQ based statistical limits may be used in place of limits based on LCS recovery.

NOTE: The lower recovery limit for the LLOQ can be no lower than 10%.

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, (*Training*), for the laboratory's training program.

13.0 Pollution Control

13.1. It is Eurofins's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Practices*, current revision. The following waste streams are produced when this method is carried out.

- Laboratory Generated Aqueous Waste (aqueous VOA vials – used and unused). This waste may have a pH of less than 2.0. These vials are collected in satellite accumulation. The vials are then transferred to the waste room. These vials are passed through a vial crusher and the liquid portion is separated from the solid portion. The solid is dumped into the municipal garbage. The liquid is pumped into the neutralization system where it is neutralized to a pH of 6 to 9 with sodium bicarbonate (Seidler Chemical SC-0219-25). When neutralization is complete, the material is transferred to the municipal sewer system**.**

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

- Methanol Preserved Samples/Returned Methanol Preservative - Methanol preserved sample vials are collected in satellite accumulation and then transferred to a 55 gallon open top steel waste drum in the waste room. This drum is then removed by a waste vendor for incineration.

  Teris Profile Number: 50016652
  Onyx Profile Number: 282493

15.0 References / Cross-References

15.2. .United States Environmental Protection Agency, “Method 8260D, Volatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS)”, Test Methods for Evaluating Solid Waste, SW846, Update VI, Revision 4, June 2018.

15.3. United States Environmental Protection Agency, “Method SW8000D: Determinative Chromatographic Separations”, Test Methods for Evaluating Solid Wastes, SW846, Laboratory Manual, Physical/Chemical Methods, Update V, Revision 4, October 2012.

15.4. U.S. EPA. 2003. "Method 5030C (SW-846): Purge-and-Trap for Aqueous Samples," Revision 3. Washington, DC.

15.5. U.S. EPA. 2002. "Method 5035A (SW-846): Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples," Draft Revision 1. Washington, DC.

15.6. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.7. Eurofins Document No. CW-E-M-001, Corporate Environmental Health and *Safety Manual,* current revision.

15.8. Eurofins Edison SOP Nos. ED-MSV-001*, Purge and Trap for Aqueous Samples, SW846 Method 5030*, current revision.

15.9. Eurofins Edison ED-MSV-002, *Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, SW846 Method 5035A,* current revision.

15.10. Eurofins Edison SOP No. ED-GCV-001, *Screening for Volatile Organics, Static Headspace with GC FID, SW846 Method 5021*, current revision.

15.11. Eurofins Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.12. Eurofins Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.13. Eurofins Edison SOP No. ED-GEN-008, *Standard Operating Procedure for Preparation, Purity and Storage of Reagents and Standards*, current revision

15.14. Eurofins Edison SOP No. ED-SPM-004, *Sample Storage & Handling Procedures for Mitigation of Sample and Laboratory Contamination,* current revision

15.15. Eurofins Edison Work Instruction No. EDS-WI-096, *8260C ICAL Procedure*, current revision.

15.16. Eurofins Edison SOP No. ED-GEN-022, *Training*, current revision.

15.17. Eurofins Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Practices*, current revision

15.18. Eurofins Edison Work Instruction Document No. EDS-WI-012, *Client Complaint/Corrective Action Form,* current revision.

15.19. Eurofins Corporate Quality Memorandum, CA-Q-QM-002, *GC/MS Tuning Policy*, current revision.

15.20. Eurofins Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision

16.0 Method Modifications:

16.1 Method 8260D requires the BFB tune standard to be analyzed once prior to an ICAL and not daily after that prior to sample analysis. The laboratory will analyze the BFB tune daily, prior to QC and sample analysis. The laboratory will use the

tighter criteria from Methods 8260B/8260C for tune evaluation, rather than the criteria suggested in Table 3 of Method 8260D.

17.0 Attachments

N/A

18.0 Revision History

- Revision 10, dated 08/09/2023
    - Throughout: added analyte 2-ethylhexyl acrylate
    - Sections 2.3 and 10.2.1: Screening clarified to indicate which samples are screened and samples that derive their dilutions from historical data.

- Revision 9, dated 10/17/2022
    - Updated throughout to Eurofins branding throughout the document.
    - Removed all references to 8260C
    - Section 8.3.1: added requirements for handing aqueous samples from chlorinated sources.
    - Section 9.2.2: updated throughout to detail requirements for Relative Standard Error (RSE) evaluation if ICAL.
    - Section 11.11: added formula for calculation of RSE.

- Revision 8, dated 07/16//2020
    - Updated throughout to include requirements of SW 8260D and 8000D.
    - Updated Table 1 with full current analyte list.
    - Add text to Section 1.1.6 detailing procedures for documenting method variations via NCMs.
    - Section 2.6: clarified text regrading lower than standard RLs.
    - Section 4.4: clarified text regarding trip blanks.
    - Section 7.1.1: revised source and details of organic free water.
    - Section 8.0: added handling and preservation details for various soil and aqueous sample types.
    - Section 9.1.1.4: Added that concentrations allowed in blanks (one half of RL), how blank concentration relates to sample concentration (<1/10) and some guidance on re-analysis when concentration exceeds criteria.
    - Section 9.1.3: added that CCV/LCS can be the same run.
    - Tune verifications as not required for daily CCV updated throughout.
    - Section 9.2.2.1.: Allowance for last calibration standard to be the start of 12-hour clock for samples analyzed after initial calibration. Calculations for verifying peak resolution
    - New Section 9.2.4.2 added: Calibration Point Read Back Criteria
    - Section 11.10: added formula for calculation of Percent Error.

- Section 12 (Method Performance) updated to include new MDL procedure and annual LLOQ procedure.
- Updated references in Section 15 as necessary.

- Revision 7, dated 04/01/2020:

  - Updated formatting and branding to Eurofins
  - Sec 7.2.8: Revised Expiration dates based on concentration level and corrected storage requirements.
  - Sec 8.1: Changed Storage Blanks storage period from 1 week to 1-2 weeks.

- Revision 6, dated 01/17/2018:

  - Revised Table 5 and 6: revised to add additional levels plus Benzene and chloroform and to updated concentration level of ICV to current level.

  - 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items.

  - Section 7.2.5: SIM IS/SS mix corrected to reflect lower concentration of IS mix .

  - Section 9.1.2.1.4: SIM MS/MSD preparation revised.

  - Section 9.1.3.2: SIM LCS/LCSD preparation revised.

  - Section 9.2.2.2: additional SIM levels added.

  - Section 9.2.3: SIM CCV level concentration revised to reflect lower concentration.

  - Section 10.2.5: New SIM analysis preparation narrative added

  - Section: 6.1: New instrumentation added.

  - Section: Section: 9.1.4.3: Revised to have any one surrogate out without the need for corrective action. This corrects previous narrative of one surrogate out of two.

- Revision 5, dated 12/11//2015:

  - Revised Table 5: new concentration of low standard (1,4-dioxane only).

- Revision 4, dated 12/08//2014:

  - Section 9.2.4.2.2: Table revised to reflect minimum RF of 0.050 for following compounds: acetone, 2-butanone, 4-methyl-2-pentanone, 2-hexanone.

  - Section 9.2.4.3: added statement ‘for poor performers the range is 50-150%’.

- Revision 3, dated 11/10/2014:

- Tables 1 and 7: added 1,2,4,5-Trimethylbenzene, 1,4-Diethylbenzene, Butadiene, 1,4-Difluorobenzene, 1-Chlorohexane, Freon 114, Freon 123a, Isooctane, 4-Ethyltoluene, t-Amyl Alcohol, Chlorofluoroethylene to list of target compounds and list of standard sources.

- Section 2.5: added chloroform, vinyl chloride and benzene to the list of SIM analytes addressed in this section.

- Section 2.6: revised the concentration of the low ketone standard to 2.5 ug/l.

- 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items. All standards prep tables revised to reflect current standard prep instructions.

- Section 8. Preservation by TSP and holding time is added.

- Section 9.1.2.1: updated source of standards used in various spiking solutions.

- Section 9.1.3: LCS/MS/MSD. Preparation tables now indicate using calibration mix and not the second source mix.

- Sections 9.1.4.3 and 9.1.1 : Revised to indicate that we are now spiking with 4 surrogates instead of the method required 3. One surrogate is now allowed to be out of limit criteria for either 1,2-Dichloroethane-d4 and Dibromofluoromethane.

- Section 9.2.2: Chloroform, Vinyl Chloride and Benzene added as SIM compounds.

- Section 9.2.4.2.3.1. A list of 'poor performing compounds' is added with a ICAL RSD criteria of 50%.

- Section 9.2.4.3: now specifies that up to 10% of the compounds are allowed to exceed the 70-130% ICV recovery criteria as long as their recoveries are within 65-135%..

- Section 9.2.4.4.1.6: Added the following to the first sentence: '…or 50%D for the poor performing compounds'.

- Section 10.1.1.2: updated masses/dwell time for Group 1 under SIM Parameters.

- Throughout document as appropriate: Replaced references to Target with references to CHROM

- Added Section 10.5.5: "The data processing service from Chrom queries LIMS for the sample processing parameters."

- Revision 2, dated 11/04/2013:

- Tables 1 and 7: added methyl acrylate, 1-methylnaphthalene and 2-methylnapthalene.

- Revision 1, dated 09/16/2011:

  - Tables 1 and 7: added cyclopentene, 2-chloro-1,3-butadiene, methacrylonitrile, propionitrile, ethyl methacrylate, 2-nitropropane, indan and isobutyl alcohol to list of target compounds and list of standards sources.

  - Section 7.2.1 and Table 2: Table in Section 7.2.1 and Table 2 updated to include complete list of standards currently in use as well as to update vendor catalog number for several items.

  - Table 3: Initial Calibration Standards Preparation: is now split into three tables to include aqueous low level analysis.

  - Table 5: added following footnote:
    Levels 1 and 2 respectively are prepared in 500ml and 100ml final volumes
    [1]This level is also used as the Continuing Calibration Verification.

- Revision 0, dated 02/15/2011: New

Table 2: Working Standards Preparation							
Target Compound Standard Name	**Lab Name**	**Vendor**	**Cat. #**	**Vol. Std. Added**	**Conc. of Stock Std.**	**Concentration of Standard**	**Final Vol/ Total vol of MeOH**
Gas Mix Hi	Gas (Hi)	Restek	567645	5ml mL	2000 ppm	500 ppm	20mL 15mL TV/M
Gas Mix Li	Gas (Li)	Restek	567645	500 uL	2000 ppm	50 ppm	20mL 19.5mL TV/M
8260Mix 1	Mix 1 (Hi)	Restek	567641 567646 567642 568022	2.5ml 2.5 ml 2.5 ml 2.5 ml	2000 ppm	500 ppm	10ml
8260 combined	Mix 1 (Li)	Restek	567641 567646 567642 568022 567643 568018 568713 568722 568723	1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml 1.0ml	2000 ppm	50 ppm	40ml 31ml TV/M
Acrolein	AC	Restek	82402	1.0ml	20000 ppm	500 ppm	40ml 39ml TV/M
8260 Mix 2	Mix 2 (Hi)	Restek	567643 568722 568019-fl 568713-fl	2.5ml 2.5 ml 2.5 ml 2.5 ml	2000 ppm	500 ppm	10mL
8260 Mix 3	Mix 3 (Hi)	Restek	568723 568021-fl	2.5ml 2.5ml	2000 ppm	500 ppm	10ml 5ml TV/M
1,4-Dioxane	1,4-Dioxane	Supelco	360481	483.6ul	Neat	50000 ppm	10ml/9.52TVM
1,4-Dioxane	1,4-Dioxane	Supelco	NA	100ul	50000 ppm	500 ppm	10ml/9.90TVM
Propenes*	Propenes	Supelco	21240202	NA	1000/2000 ppm	NA	NA
Propenes*	Propenes	Supelco	21240202	1ml	1000/2000 ppm	50 ppm (varied)	20ml/ 19ml
Gas SS	Gas SS	Restek	567645.sec	1ml	2000ppm	50 ppm	40ml 39ml/TV/M
8260 Mix 1 SIM	8260 Mix 1 SIM	Supelco	5-02111	50 ul	2000ppm	10 ppm	10ml 9.95 TV/M
1,4-Dioxane SIM	1,4-Dioxane	Supelco	NA	100 ul	50000 ppm	500 ppm	10ml/9.90TVM

Table 2: Working Standards Preparation

Target Compound Standard Name	Lab Name	Vendor	Cat. #	Vol. Std. Added	Conc. of Stock Std.	Concentration of Standard	Final Vol/ Total vol of MeOH
8260 SS	8260 SS	Restek	567641.sec 567646.sec 567642.sec 568022- sl 567643.sec 568019- sl 568713- sl 568722.sec 568723.sec 568021- sl	1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml 1 ml	2000 ppm	50 ppm	40 ml 30 ml TV/M
Acrolein SS	AC SS	Restek	568720.sec	1 ml	20000 ppm	500 ppm	40 ml 39 ml TV/M
Propenes SS	Propenes SS	Supelco		1 ml	1000/2000 ppm	50/100 ppm	40 ml 39 ml TV/M
8260Mix 1 SIM SS	SIM MIX1 SS	Supelco	5S-02111	50ul	2000 ppm	10 ppm	10ml 9.95 TV/M
Benzene/ Chloroform	Ben/chl	Absolute	70025/ 70076	100ul each	1000ppm	10ppm	10ml 9.90 TV/M
1,4-Dioxane (SS)	1,4-Dioxane	Absolute	70373	1ml	1000 ppm	500 ppm	2ml/1ml TV/M

Asterisk (*) indicates a custom standard mix.

Table 3: Initial Calibration Standards Preparation, Low Level Soil

Standard Solution	Final Volume Reagent Water (ml)	Volume of Standard Added to Reagent Water (ul)					
		1ppb *	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (50ppm)	5	-	-	2.0	5	-	-
	50	1.0	5.0			-	-
Gas Mix (500ppm)	5	-	-	-		2.0	5.0
		-	-	-			
Mix 1 (combined) (50ppm)	5	-	-	2.0	5	-	-
	50	1.0	5.0			-	-
Mix 1 (Hi) (500ppm)	5	-	-	-	-	2.0	5.0
		-	-	-	-	-	-
Freon Mix							
AC (500ppm)	5	-	-	3.0	4.0	5.0	6.0
	50	10	20			-	-
Mix 2 (Hi) (500ppm)	5	-	-	-	-	2.0	5.0
		-	-	-	-		
Mix 3 (500ppm)	5					2.0	5
Propenes (50ppm)	-	-	-	-	-	-	-
	50	10.0	20.0		-	-	-
Propenes (Hi)(500ppm)	5	-	-	2.0	5.0	20	50
	-	-	-	-	-	-	-

[1]**This level is also used as the Continuing Calibration Verification.**

Table 3a: Initial Calibration Standards Preparation, Aqueous (LOW LEVEL)

Standard Solution	Volume of Standard Added to Reagent Water (ul)						
	0.5ppb*	1ppb*	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (500ppm)	0.5	1	1	2	5	20	50
Mix 1 (Hi) (500ppm)	0.5	1	1	2	5	20	50
Mix 2 (Hi) (500ppm)	0.5	1	1	2	5	20	50
Mix 3 (varied)	0.5	1	1	2	5	20	50
AC (500ppm)	2	4	4	4	10	20	40
1,4-Dioxane (500ppm)	15	30	-	-	-	-	-
Freons mix	0.5	1	1	2	5	20	50
Propenes (1000/2000ppm)	0.5	0.5	0.5	1	2.5	10	25
Methanol Compensate	3000	2800	610	300	280	190	0
Final vol. (reagent water)	500ml	500 ml	100ml	50 ml	50ml	50ml	50ml

[1]This level is also used as the Continuing Calibration Verification.

Table 3b: Initial Calibration Standards Preparation, Aqueous

Standard Solution	Volume of Standard Added to Reagent Water (ul)					
	1.0ppb*	5ppb*	20ppb[1]	50ppb	200ppb	500ppb
Gas Mix (500ppm)	1	1	2	5	20	50
Mix 1 (Hi) (500ppm)	1	1	2	5	20	50
Mix 2 (Hi) (500ppm)	1	1	2	5	20	50
Mix 3 (varied)	1	1	2	5	20	50
AC (500ppm)	4	4	4	10	20	40
1,4-Dioxane (500ppm)	30	-	-	-	-	-
Freons Mix	1	1	2	5	20	50
Propenes (1000/2000ppm)	0.25	0.5	1	2.5	10	25
Methanol Compensate	2800	610	300	280	190	0
Final vol. (reagent water)	500 ml	100ml	50 ml	50ml	50ml	50ml

[1]This level is also used as the Continuing Calibration Verification.

Table 4 : ICV Standard Preparation, Low Level Soil

Standard Solution	Concentration	Volume of Standard Added to 5.0 ml of Reagent Water (ul)	Final Concentration (ug/L)
GAS SS (Separate lot)	50ppm	2	20
8260 SS (Separate lot)	50ppm (+varied)	2	20
AC SS (separate lot)	500ppm	3	300
Freon SS (Separate lot)	50ppm	2	20
Propenes SS(separate lot)	50ppm (varied)	2	20 (varied)

Table 4a: ICV Standard Preparation, Aqueous

Standard Solution	Concentration	Volume of Standard Added to 50 ml of Reagent Water (ul)	Final Concentration (ug/L)
GAS SS (Separate lot)	50ppm	20	20
8260 SS (Separate lot)	5000ppm (varied)	20	20
AC SS (separate lot)	500ppm	4	400
Freons SS (Separate lot)	50ppm	20	20
Propenes (second source)	50ppm (varied)	20	20 (varied)

Table 5: SIM Initial Calibration Standards Preparation

Standard Solutions	Volume Standard Solution Added to Reagent Water (Final Concentration)							
8260 Mix 1 SIM (10ppm)	1 ul (0.02 ppb)	2 ul (0.04 ppb)	1 ul (0.05 ppb)	1 ul (0.1 ppb)	1 ul (0.2 ppb)	2.5 ul (0.5 ppb)	5 ul (1 .0 ppb)	10 ul (2.0 ppb)
1,4-Dioxane (500ppm)	4 ul (0.4 ppb)	2 ul (1.0 ppb)	1 ul (5.0 ppb)	1 ul (10 ppb)	1 ul (20 ppb)	2.5 ul (30 ppb)	5 ul (40 ppb)	10 ul (50 ppb)
SIM (ben/chl) 10ppm	1 ul (0.02 ppb)	1 ul (0.02 ppb)	1 ul (0.05 ppb	1 ul (0.1 ppb)	1 ul (0.2 ppb)	2.5 ul (0.5 ppb)	5 ul (1.0 ppb)	10 ul (2.0 ppb)
Final Vol. (reagent water)	**500ml**	**500ml**	**200ml**	**100ml**	**50ml**	**50ml**	**50ml**	**50ml**

levels 1 and 2 are respectively prepared in 500ml and 100ml final volumes
[1]**This level is also used as the Continuing Calibration Verification.**

Table 6 : SIM ICV Standard Preparation

Standard Solution	Concentration	Volume of Standard Added to 200 ml of Reagent Water (ul)	Final Concentration (ug/L)
SIM MIX1 SS (Second source)	10ppm	1	0.05
1,4-Dioxane SS	50ppm	20	5

TABLE 7
Characteristic Ions of Volatile Organic Compounds

Parameter	Primary ion	Secondary ion
1,1,1-Trichloroethane	97	99,117,119
1,1,2,2-Tetrachloroethane	83	85,131,133,166
1,1,2-Trichloroethane	97	83,85,99,132,134
1,1-Dichloroethane	63	65,83,85,98,100
1,1-Dichloroethene	96	61,98
1,1-Dichloropropene	75	110. 77
1,2,3-Trichlorobenzene	180	182
1,2,3-Trichloropropane	110	75
1,2,4-Trichlorobenzene	180	182, 145
1,2,4-Trimethylbenzene	105	120
1,2-Dibromo-3-Chloropropane	75	155, 157
1,2-Dibromomethane	107	109
1,2-Dichloroethane	62	64,100,98
1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	65,114
1,2-Dichlorotrifluoroethene	67	117
1,2-Difluorotetrachloroethene	101	103, 167
1,3,5-Trimethylbenzene	105	120
1,3-Dichlorobenzene	146	148, 111
1,4-Dichlorobenzene	146	148, 111
1,4-Dioxane	88	58
1-Chloropropane	63	78
1-Methylnaphthalene	142	141
1-Propene	41	42
2,2-Dichloropropane	77	97

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
2,4,4-trimethyl-1-pentene	41	57, 97
2-Butanone	72	57
2-Chloroethyl vinyl ether	63	65, 106
2-Chloropropane	78	63
2-Chlorotoluene	91	126
2-Chloro-1,3-butadiene	88	53
2-Hexanone	43	58,100
2-Methylnaphthalene	142	141, 115
2-Nitropropane	39	42, 44
2-Octane	43	58
2-Octanol	45	55
4-Chlorotoluene	91	126
4-Methyl-2-Pentanone	43	58,100
Methacrylonitrile	67	41
Acetone	43	58
Acetonitrile	39	40, 41
Acrolein	56	55
Acrylonitrile	53	52
Allyl Alcohol	57	40, 39
Allyl Chloride	76	41
Amyl Acetate	43	70, 61
Benzene	78	--
Benzyl Chloride	91	126, 65
Bromobenzene	156	77, 158
Bromochloromethane	129	49, 130
Bromodichloromethane	83	85
Bromoform	173	171,175,
Bromomethane	94	96
Butyl Acetate	73	56, 43
Butyl Acrylate	73	56, 55
Butyl methacrylate	87	69
Camphene	93	121
Camphor	95	81
Carbon disulfide	76	78
Carbon tetrachloride	117	119,121
Chlorobenzene	112	114
Chloroethane	64	66
Chloroform	83	85
Chloromethane	50	52
Chlortrifluoroethene	116	118
cis-1,3-Dichloropropene	75	77

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
Cyclohexane	56	84, 69
Cyclopentene	67	68, 68, 53
Dibromochloromethane	129	208,206
Dibromomethane	93	95, 174
Dichlorodifluoromethane	85	87
Dimethylnaphthalene (total)	141	156, 155
Epichlorohydrin	57	62, 49
Ethanol	46	45
Ethyl Acetate	70	61, 43
Ethyl Acrylate	55	56
Ethyl Ether	59	74, 75
Ethylbenzene	106	91,
Ethyl methacrylate	69	41, 99
Freon TF	101	103, 151, 85
Hexachlorobutadiene	225	223
Hexane	56	57, 86
Indan	117	118, 58
Iodomethane (methyl iodide)	142	127
Isobutyl Alcohol (Isobutanol)	43	41, 42
Isoprene	67	53, 59
Isopropanol	45	59
Isopropyl Acetate	43	61, 87
Isopropyl Ether (DIPE)	45	87
Isopropylbenzene	105	120
Methyl Acetate	43	74
Methyl Acrylate	55	85, 42
Methyl cyclohexane	83	55, 98
Methyl Methacrylate	100	69
Methyl tert-butyl ether (MTBE)	73	57
Methylene chloride	84	49,51,86
Methylnaphthalene (total)	142	141, 115
Naphthalene	128	--
n-Butanol	56	41, 43
n-Butylbenzene	91	92, 134
n-Heptane	57	43, 71
n-Pentane	72	57
N-Propanol	60	59
n-Propylbenzene	91	120
P-Isopropyltoluene`	119	134, 91
Propyl Acetate	43	61, 73
Propionitrile	54	52, 54

TABLE 7 Characteristic Ions of Volatile Organic Compounds		
sec-Butylbenzene	105	134
Styrene	104	78,103
Tert-Amyl Methyl Ether	73	55, 87
Tert-butyl Alcohol	59	--
Tert-Butyl Ethyl Ether	59	87
Tert-Butylbenzene	119	91, 134
Tetrachloroethene	164	129,131,166
Tetrahydrofuran	42	72, 71
Toluene	92	91
Total Xylenes	106	91
trans,-1,3-Dichloropropene	75	77
Trans-1,4-dichloro-2-butene	53	75
Trichloroethene	130	95,97,132
Trichlorofluoromethane	101	103
Vinyl acetate	43	86
Dichlorofluoromethane	67	69
Chlorotrifluoroethene	116	118
1,2-tetrachlorodifluoroethane	101	103,167
1,2-Dichlorotrifluoroethane	67	117
Vinyl chloride	62	64
Isooctane	57	41, 56
1- Chlorohexane	91	93, 55, 56
1,2,4,5-Tetramethylbenzene	119	134, 91
4-EthylToluene	105	120, 77
Chlorotrifluoroethylene	66	116,118,85
Freon 114	85	87,135,137
t-Amyl Alcohol	59	55, 73, 43
1,4-Difluorobenzene	114	63
1,4-Diethylbenzene	119	105,134
Freon 123a	67	69, 117, 119
Butadiene	54	53, 39
4-Bromofluorobenzene (sur)	95	174,176
1,2-Dichloroethane-d4 (sur)	65	102, 104
Toluene-d8 (sur)	98	70,100
Fluorobenzene (istd)	96	77
Chlorobenzene-d5 (istd)	117	82,119
1,4-Dichlorobenzene-d4 (istd)	152	115,150

Title: Mercury Analysis for Water and Wastewater using EPA 245.1 and SW846 7470A; Mercury in Drinking Water using EPA 245.1; Leeman Mercury Analyzer (Cold Vapor Technique)

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

EPA Method 245.1 and SW846 Method 7470A are applicable to the determination of mercury in water matrices. Mercury may be found in water in both inorganic and organic forms. Organomercury compounds must first be broken down to respond to the cold vapor atomic absorption technique.

The typical detection limit using a 30 ml sample size is 0.2ug/L Hg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A digested sample is analyzed using cold vapor atomic absorption. 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 is measured as a function of mercury concentration.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 The addition of potassium persulfate during the digestion step can eliminate the possible interference from sulfide in the sample without affecting the recovery of inorganic mercury.

4.2 Copper may also be a potential interference although no effect has been observed for samples containing up to 10 mg/l total copper.

4.3 Samples that contain high levels of chloride have a potential to interfere due to a reaction that takes place during the oxidation step. During this step chloride is converted to free chlorine which absorbs light at 253.7 nm. The analyst must not allow the chlorine to be swept into the optical cell. The possibility of the chlorine interfering with the analysis can be minimized by using an excess of up to 7.5 ml hydroxylamine sulfate.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
a)Mercury b)(1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/M^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/M^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

6.1.1. Leeman Laboratories Inc. Hydra II AA Automated Hg Analyzer

6.1.2. Computer and Monitor with Leeman Envoy software

6.1.3. Block digestor (Environmental Express or SCP Science): Adjustable and capable of maintaining a temperature of 90 -95°C.

6.2 Supplies

6.2.1. 50 ml Hot Block Digestion Cups

6.2.2. 100 ml graduated cylinder

6.2.3. Eppendorf Pipettes and tips in various sizes

6.2.4. 100 ml volumetric flasks

6.2.5. 15 ml sample cups

6.2.6. 10 liter carboy container

6.2.7. Pump tubing:

- Sample, viton, blue tab
- Reductant, red tab
- Drain, blue tab
- Rinse, black tab

6.2.8. Drying Tube – Purchased pre-packed with Magnesium Perchlorate from Leeman Labs. Located prior to the optical cell.

6.2.9. Nitrogen or Argon supply - capable of producing 80 PSI.

7. Reagents and Standards

7.1. Reagents

Storage requirements: store at room temperature

Life of Reagent: - Concentrated acids: refer to manufacturer's instructions
- Laboratory prepared reagents and diluted acids: one year

Document prepared reagents in the Reagent module located in the TestAmerica Laboratory System (TALS).

7.1.1 Sulfuric acid - Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.2 Nitric acid - Concentrated (Trace Grade or Equivalent)

7.1.3 Hydrochloric acid-Concentrated (Trace Grade or Equivalent)

7.1.4 Potassium Permanganate (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.5 Sodium Chloride (analytical reagent grade); for stability information, refer to manufacturer's instructions.

7.1.6 Hydroxylamine Hydrochloride (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.7 Stannous Chloride (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.8 Potassium Persulfate (ACS Grade); for stability information, refer to manufacturer's instructions.

7.1.9 Deionized water - 18 megohm minimum

7.1.10 10% Hydrochloric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 800 ml of concentrated HCl and bring the final volume up to 8 liters with deionized water.

7.1.11 10% Stannous chloride solution - Add 50 g of $SnCl_2$ to 500 ml 10% HCl solution.

7.1.12 Sodium chloride/Hydroxylamine Hydrochloride solution - Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1 liter using deionized water.

7.1.13 Potassium Permanganate ($KMnO_4$) 5% solution w/v - Dissolve 100 g of $KMnO_4$ in deionized water and dilute to 2 liters using deionized water.

7.1.14 0.15% Nitric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 12mL of concentrated HNO_3 and bring the final volume up to 8 liters with deionized water.

7.1.15 Potassium Persulfate ($K_2O_8S_2$) 5% solution w/v: Dissolve 50 g of potassium persulfate in 1 liter of deionized water.

7.2 Standards

Storage requirements: all standards are stored at room temperature
Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – made fresh daily
Working standards – made fresh daily

(Note: expiration date must not go beyond the expiration date of the source stock).

Concentration: see Attachment 1 for example certificates of analysis (COA) for all of the standards mixes listed below. The COA lists the manufacturer's part number, certified concentration and shelf life.

Document standard preparation in TALS (see Section 11.5.2).

7.2.1 Stock Mercury Calibration (10 ppm Hg) - Purchase from SCP Science; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.2. Stock Mercury Quality Control Standard (10 ppm Hg) - Purchase from Inorganic Ventures; store at room temperature; for stability information, refer to manufacturer's instructions. This stock standard must be purchased from a second source vendor.

7.2.3. Intermediate Calibration Standard (DCAL-Int): Dilute 1 ml of Stock Mercury Calibration (Sec 7.2.1) to 100 ml with 0.15% HNO_3. The resulting solution will contain 100ppb Hg.

7.2.4. Intermediate Quality Control Standard (DQCS-Int): Dilute 1 ml of Stock Mercury Calibration Verification Standard (Sec 7.2.2) solution to 100 ml with 0.15% HNO_3. The resulting solution will contain 100ppb Hg.

7.2.5. Calibration Standard Preparation: Use six 50 ml hot block digestion cups to prepare the standards. Add small portion of 0.15% HNO_3 to each cup. Working in increasing order, spike the appropriate cup with 0.0, 0.06, 0.3, 0.6, 1.5, and 3.0 ml of working solution DCAL-Int (Sec 7.2.3). Bring to final volume of 30 ml and mix thoroughly. The corresponding concentrations are 0.0ppb, 0.2ppb, 1.0ppb, 2.0ppb, 5.0ppb, and 10.0ppb mercury respectively. For drinking water analysis, the 2.0ppb standard is also analyzed as the Maximum Contaminant Level (MCL) standard

7.2.6. Quality Control Standard (QCS) Preparation: Add a small portion of 0.15% HNO_3 to a 30 ml hot block digestion cup and spike 1.5 ml of DQCS-Int (Sec 7.2.4). Bring up to final volume and mix thoroughly. The resulting solution will contain 5.0 ppb of Hg.

7.2.7. Initial Calibration Verification (ICV) standard: Add a small portion of 0.15% HNO3 to a 30 ml hot block digestion cup and spike 1.5 ml of DQCS-Int (Sec 7.2.4). Bring up to final volume and mix thoroughly. The resulting solution will contain 5.0 ppb of Hg.

7.2.8. Continuing Calibration Verification (CCV) standard 5.0ppb: The 5.0 ppb

prepared from the calibration standards (Sec 7.2.5) is used as the Continuing Calibration Verification standard (CCV).

7.2.9. Maximum Contaminant Level (MCL) standard 2.0 ppb *for drinking water analysis:* The 2.0ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the Maximum Contaminant Level (MCL) standard.

7.2.10. Reporting limit check standard (CRI) 0.2 ppb: The 0.2ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the CRI standard. This standard is being analyzed as part of some client's special projects.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [2]	Reference
Waters	P. FP, G[1]	250 ml	HNO_3, pH < 2 prior to shipment; if not, acidify upon receipt in lab; Cool 4±2°C	28 Days	40 CFR Part 136.3

[1] Polyethylene, fluoropolymer, glass
[2] Inclusive of digestion and analysis
[3] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 16 hours for method 245.1 drinking water and 24 hours for non-potable water. Just prior to digestion or direct analysis, verify pH<2. If pH≥2, repeat steps (i.e., add acid, hold for 24hrs, verify ph<2).

9.0 Quality Control

9.1. Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	**245.1**: < RL or; <10%sample concentration (whichever is greater) **7470A**: < RL; **or;** < 5% of the reg limit; **or,** < 5% of the measured sample concentration (whichever is greater)
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	**245.1**: 85-115% **7470A**: 80-120%
Matrix Duplicate (DUP)[1]	1 in 20 or fewer samples	20% RPD
Matrix Spike (MS)[1]	**245.1**: 1, if 10 samples or less; 2 if 11-20 samples **7470A**: 1 in 20 or fewer samples	**245.1**: 70-130% **7470A**: 75-125%
Matrix Spike Duplicate (MSD)	When requested by client	**245.1**: 70-130% **7470A**: 75-125% **For both 245.1** & **7470A**: If MS and MSD are both ≥ 5X CRDL, then 20% RPD. If MS and MSD are less than the CRDL, the RPD is not calculated; otherwise ±CRDL.
Serial Dilution (SD) for 7470A	1 in 20 or fewer samples	±10%

[1] The sample for DUP and MS are randomly selected, unless specifically requested by a client. Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.

9.1.1 **Method Blank:** One laboratory method/preparation blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). Preparation blank is used to identify possible contamination during acid digestion. If the Mercury concentration in the MB is above the specified control limit, the batch must be prepared again and the samples reanalyzed.

- For 7470A, results must be less than RL, 5% of the regulatory limit for that analyte, or 5% of the measured concentration in the sample, whichever is greater.
- For 245.1, results must be less than RL or less than 10% of the determined Mercury concentration in the sample, whichever is greater.

9.1.2 **Laboratory Control Sample (LCS):** A laboratory control sample must be analyzed with each batch of samples digested. A blank is spiked with 0.1

ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. Results of the aqueous LCS must fall within ±15% of the true value for Method 245.1 and ±20% for Method 7470A.

9.1.3 **Matrix Duplicate (DUP):** A duplicate is analyzed for each batch of samples digested. If original sample and duplicate are both ≥ CRDL, then 20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.1.4 **Matrix Spike (MS):** A matrix spike is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.1 ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. A recovery of 70-130% for Method 245.1 and 75-125% for Method 7470A is required.

9.1.5 **Matrix Spike Duplicate (MSD):** When requested by the client, a matrix spike duplicate is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.1 ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. A recovery of 70-130% for Method 245.1 and 75-125% for Method 7470A is required. For both 245.1 & 7470A: if original sample and duplicate are both ≥ CRDL, then 20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.1.6 **Serial Dilution (SD):** For method 7470A, a five fold serial dilution must be performed on one sample per batch. The sample should contain a sufficiently high concentration; minimally a factor of 25 times above the estimated detection limit. Dilute the sample by a minimum of five fold (1+4) and reanalyze. Results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

9.2 Instrument QC

9.2.1. **Initial Calibration Verification (ICV):** Initial calibration is verified after calibration. The ICV solution should be prepared using a second source vendor; see section 7.2.7 for preparation instructions. Use a concentration of mercury at the midpoint of the calibration range (5.0ppb). The Cal standard containing 5ppb is analyzed as the ICV; see Sec 7.2.7 for preparation instructions. For 245.1, the results must not differ from the true value by more than 5%. For 7470A, the results must be within 10% of the true value. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.2. **Continuing Calibration Verification (CCV):** Calibration verification is performed after the calibration, after every 10 samples, and at the end of the run. The CCV solution should be prepared from the same CAL

standard as used to prepare the calibration solutions. Use a concentration of mercury at the midpoint of the calibration range (5.0ppb). The Cal standard containing 5ppb is analyzed as the CCV.

For 245.1 and 7470A, CCV must not differ from the true value by more than 10%. If it does, stop the analysis and recalibrate. Re-analyze the previous ten samples following the last good calibration verification.

9.2.3. **Initial and Continuing Calibration Blank (ICB/CCB):** ICB and CCB must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. For methods 245.1 and 7470A, the absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

9.2.4. **Maximum Contaminant Level (MCL):** For drinking water analysis, one MCL standard shall be analyzed per calibration. The 2.0ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the Maximum Contaminant Level (MCL) standard. The result must be within 50% of the true value. If it's outside of the acceptable limit, terminate the analysis, correct the problem, and recalibrate the instrument.

9.2.5. **Quality Control Standard (QCS):** The calibration is verified after calibration using a second source vendor; see Sec 7.2.6 for preparation instructions. For 245.1 and 7470A, the results must not differ from the true value by more than 10%. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.6. **Reporting limit check standard (CRI):** At the beginning of the analysis, verify the accuracy at the reporting limit (RL) by analyzing a solution at the RL level. RL check solution is analyzed to demonstrate that the mercury analyzer is capable of detecting the target analyte at the reporting limit (RL). Laboratory limits are 50-150% of the true value. This standard is being analyzed as part of some client's special projects.

10.0 Procedure

10.1 Sample Preparation

10.1.1 Filtration Procedure for Dissolved Mercury not filtered in the Field

10.1.1.1 The unpreserved sample must be filtered through a 0.45um filter unit as soon as practical after collection.
10.1.1.2 Collect the required volume of filtrate by using a 0.45um filter unit and a vacuum pump.
10.1.1.3 Acidify the filtrate with 1:1 HNO_3 to a pH of <2.
10.1.1.4 The method blank (MB) must be filtered and digested under the

same conditions as the lab filtered samples.

10.1.2 If sample is preserved in laboratory, hold sample for 16 hours for method 245.1 drinking water and 24 hours for wastewater following acidification. Verify and record if sample pH is < 2.0 after 16 or 24 hours (depending on the analysis- drinking water or wastewater) and prior to digestion/analysis. If pH is > 2.0, add HNO3, hold for 24 hours and verify pH.

10.1.3 Transfer 30 ml sample (DI water for MB and LCS) or standard, or an aliquot diluted to 30 ml, to an appropriately identified 50 ml hot block digestion cup. For QA samples, transfer 3 aliquots of 30 ml sample to three digestion cups labeled as SAMPLE, DUP and MS. Spike LCSW and MS with 0.1 µg of mercury (0.3 ml of DCAL-INT standard).

10.1.3.1. Due to insufficient sample volume or an unusual matrix (e.g., samples with potentially high interferences), it may be necessary to perform a pre-digestion dilution. For example, a 10X pre-digestion dilution can be performed by transferring 3 mL of sample to 50 mL hotblock cup. Add 27 ml of deionized water. Record 3 mL initial volume and 30 mL final volume in TALS (see Sec 11.5.2). Continue to section 10.1.3.

10.1.4 Add 1.5 ml concentrated H_2SO_4 and 0.75 ml concentrated HNO_3 mixing well after each addition.

10.1.5 Add 4.5 ml of potassium permanganate solution to each bottle. Mix well and let stand for 15 minutes (minimum); if the color is still present, then proceed to section 10.1.6; if the color has disappeared, then see section 10.1.5.1.

10.1.5.1 Discard the aliquot. Using a new 50mL hot block digestion cup, dilute the sample so that the final volume is 30mL. For example, for a 3X dilution: add 20mL of 0.15 Nitric Acid (sec 7.1.14), 10mL of sample, and mix. Record the initial and final volumes in TALS. Add 4.5mL of potassium permanganate as described in sec 10.1.5 and wait 15min. If purple color is still present, proceed to sec 10.1.6; if not, a larger dilution is necessary.

10.1.6 Add 2.4 ml potassium persulfate solution to each bottle.

10.1.7 Heat for 2 hours in a 95^{o} C hot block digestor. Remove from block digestor and cool.

10.1.8 Add 1.8 ml Sodium chloride - Hydroxylamine hydrochloride solution to reduce the excess permanganate. Mix well; solution should become colorless. If necessary additional Sodium chloride - Hydroxylamine HCl solution may be added.

10.1.9 Due to differing rates of evaporation, the final volume of the standards/samples maybe different. The standards and samples, including all QC, must be at the same final volume before analysis; if not, find the sample or standard with the highest final volume (e.g., 41 mL) and add deionized water to any sample or standard to bring them to the same final volume . Mix well. Wait at least 30 seconds after decolorization before analyzing.

10.2 Calibration

10.2.1. The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated according to the manufacturer's specifications and must contain at least four standards and a blank. The laboratory currently uses five standards and a blank. The correlation coefficient of the calibration curve must be ≥0.995. If it does not, the problem must be corrected, and the instrument must be recalibrated. Standard preparations must be documented in TALS (see Sec 11.5.2).

10.2.2. Prepare five calibration standards, blank and calibration verification standard as detailed in sections 7.2.5 and 7.2.6.

10.2.3. Calibration Curve Read-Back:

10.2.1.1 Low-Level Readback (at the RL) – evaluate the 0.2 ug/L calibration standard. The %RE (relative error) must be +/- 20% of the true value (see Sec 11.4). If %RE is outside of criteria limits, stop the analysis and recalibrate.

10.2.1.2 Mid- Level Readback – evaluate the 5.0 ug/L calibration standard (the mid-level calibration standard). The %RE (relative error) must be +/- 10% of the true value (see Sec 11.4). If %RE is outside of criteria limits, stop the analysis and recalibrate.

10.3 Sample Analysis

10.3.1 Following a sample digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data.

10.3.2 The following analytical run sequence is currently used:

Instrument Calibration (Blank and five standards)

ICV
ICB

CRI
QCS
MCL (for drinking water analysis)
CCV
CCB
10 Samples
CCV
CCB
Repeat until run is completed
CCV
CCB

10.3.3 Instrument Operation:

10.3.3.1. Turn on Computer, Monitor and the Hg analyzer (Hydra IIAA).

10.3.3.2. Plumbing the Reagent Lines:

10.3.3.2.1. One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic tab is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and click the clamp, lever up. The tab end of the tube should be located at the front of the pump head.

10.3.3.2.2. Reductant (Red): Connect tab end of tube to the reductant tubing that is connected to the reductant bottle and the other end to the mixing tee.

10.3.3.2.3. Sample (Blue): Connect tab end of tube to the autosampler probe and the other end to the mixing tee.

10.3.3.2.4. Drain (Blue): Connect the tab end of tube to the sample discharge tube connected on the Liquid/Gas separator and the other end to the waste line.

10.3.3.2.5. Rinse (Black): Connect tab end of tube to rinse tubing that is connected to the rinse bottle. Connect the other end to the rinse tubing leading to the rinse cup.

10.3.3.3. Preparation of Reagents:

10.3.3.3.1. Pour the 10% $SnCl_2$ solution into the reductant bottle.

10.3.3.3.2. Pour the 10% HCl solution into the rinse reservoir bottle.

10.3.3.4. Start the Program:

10.3.3.4.1. Click the Envoy icon on the computer desktop
10.3.3.4.2. Click Method, select 245.1_7470A, and select OK
10.3.3.4.3. On the main screen, click the StartUp icon. Wait 15 minutes before analyzing calibrating.
10.3.3.4.4. Click Sequence Tab on bottom of screen
10.3.3.4.5. Click Sequence on top of screen
10.3.3.4.6. Select Open, then select New
10.3.3.4.7. Using the Prep Batch Sheet and hand scanner, enter the sample barcodes into the Sample ID column. Include the Serial Dilution and all needed sample dilutions
10.3.3.4.8. Pour out the digested calibration standards and samples into the proper locations on the autosampler.
10.3.3.4.9. Click the Run Sequence icon to begin calibration and to run samples.

10.3.3.4.10. To shut down the instrument, click the Stop icon on the main screen.

11.0 Calculations / Data Reduction

11.1 Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2 Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Final results calculation in aqueous samples :

$$\text{Concentration = mg/ L} = \frac{C \times V1 \times D}{V2}$$

Where:
C= Element concentration from instrument (ppb)

V1= Final volume of sample digested (in liters)
D= Dilution performed on sample

V2= Initial volume of sample digested (in liters).

11.4. Relative Error (%RE):

% Relative Error = (100) (MC-TC)/TC

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.5. Data Processing:

11.5.1. All data is recorded directly in TALS' Analyst Desktop II program.

11.5.2. Record standard/sample preparations in the Analyst Desktop II program located in the TestAmerica Laboratory System (TALS) Reagent module. The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers, creation and expiration dates.

11.5.3. Record the following reagents and the volume used for sample preparation in the batch information page under "batch comments" in TALS Analyst Desktop II: concentrated sulfuric acid, concentrated nitric acid, potassium permanganate, potassium persulfate and sodium chloride-hydroxylamine hydrochloride. Record reagents in TALS by opening the prep batch, click on "edit" and then right click to choose "view batch information." Enter the information in the "batch comments" section.

11.5.4. Import Data to TALS

Click the Analysis tab

11.5.4.1. Select the analytical run that needs to be imported
11.5.4.2. Select Statistics
11.5.4.3. Click Load and select TALS Import, click OK
11.5.4.4. Click Report, click CSV File
11.5.4.5. Name the import file (e.g., batch name, today's date)
11.5.4.6. The newly created import file is in the Import Folder on the desktop Send to TALS Import Folder

11.5.5. Creating the Raw Data PDF:

11.5.5.1. Click the Analysis Tab, select Detailed

11.5.5.2. Click Load, select Use This To Make PDF

11.5.5.3. Click Report, Click Printer

11.5.5.4. Include the calibration curve graph:

11.5.5.4.1. Go to Methods Tab and click Calibration, click Print

11.5.5.5. Combine both documents using the PDF Creator program. The new document is located in the Documents folder on the C:\ drive. Add this document to the "Doc's" location in the analytical batch.

11.5.6. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch.

11.5.6.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.5.6.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.6.3. Acknowledge by filling in responses to all unacknowledged findings.

11.5.6.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.6.3.2. Fill in appropriate comments in the response box, then hit 'OK.'

11.5.6.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer

12. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Instrument Detection Limit:

The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined annually or if the instrument is adjusted in any way that may affect the IDL. For 245.1, the IDL is determined by multiplying the average of the standard deviations obtained from the analysis of 10 reagent blanks by 3. For 7470A, the IDL is determined by multiplying the average of the standard deviations obtained from the analysis of 7 reagent blanks by 3.14

12.3 Linear Dynamic Range (LDR)

The upper limit of the LDR must be established. It must be determined from a linear calibration prepared from a minimum of three different concentration standards, one of which is close to the upper limit of the linear range. The LDR should be determined by analyzing succeedingly higher standard concentrations of mercury until the observed analyte concentration is no more than 10% below the stated concentration of the standard. The determined LDR must be documented and kept on file. The LDR which may be used for the analysis of samples should be judged by the analyst from the resulting data. Determined sample analyte concentrations that are greater than 90% of the determined upper LDR limit must be diluted and reanalyzed. The LDR should be verified annually or whenever, in the judgement of the analyst, a change in analytical performance caused by either a change in instrument hardware or operating conditions would dictate they be redetermined.

12.4 Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.5 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

12.6 Lower Limit of Quantitation (LLOQ)

12.6.1. LLOQ verification: prior to analyzing samples under method 7470A, after any change that may affect the LLOQ, and quarterly, the LLOQ must be verified. The LLOQ is initially verified by the analysis of 7 replicate samples spiked at the LLOQ (0.2 ppb). The seven replicates must be digested prior to analysis. The LLOQ is verified when the mean recovery of the 7 replicates is +/-30% of the true value and the RSD < 20%.

12.6.2. LLOQ on-going verification: the LLOQ is re-verified quarterly by the analysis of a method blank (MB) and 1 blank sample (deionized water spiked at the LLOQ (0.2 ppb). The MB and LLOQ samples must be digested prior to analysis. The LLOQ is re-verified if the MB result is +/- 0.5 times the reporting limit for each analyte and when the recovery of the LLOQ sample is +/-30% of the .true value.

13. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out.

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

15. References / Cross-References

15.1. Determination of Mercury in Water by Cold Atomic Absorption Spectrometry, EMSL-Cincinnati, EPA/600/R-94/11, May 1994; Method 245.1 Revision 3.0.

15.2. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. Corporate Environmental Health and Safety Manual CW-E-M-001, most current revision.

15.7. Leeman Hydra II AA Operating Manual

16. Method Modifications:

Item	Method No.	Modification
Sample Preparation	SW 7470A	Stannous Chloride is automatically added via the instrument versus the manual addition of Stannous Chloride as stated in the method. This is an instrument manufacturer's improvement that will reduce error due to loss of Mercury.
Sample Preparation	SW 7470A EPA 245.1	The hotblock has replaced the hot-water bath for digestion. This modification has been made to reduce cross-contamination (the hotblock tubes are disposable).
Sample Preparation	SW 7470A EPA 245.1	The typical prep sample size is reduced to 30ml (previously 100ml). This modification was made to allow for limited available sample volumes. Reagent volumes were adjusted to maintain sample to reagent volume ratio
Sample Preparation	EPA 245.1	The Initial Calibration Verification (ICV) will be prepared from a secondary source (as required in Method SW7470A), rather than the calibration standard source. Method 7470A and 245.1 will be prepared and analyzed in the same batch which improves efficiency and reduces waste. CCV will be prepared using the same source as the calibration standards.
Sample Preparation	SW 7470A EPA 245.1	When KMnO4 is added to the sample and the purple color is not present in the sample in the 15 minute period, the aliquot is discarded and a dilution is preformed using a lesser amount of sample. The purple color is checked after 15 minutes and the reporting limit is adjusted to reflect the dilution.

17. Attachments

Attachment 1: Example Certificate of analysis (10 ppm Hg)

18. Revision History

- Revision 15, dated 20 September 2023
  - Sec 10.1.5: Revised to remove instructions for adding more KMnO4 to all samples and standards when purple color persist after 15 minutes of addition.
  - Sec 10.1.5.1: Added sample dilution instructions for a sample that doesn't maintain the purple color for 15 minutes.
  - Sec 16: Added method modification notation for samples that don't maintain the purple color after 15 minutes.

- Revision 14, dated 11 August 2022
  - Sec 12.6: Added the lower limit of quantitation (LLOQ), associated instructions and recovery limits.

- Revision 13, dated 13 September 2021
  - Sec 7.2.10 & 9.2.6: Revised to clarify use of CRI standard.

- Revision 12, dated 03 August 2020
  - Updated header with Eurofins logo.
  - Sec 6.1.1: Replaced Hydra AA with Hydra IIAA
  - Sec 6.1.2: Updated Hg software from WinHg to Envoy.
  - Sec 8.0: Revised the required sample volume from 500ml to 250ml.
  - Sec 9.1: Revised MB control limits for 245.1 and 7470A to <RL; added MSD to the QC table.
  - Sec 9.1.1: Updated the control criteria for 245.1 and 7470A from <MDL to <RL.
  - Sec 9.1.3: Updated the criteria for the matrix duplicate to reflect current laboratory practices.
  - Sec 9.1.5: Added MSD to the list of Sample QC.
  - Sec 9.2.2: Revised the CCV recovery limit for method 7470A to 10%.
  - Sec 10.2.3: Added calibration curve read-back criteria.
  - Sec 10.3.3.4: Updated instructions for the instrument/software operation.
  - Sec 11.4: Added Relative % error calculation.
  - Sec 11.5.2: Added reagent/standard information to document in TALS ADII.
  - Sec 11.5.3.: Added requirements to record reagent volumes in the batch information page of TALS ADII; subsequent sections adjusted accordingly.
  - Sec 11.5.4 & 11.5.5: Updated instructions for data import and raw data pdf generation.
  - Sec 11.5.6: Added Data Review Checker (DRC) instructions.
  - Sec 15.7 & 15.8: Deleted work instructions EDS-WI-007 (TestAmerica Edison Metals Data Review Checklist) and WI EDS-WI-125 (TestAmerica Edison Metals Initial-Calibration Data Review Mercury checklist), not applicable.

- Revision 11, dated 25 April 2018
  - Sec 8: Added footnote 3 to clarify instructions for metals sample preservation (verify pH <2.0 prior to digestion).
  - Sec 10.1.2: Added procedure for samples which pH are >2.0 and acid preservation is performed in laboratory.
  - Sec 12.3: Added procedure for the determination of linear dynamic range.

- Revision 10, dated 05 February 2018
  - Sec 7.2.10: Added CRI to the list of standards.
  - Sec 9.2.6: Added CRI to the list of instrument QC.
  - Sec 10.3.2: Revised the instrument sequence list to include CRI.

- Revision 9, dated 09 December 2015
  - Sec 7.2.7: Revised preparation procedure for ICV, standard solution should be obtained from a source different from the calibration standard. Removed reference to CCV.
  - Sec 7.2.8: Added CCV preparation procedure; standard solution same source as the calibration standard. Subsequent sections adjusted accordingly.
  - Sect 9.2.1: Revised - ICV solution should be prepared from a separate source.
  - Section 16: Added Method Modifications for using the secondary source when preparing the ICV standard for method EPA 245.1.

- Revision 8, dated 13 January 2014
  - Sec 7.1.8: Added the stock standard Potassium Persulfate to the list of reagents; subsequent sections adjusted accordingly.
  - Sect 7.1.15: Added preparation instructions for the 5% Potassium Persulfate solution.
  - Sec 7.1: Revised to include recording of reagent prep information in TALS and discontinue the use of Mercury Prep Logbook.
  - Sec 7.2.5 & 7.2.6: Revised the spiking amount to add when preparing calibration standards in 30 ml final volume; also replaced the 100 ml flasks with 50 ml digestion cups.
  - Sec 9.0: The matrix spike (MS) frequency for Method 245.1 has been revised to 1 per 10 samples to comply with the method.
  - Sec 11.4.4: Added the Mercury calibration checklist (EDS-WI-125).
  - Sec 15.8: Added WI# ED-WI-125 on the list of references; subsequent sections adjusted accordingly.
  - Sec 17: Updated the attached COA for the Hg standards.

- Revision 7, dated 02 December 2011
  - Sec 1.1: Revised detection limit to 0.2 ug/L Hg to reflect actual laboratory limits.
  - Sec 1 & 12: Revised the LQM reference for DOC and Test Methods and Method Validation to Section 19.
  - Sec 3: Revised LQM reference for the definitions.
  - Sec 5: Revised in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002, Writing a Standard Operating Procedure (SOP), most current revision.
  - Sec 7.1: Added requirements that all prepared reagents are recorded in TestAmerica Edison Mercury Std Prep Log.
  - Sec 7.2 & 10.2.1: Revised the documentation of standard preparation from logbook to TALS.
  - Sec 7.2.5: Replaced cal std 0.1 ug/L (Hg) with 0.2 ug/L.
  - Sec 9.1 and 9.1.1: Expanded MB control limit for 245.1 to reflect method's criteria.
  - Replaced PB (preparation blank) with MB (method blank) in various sections where applicable.

- Sec 10.1.1: Added procedure for filtering dissolved samples in the lab.
- Sec 10.1.2.1: Added procedure for pre-digestion dilutions.
- Sec 10.1.8: Added procedure for visually checking and adjusting final volumes for all standards and samples.
- Sec 10.3.3.11.7: Added procedure for post-digestion dilutions.

- Revision 6, dated 03 September 2009

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Combined SOP ED-MT-014 and ED-MT-015 with SOP ED-MT-017; retired SOP ED-MT-014 and ED-MT-015 at the effective date of this SOP.
  - Sec 6.1.3: Replaced water bath with block digestor.
  - Sec 6.2.1: Replaced 300 ml BOD bottles with 50 ml hot block digestion cups
  - Sec 6.2.3. Added 'Rinse Black' and changed Drain 'Black' to Drain 'Blue' in the list of Reagent lines
  - Sec 7.1: Added 0.15% HNO3 to list of reagents. Deleted Potassium Persulfate, Magnesium Perchlorate, 0.5N H2SO4 & 5% Potassium Persulfate solution to list of reagents; Reagents deleted are not applicable to this method.
  - Sec 7.2 Standards: Revised the Hg stock standard concentrations and preparation of standards.
  - Sec 7.1.9 & 7.1.13: Revised preparation procedure to reflect actual lab practices.
  - Sec 7.2.6: Renamed the second source standard 'Initial Calibration Verification standard' to 'Quality control standard.' QCS is added in the Instrument QC and analytical run sequence.
  - Section 8: Updated the section into Table format and have included the sample container, sample size requirements and method reference.
  - Changed the verification standard concentration of ICV (3ppb) to 5ppb; CCV concentration remains 5ppb.
  - Revised control limits to comply with Method 245.1
  - Sec 9.1.1: Clarified QC limits for the Preparation Blank
  - Sec 9.1.2: Revised the LCS limits for wastewater samples analyzed via Method 245.1 from ±20% to ±15%.
  - Sec 9.1.4: Revised the MS limits for wastewater samples analyzed via Method 245.1 from ±20% to ±30%.
  - Sec 9.1.5: Added Serial Dilution (L) in Sample QC
  - Sec 9.2.1: Clarified the recovery limits of ICV for method 245.1; % Rec limits were revised from 10% to 5% to reflect actual laboratory practices.
  - Sec 10.3.2: Added MCL in the analytical run sequence.
  - Sec 10.3.3.2: Added 'Rinse Black' and changed Drain 'Black' to Drain 'Blue' in the list of Reagent lines.
  - Sample size reduced from 100 ml to 30ml; preparation of the LCS (Sec 9.1.2) and MS (Sec 9.1.4) were revised to reflect this change in sample volume.
  - Sec 10.1: Adjusted reagent volume to maintain sample to reagent ratios.
  - Sec 11: Updated data processing in accordance with the new TALS.
  - Sec 15: Added applicable references.
  - Sec 16: Described the elimination of stannous chloride in the sample preparation and replacement of hot water bath as a method modification.

Attachment 1

SCP SCIENCE
Providing Innovative Solutions to Analytical Chemists

ACCREDITED

Certificate of Analysis

1.0 DESCRIPTION : ***Plasma*** **CAL – Custom Standard**

Catalogue Number : **141-110-11X**
Lot Number : **S130418021**
Matrix: 10.0% HNO3
Expiration Date : **July 2014**

2691783
ID: ME_Hg_1st_00013
Exp: 07/01/14 Post DLE
Mercury Calibration Stand

2.0 CERTIFIED VALUES AND ASSOCIATED UNCERTAINTY:

Method of Analysis: Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES)

Traceability: Applicable NIST Standard Reference Materials (see list below):

3101a	Al	3109a	Ca	3117a	Eu	3126a	Fe	3134	Mo	3142a	Pr	3151	Ag	3159	Th	3167a	Y
3102a	Sb	3110	Ce	3118a	Gd	3127a	La	3135a	Nd	3143	Re	3152a	Na	3160a	Tm	3168a	Zn
3103a	As	3111a	Cs	3119a	Ga	3128	Pb	3136	Ni	3144	Rh	3153a	Sr	3161a	Sn	3169	Zr
3104a	Ba	3112a	Cr	3120a	Ge	3129a	Li	3137	Nb	3145a	Rb	3154	S	3162a	Ti		
3105a	Be	3113	Co	3121	Au	3130a	Lu	3138	Pd	3147a	Sm	3155	Ta	3163	W		
3106	Bi	3114	Cu	3122	Hf	3131a	Mg	3139a	P	3148a	Sc	3156	Te	3164	U		
3107	B	3115a	Dy	3123a	Ho	3132	Mn	3140	Pt	3149	Se	3157a	Tb	3165	V		
3108	Cd	3116a	Er	3124a	In	3133	Hg	3141a	K	3150	Si	3158	Tl	3166a	Yb		

Certified Concentrations:

Hg 9.96 ± 0.06 µg/ml

Note: The uncertainty of the certified value has been calculated from applicable uncertainty contributors (u_i) such as the SRM inherited uncertainty, weighing and dilution errors and instrument variability. The combined uncertainty ($u_c = \sqrt{2u_i^2}$) has been multiplied by a coverage factor (k) of 2 to provide a 95% confidence interval.

3.0 REFERENCE VALUES:

Density: **1.051 g/ml** @ **20.9 °C**

4.0 APPROVAL AND DATE OF CERTIFICATION:

Certification Approval: Yaling Sui, Chemist Yaling Sui
Certification Date: **April 19, 2013**

INORGANIC VENTURES
300 Technology Drive
Christiansburg, VA 24073 • USA
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tel: 800.669.6799 • 540.585.3030
fax: 540.585.3012
info@inorganicventures.com

C.O.C. CERTIFICATE OF ANALYSIS

1.0 **INORGANIC VENTURES** is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."

ACCREDITED Testing Laboratory Certificate 863.01

ACCREDITED Reference Material Producer Certificate 863.02

2.0 **DESCRIPTION OF CRM** **10 µg/mL Mercury in 10% (v/v) HCL**

2035815
ID: ME_Hg_00019
Exp: 04/01/14 Prpd: CDC
Mercury Calibration Stand

Catalog Number:	MSHG-10PPM
Lot Number:	F2-HG02097
Starting Material:	Hg metal
Starting Material Purity (%):	100.0000
Starting Material Lot No:	R307HGA1
Matrix:	10% (v/v) HCL

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 9.990 ± 0.074 µg/mL

Certified Density: 1.026 g/mL (measured at 20 ± 1°C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

Certified Value $(\bar{x}) = \frac{\sum x_i}{n}$ ($\bar{x}$) = mean

x_i = individual results

n = number of measurements

Uncertainty (±) = $2\,[\,\Sigma(s_i)^2\,]^{1/2}$ 2 = the coverage factor.

$[\,\Sigma(s_i)^2\,]^{1/2}$ = The square root of the sum of the squares of the most common errors (where 's' stands for the standard deviation) from instrumental measurement, density, NIST SRM uncertainty, weighing, dilution to volume, homogeneity, long term stability and short term stability.

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed.,

Title: SW-846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS

Once printed, this is considered an uncontrolled document

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

This SOP describes the procedures used to determine the concentration of dissolved elements in water samples, various elements in groundwater, aqueous samples, industrial wastes, soils, sludges, sediments, and other solid wastes for which total (acid-soluble) elements are required by Inductively Coupled Plasma - Mass Spectrometry (ICP-MS) using EPA SW-846 Method 6020B.

The routine target analytes and reporting limits are as follows:

Table 1

Analyte List and Method Reporting Limits (RL)

Element	Digested Water RL	Soil RL
	Prep Method: 3005A &3010A (ug/L)	Prep Method: 3050B (mg/Kg)
Aluminum (Al)	40	20
Antimony (Sb)	2	1
Arsenic (As)	2	1
Boron (B)	80	40
Barium (Ba)	4	2
Beryllium (Be)	0.8	0.4
Cadmium (Cd)	2	1
Calcium (Ca)	500	100
Cobalt (Co)	4	2
Chromium (Cr)	4	2
Copper (Cu)	4	2
Iron (Fe)	120	60
Lead (Pb)	1.2	0.6
Magnesium (Mg)	200	100
Manganese (Mn)	8	4
Molybdenum (Mo)	4	2
Nickel (Ni)	4	2
Potassium (K)	200	100
Selenium (Se)	2.5	1.25
Silver (Ag)	2	0.40
Sodium (Na)	500	100
Strontium (Sr)	4	2
Thallium (Tl)	0.8	0.4
Tin (Sn)	16	8
Titanium (Ti)	4	2

Vanadium (V)	4	2
Zinc (Zn)	16	8

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Prior to analysis by ICP-MS, the sample must be solubilized or digested using the sample preparation method appropriate to the matrix. Sample digestates are aspirated and nebulized into a spray chamber. A stream of argon gas carries the sample aerosol through the innermost of three concentric tubes and injects it into the middle of the donut-shaped plasma. The sample elements are dissociated, atomized, and excited to a higher energy level. The ions that are produced are entrained in the plasma and introduced, by means of an interface, into a mass spectrometer. The ions are sorted according to their mass to charge ratios and quantified with a channel mass spectrometer.

For total analysis of aqueous samples containing undissolved material, aqueous samples must undergo digestion prior to analysis using SW846 Method 3005A or 3010A. For the determination of dissolved analytes in a filtered aqueous sample, the sample doesn't require digestion prior to analysis. The sample is ready for analysis after the addition of nitric acid. Soil samples are digested using Method 3050B. Refer to Eurofins Edison SOP Nos. ED-MTP-002, ED-MTP-003 and ED-MTP-005, for the digestion procedure (see Section 15.0 for complete references).

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Isobaric elemental interferences are caused by isotopes of different elements which form singly or doubly charged ions of the same nominal mass-to-charge ratio and which cannot be resolved by the mass spectrometer in use. Isobaric polyatomic/molecular ion interferences are caused by ions consisting of more than one atom which have the same nominal mass-to-charge ratio as the isotope of interest and which cannot be resolved by the mass spectrometer in use. Most isobaric interferences that could affect the ICP-MS analysis for elements in this SOP have been identified. These can be managed by the selection of an alternate isotope, the use of the collision/reaction cell (used for non-potable samples only), or by the use of elemental interference equations when the collision/reaction cell is

not in use.

4.2. Krypton affects the determination of both arsenic and selenium but can be greatly reduced with the use of high purity Krypton free argon. Krypton must be analyzed for every sample and standard.

4.3. Physical interferences are effects associated with the sample nebulization and transport processes as well as ion-transmission efficiencies. Changes in viscosity can cause significant inaccuracies, especially in samples containing high concentrations of dissolved solids or high acid concentrations. These changes in matrix can cause significant signal suppression or enhancement. Dissolved solids can deposit on nebulizer tips and interface cones (reducing the orifice size and the instrument's performance). Internal standards can be used to correct for physical interferences if they are carefully matched to the analyte so that both elements react similarly to the matrix changes.

4.4. Memory interferences can occur when analytes from a previous sample contribute to signals measured from subsequent samples. The memory effects can result from analyte deposition of sample on the sample tubing, joints, nebulizer, spray chamber, torch, and/or interface cones. Routine maintenance on the sample introduction system is necessary in order to minimize the memory interferences. The memory effects must be taken into account when setting up a suitable rinse times. The evaluation of a minimum of three replicate integrations will help to determine memory problems.

4.5. Contamination of the sample can occur when the preparation glassware and/or reagents contain the target elements. Reagent blanks (method blanks) must be analyzed as a check on contamination due to the sample digestion.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Agilent Agilent 7800, and Agilent 7900: inductively coupled plasma mass spectrometer with data system (microprocessor, monitor, printer) and autosampler. Mass Range 2-260 amu. Vacuum purged spectrophotometer with an axial plasma torch. Collision/Reaction Cell.

- Heat exchanger –PolyScience Model 6106T Recirculating Chiller or equivalent

- Autosampler –Agilent SPS 4 Autosampler

6.2. Supplies

- Reagent Water -18 megohm Reagent grade Type II water
- Volumetric Flasks (Class A): 50 mLs, 100 mLs, 500mls & 1000mls
- Eppendorf & Fisher Pipettes, varying volumes
- Polypropylene tubes

7.0. Reagents and Standards

7.1. Reagents

7.1.1. Concentrated Nitric Acid (HNO3) - Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions. The assay sheet of each lot of acid received into the lab must be reviewed to ensure the quality of the acid is sufficient for trace analysis of metals.

7.1.2. Concentrated Hydrochloric Acid (HCL) - Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions. The assay sheet of each lot of acid received into the lab must be reviewed to ensure the quality of the acid is sufficient for trace analysis of metals.

7.1.3. Argon supply - 99.9% (Liquid)

7.1.4. Helium and Hydrogen supply – 99.999% (Gas)

7.1.5. 18 megohm Reagent grade Type II water.

7.1.6. 5% HNO3 + 5% HCl: Add 1 liter of concentrated HNO3 and 1 liter of concentrated HCl to deionized water and bring to 20 liter volume with deionized water. Varying amounts of this reagent may be made (e.g., 10L) by proportionally adjusting the volume of acids used. Note: Always add acid to water. Record preparation in the TALS Reagent Module. Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature.

7.2. Standards

Calibration and spike solutions are prepared from either certified stock solutions or from stock solutions purchased from vendors. Certificates of analysis or purity must be received with all neat compounds or stock solutions. Certificate of analysis are filed in Metals manager's office.

Storage requirements: all standards are stored at room temperature

Shelf-life: Stock standards – refer to manufacturer's instructions
Working calibration standards – two weeks
Working Initial calibration verification standard – prepared fresh daily
Interference Check Standards (A&B) – weekly
Intermediate and all other standards- 6 months
(Note: expiration date must not go beyond the expiration date of the source stock).

Concentration: Final concentrations for the calibration standards are given in Attachment 1. All standards must be prepared in 5% HNO3 / 5% HCl (Sec. 7.1.6).

Standards must be prepared in accordance to the shelf life (Sec 7.2) "or sooner if needed or required." "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

All standards must be prepared in 5% HNO3 / 5% HCl (Sec. 7.1.6). Final concentrations for the calibration standards are given in Attachment 1. Varying amounts of these standards may be made by proportionally adjusting the volume of standards and reagent water used.
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7.2.1. Calibration Standards: purchased from CPI International. Refer to manufacturer's instructions for stability and storage information.

6020B RL Standard contains: 20 mg/L Ca, K, Mg, Na
12 mg/L Fe
8 mg/L B
4 mg/L Al
1.6 mg/L Sn, Zn
0.8 mg/L Mn
0.4 mg/L Ba, Co, Cr, Cu, Mo, Ni, Sr, Ti, V
0.25 mg/L Se
0.2 mg/L Ag, As, Cd, Sb,
0.12 mg/L Pb
0.08 mg/L Be, Tl

TA-CAL-1 contains: 1,000 mg/L Si

100 mg/L As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V

TA-CAL-2 contains: 1,000 mg/L Al, Ca, Fe, K, Mg, Na

Single Element Standard: Ag 1,000 mg/L
Single Element Standard: B 1,000 mg/L
Single Element Standard: Zn 1,000 mg/L

7.2.2. Working Calibration Standards:

CAL5: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 200ml flask. Add 0.4ml of TA-CAL-1 and 5ml of TA-CAL-2. Add 10 ml of MS-Cal-Int (see Sec 7.2.2.1). Bring to volume with 5% HNO3 / 5% HCl.

CAL4/CCV: Add 20ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 50ml polypropylene tube. Add 20ml of working Cal5 and mix.

CAL3: Add 15ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 50ml polypropylene tube. Add 5ml of working Cal5 and mix.

CAL2: Add 100ml of 5% HNO3 / 5% HCl to a clean flask. Add 10ml of 6020B RL Standard. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.6).

CAL1: Add 16ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 50ml polypropylene tube. Add 4ml of working Cal2 and mix).

7.2.2.1. MS-Cal-Int: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 200ml flask. Add 2ml each of B and Zn. Add 0.4ml of Ag. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.6).

Note: see Attachment 1 for final elemental concentrations.

7.2.3. Initial Calibration Verification Stock Standards: purchased from CPI International. Refer to manufacturer's instructions for stability and storage information. Standards must be from a different source than those used for the calibration standards.

TA-CAL1-SS contains: 1,000 mg/L Si
100 mg/L As, Ba, Be, Cd, Co, Cr, Cu, Li, Mn, Mo, Ni, Pb, Sb, Se, Si, Sn, Sr, Ti, Tl, V

TA-CAL2-SS contains: 2,000 mg/L Al, Ca, Fe, K, Mg, Na

2nd Source Single Element Standard: Ag 1,000 mg/L
2nd Source Single Element Standard: B 1,000 mg/L
2nd Source Single Element Standard: Zn 1,000 mg/L

7.2.4. ICV Working Standard: The ICV Working Standard must be at a concentration other than the concentrations used for the CCV working standard. Add 10ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 50ml polypropylene tube. Add 10ml of working Interm_ICV (Sec 7.2.4.2) and mix. The final concentrations of the various elements are listed in Attachment 1. This standard is prepared fresh daily and stored at room temperature.

7.2.4.1. MS-ICV-Int: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 200ml flask. Add 2ml each of 2nd Source: B & Zn. Add 0.4ml of 2nd Source: Ag. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.6).

7.2.4.2. Interm ICV: Add 100ml of 5% HNO3 / 5% HCl (Sec 7.1.7) to a clean 200ml flask. Add 0.32ml of TA-CAL-1-SS and 4ml of TA-CAL-2-SS. Add 8 ml of MS-ICV-Int (see Sec 7.2.4.2.1). Bring to volume with 5% HNO3 / 5% HCl.

7.2.5. Continuing Calibration Verification standard: see CAL4/CCV (Sec. 7.2.2).

7.2.6. Internal Standards: The following elements are used as the internal standards: Bi, In, Li6, Sc, Tb, and Ge.

- Internal Standard Multi-Element Mix 4 purchased from Agilent Technologies. Contains: 50 mg/L each: ^{6}Li, Sc; 25 mg/L each: Ge, Te; 10 mg/L each: Bi, In, Tb. Refer to manufacturer's instructions for stability and storage information.
- Triton X-100 (Standard Stock Solution: Non-ionic Surfactant) purchased from Fisher Scientific, p/n BP151-100. Refer to manufacturer's instructions for stability and storage information. According to the ICPMS manufacturer (Agilent Technologies), Triton-X 100 will reduce the %RSD of the triplicate analysis.

Prepare a working internal standard solution in a 1000 ml volumetric flask: Add 5 ml of Internal Standard Multi-Element Mix 4 and 0.1 ml of Triton X-100. Bring to volume with 5% HNO3 / 5% HCl.

The following are the recommended analysis masses and internal standards:

Element	IS
Be9	Li6
B11	Li6
Na23	Sc45
Mg24	Sc45
Al27	Sc45
K39	Sc45
Ca40	Sc45
Ti47	Sc45
V51	Sc45
Cr52	Sc45
Mn55	Sc45
Fe56	Sc45
Co59	Sc45
Ni60	Sc45
Cu63	Sc45
Zn 66	Sc45
As75	Sc45
Se78	Sc45
Sr88	In115
Kr83**	(none)
Mo95	In115
Ag107	In115
Cd111	In115
Sn118	In115
Sb121	In115
Ba137	Tb159
Tl205	Bi209
Pb 208*	Bi209

*Pb 208 = Pb 208 + Pb 207 + Pb 206
**Kr83 is used for monitoring Krpyton levels only.

Different masses and internal standards may be utilized as matrix issues deem necessary. Samples are analyzed using collision/reaction cell technology (helium/hydrogen gas modes). Typical Collision/Reaction Cell usage: Calcium, Iron, and Selenium are analyzed in the Hydrogen reaction mode; Beryllium, Boron and Titanium are analyzed in the No Gas mode; all other elements are analyzed in the helium collision mode.

Tune check criteria:

Instrument tunes must be performed daily, before calibration. A solution at ~10ppb of Be, Mg, Co, In, and Pb is analyzed, and the precision, mass calibration, and resolution are checked. Resolution is checked by analyzing Magnesium isotopes 24, 25, 26 and Lead isotopes 206, 207, 208.

The following limits are used to evaluate mass calibration, resolution, and instrument stability:

The following limits are used to evaluate the tune:

Mass calibration: +/-0.1 amu
Resolution check: <0.9 amu at 10% peak height
Stability (5reps): <5%

7.2.7. Spectral Interference Check (SIC) Solutions: A working stock solution may be purchased for the ICSA solution. The stock solution which is purchased from CPI International contains the following elemental concentrations:

TA-ICPMS-ICSA contains: 10000 mg/L Chloride; 2000 mg/L Carbon; 1000 mg/L ea: Al, Ca, Fe, K, Mg, Na, P, S; 20 mg/L ea: Mo, Ti

7.2.8. Working SIC Standards (ICSA):

ICSA (6020B): Add 50ml of 5% HNO3 / 5% HCl (Sec 7.1.6) to a clean 100ml flask. Add 10ml of TA-ICPMS-ICSA stock solution. Bring to volume with 5% HNO3 / 5% HCl (Sec 7.1.6). This standard is made fresh weekly; store at room temperature. Refer to Attachment 2 for final elemental concentrations.

7.2.9. ICP-MS Matrix Spiking Solution, ICPMS LCS/SPK: purchased from High-Purity Standards. Refer to manufacturer's instructions for stability and storage information. Refer to attachment 1 for final elemental concentrations. Stock solution contains the following elemental concentrations:

Element	Conc. Of Stock (mg/L)
Aluminum (Al)	500
Antimony (Sb)	5
Arsenic (As)	10
Barium (Ba)	10
Beryllium (Be)	5
Boron (B)	100

Element	Conc. Of Stock (mg/L)
Cadmium (Cd)	5
Calcium (Ca)	500
Chromium (Cr)	10
Cobalt (Co)	5
Copper (Cu)	10
Iron (Fe)	500
Lead (Pb)	5
Magnesium(Mg)	500
Manganese (Mn)	50
Molybdenum (Mo)	10
Nickel (Ni)	10
Potassium (K)	500
Selenium (Se)	10
Silver (Ag)	5
Sodium (Na)	500
Strontium (Sr)	10
Thallium (Tl)	4
Tin (Sn)	10
Titanium (Ti)	10
Vanadium (V)	10
Zinc (Zn)	50

7.2.10. EPA Tune Check

7.2.10.1. EPA Tune Check Stock Standard: 2008TS purchased from Inorganic Ventures. Refer to manufacturer's instructions for stability and storage information. Contains: 10 ug/mL each: Be, Co, In, Mg, Pb.

7.2.10.2. Working EPA Tune Check Standard: add 100ml of 5% HNO3 / 5% HCl to a clean 1000ml flask. Add 1 mL of 2008TS. Bring to volume with 5% HNO3 / 5% HCl . This solution contains 10 ug/L of Be, Co, In, Mg and Pb.

8.0. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Metals Waters	Polyethylene or Glass	250 mLs	HNO_3 to pH < 2 prior to shipment; if not, acidify upon receipt in lab [2,3]	6 months	40 CFR Part 136.3
Soils	8 oz or 16 oz.Polyethylene or Glass	10 grams	Cool 4 ± 2°C	6 months	SW846 Method 6020B

[1] Inclusive of digestion and analysis.
[2] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 24 hours. Just prior to digestion or direct analysis, verify pH<2. If pH≥2, repeat steps (i.e., add acid, hold for 24hrs, verify ph<2).
[3] Aqueous samples may be stored at room temperature.

Note: Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered; and NCM added that dissolved samples were not handled in accordance with 40 CFR Part 136 Table II. Properly filtered and nitric preserved samples requiring analysis for dissolved analytes don't require digestion prior to analysis.

Leachates are transferred to a plastic container after the applicable leachate extraction procedure. The sample is preserved with HNO3 to a pH<2. (If a matrix spike is necessary, then an aliquot is spiked prior to acid preservation). Leachate samples must be analyzed within 6 months of the extraction.

9.0. Quality Control

Note: If a batch of samples requires digestion, then the relating QC samples must be carried through the entire digestion process.

9.1. Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Control	Frequency	Control Limit
Method blank	One per batch of twenty samples or less	<1/2 the RL; or project specific requirements; or <10% of the measured concentration in the sample

Quality Control	Frequency	Control Limit
Lab Control Sample Water (LCSW)	One per batch of twenty samples or less	80-120%
Matrix Duplicate	One in 20 or fewer samples	If original sample and dup are both ≥ 5X CRQL, then ≤ 20% RPD. If original sample and duplicate are less than the CRQL, the RPD is not calculated; otherwise ±CRQL.
Matrix Spike[1]	One per batch of twenty samples or less	75-125%
Matrix Spike Duplicate[1]	Whenever the client requests	75-125%; If original sample and dup are both ≥ 5X CRQL, then ≤ 20% RPD. If original sample and duplicate are less than the CRQL, the RPD is not calculated; otherwise ±CRQL.
Post Digestion Spike	One per batch of twenty samples or less	See Section 9.1.7
Laboratory Control Sample Soil Reference Material (LCSSRM)	1 in 20 or fewer samples	Vendor's certified limits
Serial Dilution (1/5 dilution)	One per batch of twenty samples or less	See Section 9.1.6

[1] The sample for MS/MSD is randomly selected, unless specifically requested by a client.

9.1.1. Method Blank (MB): One laboratory method blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). The method blank is used to identify possible contamination during acid digestion. Results must be less than the highest of: (i) ½ the RL, (ii)project specific requirements, or (iii)10% of the measured concentration of the sample. If any analyte concentration in the blank is above the control limit, the batch must be prepared again for the element in question and the samples reanalyzed.

9.1.2. Laboratory Control Sample Water (LCS): A laboratory control sample must be analyzed with each group of samples digested. Refer to Water Matrix Spike (MS) concentration in Attachment 1 for final elemental concentrations. Results must be within the acceptable control limits, if not, all samples prepared in association with the LCS must be redigested and reanalyzed.

- Water samples - prepare the LCS by adding 0.25 ml of ICP-MS LCS/SPK (Sec. 7.2.10) in 50 ml deionized water. Results must be within ± 20% of the true value.

- Soil samples - LCS for solid matrices (LCSSRM) is obtained from a vendor supplied solid matrix and is carried through the same preparation procedure as the samples. The results of the solid LCS must fall within the 'QC Performance acceptance limits' of the reference material used for that sample. If not, all samples prepared in association with the LCS must be redigested and reanalyzed.

9.1.3. Matrix Duplicate: A duplicate is analyzed for each batch of samples. If original sample and duplicate are both ≥ CRDL, then ≤20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL.

9.1.4. Matrix Spike (MS): A matrix spike is prepared and analyzed for each batch of samples digested. A recovery of 75-125% is required (an exception to this occurs if the sample concentration exceeds the spike concentration by a factor of four or more). If the recovery is not within specified limits, a post digestion spike is required (see Sec 7.2.9). See Attachment 1 for final Matrix Spike concentrations for water and soil.

9.1.4.1. For aqueous MS sample, spike 0.25 ml of ICP-MS LCS/SPK (Sec 7.2.9) into 50 ml sample.

9.1.4.2. For soil MS sample, spike 1.0 ml of ICP-MS LCS/SPK into 1 gram of sample.

9.1.5. Matrix Spike Duplicate (MSD): A matrix spike duplicate is analyzed when requested by the client. Spike the QC sample the same way as the Matrix Spike (MS) sample. If original sample and duplicate are both ≥ CRDL, then ≤20% RPD. If original sample and duplicate are less than the CRDL, the RPD is not calculated; otherwise, ±CRDL. A recovery of 75-125% is required (an exception to this occurs if the sample concentration exceeds the spike concentration by a factor of four or more). If the recovery is not within specified limits, a post digestion spike is required (see Sec 9.1.7). See Attachment 1 for final Matrix Spike concentrations for water and soil.

9.1.6. Serial Dilution QC Check: A 1/5 dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present.

9.1.6.1. Select a sample and dilute the digestate by a factor of 5 (DF=5).

9.1.6.2. Analyze the dilution using the same procedures used for the un-diluted aliquot.

9.1.6.3. Compare the results of the diluted and un-diluted aliquots of sample digestate.

9.1.6.4. If the analyte concentration is minimally a factor of 25 above the RL (Reporting Limit), then the results of the dilution should be within ±20% of the result of the undiluted sample (original sample). If not, then a chemical or physical interference effect should be suspected.

9.1.7. Post-Digestion Spike: To check for possible matrix interference, analyze a post digestion spike on a representative sample (minimum of 1 per batch).

9.1.7.1 Transfer 10mL of a digestate, preferably the same sample that was selected for the matrix spike, to a suitable vial.

9.1.7.2 Spike the sample with 0.01 ml of ICP-MS LCS/SPK (Sec 7.2.9). Analyze the spiked aliquot and an un-spiked aliquot (the un-spiked may have been analyzed previously and does not need to be reanalyzed). The final concentration of the post digestion spike is as follows:

Element	Post spike concentration (ug/L)
Aluminum (Al)	500
Antimony (Sb)	5
Arsenic (As)	10
Barium (Ba)	10
Beryllium (Be)	5
Boron (B)	100
Cadmium (Cd)	5
Calcium (Ca)	500
Chromium (Cr)	10
Cobalt (Co)	5
Copper (Cu)	10
Iron (Fe)	500
Lead (Pb)	5
Magnesium(Mg)	500
Manganese (Mn)	50
Molybdenum (Mo)	10
Nickel (Ni)	10
Potassium (K)	500
Selenium (Se)	10
Silver (Ag)	5

Element	Post spike concentration (ug/L)
Sodium (Na)	500
Strontium (Sr)	10
Thallium (Tl)	4
Tin (Sn)	10
Titanium (Ti)	10
Vanadium (V)	10
Zinc (Zn)	50

9.1.7.3 Calculate the percent recovery of the post digestion spike as follows:

$$\%REC = \frac{Cps - Cs}{C2} \otimes 100$$

Where: Cps = concentration of post digestion spike (ug/L)
Cs = concentration of un-spiked sample (ug/L)
C2 = concentration of spike (ug/L)

9.1.7.4 Recovery limits for post digestion spikes for Method 6020B are 75-125%; If both the MS and post digestion spike fail, then matrix effects are confirmed.

9.2. Instrument QC

Table 2 below describes the frequency, criteria, and corrective actions for the calibration and Quality control samples.

Table 2: Calibration, Quality control and Corrective Action Summary

QC Item	Frequency	Criteria	Corrective Action
Tune validation-Mass Calibration	Daily	Within 0.1amu from mass unit	Terminate analysis, fix problem and repeat
Tune validation-Resolution Check	Daily	<0.9 amu full width at 10% peak height	Terminate analysis, fix problem and repeat
Instrument Stability Check	Daily	<5% RSD for five replicates	Terminate analysis, fix problem and repeat
Initial Calibration: Multi-point-minimum 5 stds and 1 blank	Daily	Correlation ≥0.995	Recalibrate
Initial Calibration Verification Standard (ICV)	At the beginning of the analysis	+/- 10%	Recalibrate
Continuing Calibration	At the beginning and	Within +/- 10% of the	Fix the problem and

QC Item	Frequency	Criteria	Corrective Action
Verification Standard (CCV)	end of the analysis, and every 10 samples	true value	reanalyze the previous 10 samples.
Initial Calibration Blank (ICB)	After ICV	must be less than ½ the LLOQ (Lower limit of Quantitation)	Terminate the analysis, correct the problem and reanalyze the previous 10 samples
Continuing Calibration Blank (CCB)	After every CCV	Less than the LLOQ	
Interference check standards (ICSA)	At the beginning of an analysis and after every 12 hours of analysis	+/-20% of the true value or +/-2 times the LLOQ, whichever is greater	If an element is consistently out of the +/-20% or +/-2 times the LLOQ, the problem should be investigated.
Internal Standards	Analyzed with every standard and sample	30-140%	Standard-terminate the analysis and recalibrate Sample- a 5X dilution is required (The analyst should check the IS response for the bracketing CCV/CCBs to see if there is a similar response to the sample. Recalibration may be required.)
Linear Range Check (LRC)	After calibration	90-110%	If LRC is not within 90-110% recovery, then the linear range is the highest point in the calibration

9.2.1. Initial Calibration Verification (ICV): Analyze an initial calibration verification solution at the beginning of the run. ICV solution must have the same acid matrix as the calibration standard and it must come from a source other than the calibration standard source. The final concentrations of the various elements are listed in Attachment 1. The results for the target elements in the initial calibration verification (ICV) must be within +/-10% of the true value. If results are outside of the specified limits, terminate the analysis, correct the problem and recalibrate the instrument. See Sec 7.2.4 for the ICV standard preparation instructions.

9.2.2. Continuing Calibration Verification (CCV): The calibration of the ICP-MS must be verified every 10 samples and at the end of the analysis run by analyzing the QC Check Solutions (CCV). The same solution used for the calibration standard 3 is used for the CCV standard. The concentration of

the CCV standard must be at or near mid-range levels of the calibration. The final concentrations of the various elements are listed in Attachment 1. The results for the target compounds must be within +/-10% of the true value. If results are outside of the specified limits, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the samples following the last good CCV. See CAL3/CCV of Sec 7.2.2 for the preparation instructions.

9.2.3. Initial and Continuing Calibration Blank (ICB/CCB):

9.2.3..1. Initial Calibration Blank (ICB) must be analyzed after the calibration curve. The value of the initial calibration verification blank must not exceed ½ LLOQ/reporting limit (RL). If it does, terminate the analysis, correct the problem, and recalibrate. The initial calibration verification blank is the same blank solution as used for the calibration blank.

9.2.3..2. Continuing Calibration Blank (CCB) must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. The result of the calibration verification blank must not exceed the LLOQ/reporting limit (RL). If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

9.2.4. Spectral Interference Check (SIC) Sample (ICSA): Verify the inter-element and background corrections by analyzing the interference check solution (ICSA) at the beginning of the analysis run and after every 12 hours of analysis. The ICSA is analyzed in order to demonstrate that proper corrections are being utilized for known interferences. Analyst must evaluate this solution in order to detect trends that require corrective actions. Pay particular attention to false positives and false negatives for elements not present in the interference check solution. Results should fall within the control limit of +/-20% of the true value or +/-2 times the LLOQ of the true value, whichever is greater. See Attachment 2 for list of elements and the corresponding concentrations in ppb. See Sec 7.2.8 for preparation instructions.

9.2.5. Linear Range Standard (LRC): the LRC standards are prepared from single element stock standards of each metal obtained from a commercial source. The LRC's are prepared by taking the appropriate volume of each stock and adding it to a 500 mL volumetric flask partially filled with 5% HNO3 / 5% HCl and diluted to the mark after all elements have been

added. The tables below lists the volume of each stock solution of each metal, volume used and final concentrations:

Linear Range Check (LRC) - A			
Elements	Stock Conc (mg/L)	Volume of stock (mL)	Final Conc (mg/L)
Tl	10000	0.05	1
Be, Co, Mo	10000	0.1	2
As, Cd,Sr, V	10000	0.25	5
Ba, Cr, Pb, Cu, Mn, Ni, Zn	10000	1	20

Linear Range Check (LRC) - B			
Elements	Stock Conc (mg/L)	Volume of stock (mL)	Final Conc (mg/L)
Al, Fe, Mg, Na, K	10000	25	500

Linear Range Check (LRC) - C			
Elements	Stock Conc (mg/L)	Volume of stock (mL)	Final Conc (mg/L)
Ag	1000	0.5	1
B	1000	1	2
Sb	10000	0.05	1
Sn	10000	0.1	2
Se	10000	0.15	3
Ti	10000	0.5	10
Ca	10000	50	1000

The LRC standards are analyed after each calibration. If the results fall within the control limit of +/-10% of the true value, then the linear range at that concentration has been verified; if not, the linear range is the highest point in the calibration.

9.3. Instrument Performance Criteria

Prior to the analysis of any samples, the following must be performed.

9.3.1. Instrument Detection Limits (IDLs): The IDL for each analyte must be determined for each wavelength used on each instrument.

9.3.1.1. The IDLs for each analyte must be determined for each prep

method. The IDL is determined by analyzing 10 replicate analysis of reagent blank solution (5% HNO3 / 5% HCl). Analyze the ten blanks and calculate the standard deviation and the mean (use zero for the mean if the mean is negative). The IDL = mean + (3 x Std Dev). The IDL must be determined when the instrument is initially set up, when the instrument is adjusted in any way that may affect the IDL (e.g., detector replacement), and annually.

10.0. Procedure

10.1. Sample Preparation

10.1.1. For samples to be analyzed for total analytes by Method 6020B, follow digestion procedure in Eurofins SOP No. ED-MTP-002 (Method 3005A) or ED-MTP-003 (Method 3010A) for waters and ED-MTP-005 (Method 3050B) for soils.

10.2. Calibration

10.2.1. Turn the ICP-MS on and initiate the tune screen. Start the tune screen to allow the instrument to become thermally stable before beginning to analyze the calibration standards. While the instrument is warming up, aspirate the interference check solution (or similar solution) to precondition the cones.

10.2.2. Check the tune parameters for the proper sensitivities, precision, oxides, and double charged values following the instrument manufacturer's recommendations. Refer to sections *Preparations Before Analysis*, *Plasma ON/Executing a Startup*, and *Appendix (Recommended Values for Tuning Parameters)* of the MassHunter Workstation User Guide (*Agilent Technologies 7800/7900, p/n G7201-90403).*

10.2.3. Prior to calibration, run an EPA Tune check solution. Refer to Sec. 7.2.6 for the Tune check criteria.

10.2.4. Using method *BM New Method.icpms.template* (program in the Mass Hunter Workstation software containing calibration standards, check tables, etc.), calibrate the instrument using a blank and five standards. Refer to the section *Creating a Method* in the instrument manufacturer's instructions *Agilent ICP-MS (7800/7900) MassHunter Workstation User Guide (p/n G7201-90403)*.

10.2.5. Internal standards are added to the calibration standards, blank and

samples in-stream by the use of T-fitting.

10.2.6. Analyze the calibration standards and calibrate the ICP-MS in accordance with the manufacturer's recommendations and the Eurofins Corporate SOP No. CA-Q-S-005 Calibration Curves (General).

10.2.7. The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The correlation coefficient (r) of the calibration curve must be ≥0.995 If not, the problem must be corrected, and the instrument must be recalibrated.

10.2.8. Calibration Curve Read-Back:

10.2.8.1. Low-Level readback for aqueous samples and for Silver for soil samples – evaluate Cal 1 which is at the LLOQ for aqueous samples. The %RE (relative error) must be +/- 20% (see Sec 11.5); if not, stop the analysis and recalibrate.

10.2.8.2. Low-Level readback for soil samples except Silver- evaluate Cal 2 which is at the LLOQ for soil samples. The %RE (relative error) must be +/- 20% (see Sec 11.5); if not, stop the analysis and recalibrate.

10.2.8.3. Mid-Level readback – evaluate Cal 4. The %RE (relative error) must be +/- 10% (see Sec 11.5); if not, stop the analysis and recalibrate.

10.3. Sample Analysis

10.3.1. All sample measurements must be made within the calibration range of the instrument. Preparation of all reference materials used for calibration must be documented.

10.3.2. Typical dilutions for samples and relating QC samples:

10.3.2.1. Water, SPLP, soil, and their relating batch QC (MB, LCS, LCSSRM, Dup, MS, MSD) samples are typically analyzed undiluted. Dilutions are prepared in 5% HNO3 / 5% HCl..

10.3.2.2. TCLP and the relating batch QC samples (LCS, Dup, MS, MSD, LB) are typically diluted at a 10X prior to analysis with the exception of the TCLP method blank (MB). The TCLP method blanks (MB) for method 3010A are typically analyzed undiluted. Dilutions are prepared in 5% HNO3 / 5% HCl.

10.3.3. The samples are analyzed only after the ICV, and ICB criteria are met.

10.3.4. The samples are analyzed in a sequence as follows:

Typical Analytical Sequence
INSTRUMENT WARM-UP (approx. 30 minutes)
TUNE CHECK
STANDARDIZATION/CALIBRATION (Blank & 5 Cal Stds)
ICV
ICB
ICSA
LRC-A
LRC-B
LRC-C
CCV
CCB
10 Samples
CCV
CCB
10 Samples
CCV
CCB
{continue until done; after 12 hrs run ICSA}

Note: The analytical sequence must end with the analysis of the CCV and CCB. The 10 samples include all QC samples/standards with the exception of CCVs and CCBs.

10.3.5. Determine the concentration of the samples and QC items using the procedures of Section 11.

10.3.5.1. The amount of sample digestate needed to prepare the desired dilution is determined from the following equation:

$$\text{V digest} = \frac{Vf\ vol}{DF}$$

Where:
Vf vol = final volume of diluted sample (mL)
V digest = volume of sample digestate used to make the dilution (mL)
DF = Dilution Factor

10.3.5.2. The dilution factor is calculated as follows:

$$\text{DF} = \frac{Vf\ vol}{V\ digest}$$

Where: Vf vol = final volume of diluted sample extract (mL)
V digest = volume of sample extract used to make the dilution (mL)

Note: The following examples are based on a final volume of 100mL. It may be more convenient to prepare dilutions at smaller final volumes.

EXAMPLE

A sample digestate is analyzed and one of the target analytes exceeds the linear range of the ICP-MS. 1.0mL of the digestate is added to a 100mL volumetric flask and the extract brought up to volume with 5% HNO3 / 5% HCl. What is the dilution factor?

$$DF = \frac{100ml}{1.0ml} = 100$$

Some samples may require multiple dilutions; that is, a dilution of a dilution will have to be made. In this case, the final dilution factor is the product of the individual dilutions.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Aqueous and Leachate Samples:

Leachate samples are routinely reported in mg/L while the ICP-MS is routinely calibrated in ug/L. If the results are reported in ug/L, the conversion factor is omitted from the calculation.

11.3.1. The concentration of the target analyte in liquid samples is calculated as follows:

Concentration (mg/L) = $ug/L(from\ pr\,\text{int}\,out) \otimes \frac{F}{V} \otimes DF \otimes \frac{1mg}{1000ug}$

Where: F = final volume of the sample digestate (L) -usually 50mL
V= volume of sample digested (L)
DF = dilution factor

11.3.2. The Reporting Limit (RL) of the target analyte in liquid samples is calculated as follows:

Concentration(mg/L) = $RL_{table1} \otimes \frac{F}{V} \otimes DF \otimes \frac{1mg}{1000ug}$

Where: $RL_{table\ 1}$ = reporting limit from this SOP (ug/L)
F = final volume of the sample digestate (L)
V= volume of sample digested (L)
DF = dilution factor

The Table 1 Reporting Limits assumes:
F = 50ml
V = 50ml
DF = 1

11.4. Soil/Solid Samples

Soils and solids are routinely reported in mg/kg while the ICP-MS is routinely calibrated in ug/L. If the results are reported in ug/kg, the conversion factor is omitted from the calculation.

11.4.1. The concentration of the target analyte in soil and solid samples is calculated as follows:

Concentration (mg/kg,dw) = $ug/L(from\ pr\,\text{int}\,out) \otimes \frac{F}{W \otimes solids} \otimes DF \otimes \frac{1mg}{1000ug}$

Where: F = final volume of the sample digestate (L)
W = volume of sample digested (kg)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)

For example, if the percent solid is 85%, the decimal equivalent is 0.85; if the %solid is 100%, the decimal equivalent is 1.0.

11.4.2. The Reporting Limit (RL) of the target analyte in soil/solid samples is calculated as follows:

$$\text{Concentration(mg/kg,dw)} = RL_{table1} \otimes \frac{0.0010kg}{W \otimes solids} \otimes \frac{F}{0.100L} \otimes DF$$

Where:
$RL_{table\ 1}$= reporting limit from Table 1 of this SOP
W = weight of sample digested (kg)
F = final volume of the sample digestate (L)
V = volume of sample digested (L)
DF = dilution factor
solids = decimal equivalent of the percent solids (percent solids/100)
dw = dry weight

The Table 1 Reporting Limit assumes:
F = 0.100L
DF = 1
W = 0.0010kg (1.0g)
solids = 1.0

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.5. Relative Error (%RE):

% Relative Error = (100) (MC-TC)/TC

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.6. Data Reduction:

11.6.1. All data is recorded directly in TALS' Analyst Desktop II program.

11.6.2. Record standard preparations in TALS Reagent module.

11.6.3. Sample and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, Initial and

final sample volume, spike name and amount used, all reagents and their corresponding lot numbers, creation dates and expiration dates.

11.6.4. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch.

11.6.4.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.6.4.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.6.4.3. Acknowledge by filling in responses to all unacknowledged findings.

11.6.4.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.6.4.3.2. Fill in appropriate comments in the response box, then hit 'OK.'

11.6.4.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

12.0. Method Performance

12.1. Method/Instrument Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to Eurofins Edison SOP No. ED-GEN-022, Training, for the laboratory's training program.

12.4. Lower-Limit of Quantitation (LLOQ):

12.4.1. LLOQ verification: prior to analyzing samples under method 6020B, after any change that may affect the LLOQ, and quarterly, the LLOQ must be verified. The LLOQ is initially verified by the analysis of 7 replicate samples spiked at the LLOQ. For aqueous, prep method 3005A and 3010A, use the same concentration as Cal 1 (see Sec 7.2.2 for preparation procedures). For LLOQ soil except Silver, preparation method 3050B, use the same concentration as Cal 2 (see Sec 7.2.2 for preparation procedures). For Silver, spike 2mL of the 6020B RL standard into a clean 50mL digestion cup and prepare using method 3050B. The seven replicates must be digested prior to analysis. See SOPs ED-MTP-002, ED-MTP-003 and ED-MTP-005 for digestion instructions. The LLOQ is verified when the mean recovery of the 7 replicates is +/-35% of the true value and the RSD $\leq$ 20%.

12.4.2. LLOQ on-going verification: the LLOQ is re-verified quarterly by the analysis of a method blank (MB) and 1 blank sample (deionized water) spiked at the LLOQ. Use the same concentrations as CAL 1 (3005A and 3010A) and Cal 2 (3050B) for soil, except Silver. For Silver, spike 2mL of the 6020B RL standard into a clean 50mL digestion cup and prepare using method 3050B. See Sec 7.2.2.2 for the preparation procedures. The MB and LLOQ samples must be digested prior to analysis. See SOPs ED-MTP-002, ED-MTP-003 and ED-MTP-005 for digestion instructions. The LLOQ is re-verified if the MB result is +/- 0.5 times the reporting limit for each analyte and when the recovery of the LLOQ sample is +/-35% of the true value.

13.0. Pollution Control

It is Eurofins's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal

restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out.

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization. The neutralization system consists of a 55-gallon poly open top container. There is also a local snorkel exhaust for venting any noxious odors. Once the liquid is transferred into the container, the contents are sparged with air and a representative pH is measured and the amount of material is recorded in the discharge log. The pH value is recorded in the discharge log, and the liquid is neutralized to a pH of 6-9 SU using food grade sodium bicarbonate. Once the secondary pH has been taken, confirmed and recorded, the discharge valve is opened to the municipal sewer system and the neutralization process is complete. The date, type of waste stream, and volume discharged are recorded on a discharge log.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  TSDF under Veolia Profile Number: 590598

- Soil Retain Samples - These containers are stored in the main sample-receiving laboratory cooler until they are designated for disposal. Once designated for disposal, the samples are transferred to the waste disposal area and placed into cubic-yard disposal containers, e.g., Jupiter-boxes. This waste is shipped directly to the TSDF under Veolia Profile Number 402535 and is subject to incineration.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed.,U.S. Environmental Protection Agency, SW846 Manual, Method 6020A, Revision 1, February 2007.

15.2. TestAmerica Edison SOP ED-MTP-005, *Hot Block Digestion of Sediments and Sludges and Soils Using SW846 Method 3050B,* most current revision.

15.3. TestAmerica Edison SOP No. ED-MTP-003, *Digestion of Water and Wastewater Samples for Analysis by ICP and ICP-MS, SW846 Method No. 3010A,* most current revision.

15.4. TestAmerica Edison SOP ED-MTP-002, Digestion of Water and Wastewater

Samples for Analysis by ICP and ICPMS, SW846 Method 3005A, most current revision

15.5. TestAmerica Corporate SOP No. CA-Q-S-005, *Calibration Curves (General)*, most current revision.

15.6. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.7. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.8. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.9. Agilent ICP-MS (7800/7900) MassHunter Workstation User Guide, p/n G7201-90403, Rev. A, June 2017.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Standards and Matrix Spiking concentrations
Attachment 2: Interference Check standard elemental concentrations

18.0. Revision History

- Revision 12, dated 21 September 2024
  - Sec 7.2: Updated the preparation frequency for the working calibration standards and Intermediate standards.
  - Sec 7.2.2: Updated the preparation procedures for standards Cal1 and Cal4/CCV to reflect current procedures.
  - Sec 7.2.6: Replaced Internal standard stock solution Testamerica-1 with stock solution Internal Standard Multi-Element Mix 4. Removed stock standard MSGE-100PPM (no longer in use). Updated the Working Internal Standard preparation instructions to reflect current practices.
  - Sec 10.3.4 Removed CRI, ICSAB, and Rn Chks from the typical analytical sequence table, not applicable to this method.

- Revision 11, dated 21 April 2023
  - Updated to Eurofins branding throughout the SOP.
  - Sec 1.1, Table1: Updated the reporting limit concentrations for Ca and Na in Water to reflect current laboratory limits.
  - Sec 2.0 & Sec 8.0: Clarified instructions for the analysis of dissolved samples (i.e.,

digestion not required for the analysis of dissolved samples)
- Sec 7.2.2: Updated preparation instructions for calibration standards to reflect current lab practices.
- Sec 7.2.4: Updated preparation instructions for the ICV standard to reflect current lab practices.
- Sec 7.2.4.2: Added preparation instructions for Interm_ICV std.
- Sec 7.2.6: Added Titanium to the list of analytes that are analyzed in the No-gas mode.
- Sec 7.2.10.2: Updated preparation instructions for the working EPA Tune check standard.
- Sec 8: Added requirements to add NCM note for dissolved samples which are lab filtered.
- Sec 9.2, Table 2: Replaced minimum # of standards from 3 to 5 (and 1 blank).
- Sec 9.2.5: Updated preparation instructions for LRC-A and LRC-B; added preparation instructions for LRC-C.
- Sec 10.1: Added 'total analytes' to clarify the digestion requirements prior to analysis.
- Sec 10.3.2.1: Added LCSSRM to the list of QC items that are typically analzed undiluted.
- Sec 10.3.2.2: Updated typical dilution instructions (i.e., 10X) for TCLP analysis (exception MB).
- Sec 10.3.4: Added LRC-C in the typical analytical sequence table.

- Revision 10, dated 16 June 2022
  - Sec 1.1, Table 1 and throughout the SOP, revised to include digestion method SW846 3005A and SOP# ED-MTP-002 as prep method reference.
  - Sec 1, Table 1: Revised RL for Ag to 0.40 mg/kg.
  - Sec 14.2: Updated waste disposal procedures.
  - Sec 15.4: Added SOP# ED-MTP-002 to the list of references; adjusted subsequent sections accordingly.
  - Sec 10.2.8 and 12.4: Added information related to Ag QC spiking for LLOQ.

- Revision 9, dated 29 September 2021
  - Sec. 7.2.1 and 7.2.3: Revised the concentration of TA-CAL2 to 2,000 mg/L to reflect the concentration in the COA for the standard.
  - Sec. 7.2.4: Revised the volume of TA-CAL2-SS added in the preparation of the ICV working standard to 2.0 ml to reflect actual laboratory practices.

- Revision 8, dated 10 June 2020
  - Updated SOP header to Eurofins logo.
  - Throughout the SOP removed all method 6020A references.
  - Throughout the SOP removed all references to 2%HNO_3/0.5%HCl and replaced with 5%HNO_3/5%HCl.
  - Sec 1.1, Table 1: removed references to typical dilutions. Updated the RLs for Selenium

- Sec 6.1: Added two ICPMS instruments, Agilent 7800 & 7900. Removed reference to resolution and grating. Added instrument mass range. Updated the models of the heat exchanger and autosampler.
- Sec 7.1.6: Added preparation instructions for 5%HNO_3/5%HCl.
- Sec 7.2.1: Replaced Inorganic Ventures with CPI International as the vendor for the stock cal stds. Updated all stock standards to reflect this change.
- Sec 7.2.2: Updated preparation instructions for the working cal standards.
- Sec 7.2.2.1: Added preparation instructions for MS-Cal-Int.
- Sec 7.2.3: Updated the Initial calibration verification stock standards.
- Sec 7.2.4: Updated preparation instructions for the ICV working standard.
- Sec 7.2.4.1: Added preparation instructions for MS-ICV-Int.
- Sec 7.2.6: Updated preparation instructions for the Internal standard. Changed recommended mass for Ca from 44 to 40. Updated typical referenced internal std for As & Se from Ge to Sc. Updated typical analysis mode used for Iron, Beryllium, and Boron.
- Deleted Sec 7.2.7, LLICV; subsequent sections adjusted accordingly.
- Sec 7.2.7: Replaced Inorganic Ventures with CPI International as the vendor for the stock ICSA standard. Added stock concentrations for ICSA.
- Sec 7.2.8: Updated preparation instructions for working ICSA.
- Sec 9.1.2: Updated preparation instructions for the LCS water samples.
- Sec 9.1.4.1: Updated preparation instructions for the aqueous matrix spikes (MS).
- Deleted Sec 9.2.4, LLICV/LLCCV; subsequent sections adjusted accordingly.
- Sec 9.2.5: Updated preparation instructions for Linear Range Standard (LRC), solutions (LRC)-A and (LRC)-B. Deleted (LRC)-C.
- Deleted Sec 9.3.2, Linear Dynamic Range.
- Sec 10.2.2: Added reference material, MassHunter Workstation User Guide, for the ICPMS instruments 7800 & 7900. Removed references to ICPMS1&2 (includes Chemstation software and relating Operation Manuals) from this SOP.
- Sec 10.2.4: Added instrument method program and reference material, MassHunter Workstation User Guide, for the ICPMS instruments 7800 & 7900.
- Sec 10.2.8.1 & 10.2.8.2: Added Low-Level readback evaluation instructions for aqueous and soil samples.
- Sec 10.2.8.3: Updated Mid-Level readback evaluation instructions.
- Sec 10.3.2: Updated typical dilution instructions.
- Sec 10.3.4: Updated Typical Analytical Sequence table to reflect current laboratory practices.
- Sec 11.5: Added %Relative Error calculation equation.
- Deleted Sec 12.4 LLQC; subsequent sections adjusted accordingly.
- Sec 12.4.1: Updated preparation instructions for LLOQ.
- Sec 12.4.2: Added instructions for the On-going LLOQ verification.
- Deleted references, ICPMS Operator's Manual, Sec 15.2, 15.9, 15.10, not applicable.
- Sec 15.8: Added reference for the Agilent ICPMS 7800/7900 User Guide.
- Sec 18.0, Attachment 1: Updated calibration, ICV, CCV concentrations; removed dilution instructions.

- Sec 18.0, Attachment 2: Updated ICSA analyte list and concentrations.

- Revision 7, dated 3 July 2018
  - Sec 1.1: Added Method 6020B to the list of methods applicable to this SOP
  - Throughout the SOP removed all references to Reagent Water and replaced with 2%HNO_3/0.5%HCl
  - Sec 7.2 & Sec 7.2.4: Updated the ICV preparation frequency for method 6020B to daily.
  - Sec 7.2.8 & 7.2.9: Renamed “ICP Interference Check Solutions” and ‘Working Interference Standards” to Spectral Interference check solution (SIC).
  - Sec 7.2.9: Added ICSA preparation instructions for method 6020B
  - Sec 9.1 Table & Sec 9.1.1.2: Added MB control limit criteria for method 6020B
  - Sec 9.1.6.5: Added serial dilution control limit criteria for method 6020B
  - Sec 9.1.7.4: Added PDS control limit criteria for method 6020B
  - Sec 9.2 Table 2: Added calibration curve correlation coefficient criteria, ICB/CCB criteria, Internal Standard recovery criteria, and Linear Range Check control criteria and frequency for Method 6020B.
  - Sec 9.2.3: Added ICB and CCB control criteria for method 6020B
  - Sec 9.2.6: Added LRC preparation and control limit criteria for method 6020B
  - Sec 9.3.2.2: Added Linear Range check (LRC) to the Instrument Performance Criteria.
  - Sec 10.2.7: Added calibration curve correlation coefficient criteria for 6020A and 6020B
  - Sec 10.2.8: Added calibration curve read-back instructions for method 6020B
  - Sec 10.3.4: added LRC-A, LRC-B, LRC-C to the typical Analytical Sequence Table.
  - Sec 11.5.4: Removed the Metals Data Review Checklist. Added instructions to complete the Data Review Checker (DRC) in TALS.
  - Sec 12.5: Added LLOQ standard preparation and criteria information.
  - Remove Sec 15.11 – Removed reference to WI CA-Q-WI-044, WI not applicable.
  - Attachment 1: Added LLQC and LLOQ on this Table.

- Revision 6, dated 27 April 2018
  - Sec 7.2.11.1: Added Magnesium as one of the working tune check standard constituents.
  - Sec 9.1.2: Revised to clarify the acceptance criteria used in the COA when evaluating recoveries for LCS in soil matrix.

- Revision 5, dated 29 November 2016
  - Sec 7.1.6: removed ICPMS Reagent Dilution logbook and replaced with TALS Reagent Module.
  - Sec 7.2.1: added calibration stock standards STLNJ-STD-1 & STLNJ-STD-2
  - Sec 7.2.2: added updated preparation instructions for Cal1 to reflect lab practices
  - Sec 7.2.6: added typical analysis mode for Selenium, i.e., Hydrogen. Updated the resolution check criteria to reflect lab practices
  - Sec 11.5.4: removed reference to checklist CA-Q-WI-004 and replaced with EDS-WI-

146
- Attachment 1: updated Cal1 standard concentrations; removed the linear range column

- Revision 4, dated 13 November 2015
  - Throughout the SOP removed all reference to SOP# ED-MTP-002 (SW method 3005).
  - Sec 1.1: Revised RL for Ag to reflect actual laboratory's RL.
  - Sec 7.2.1: Removed calibration standard solution STLNJ-STD-1 and STLNJ-STD-2 (Cal 1) and added standard TA-88.
  - Sec 7.2.2: Revised preparation procedure for CAL1; replaced calibration standard solution STLNJ-STD-1 and STLNJ-STD-2 with standard TA-88. Deleted prep instructions for ME_Se_1ppm.
  - Sec 7.2.6: Updated the preparation instruction of the working internal standard solution to include Triton X-100.
  - Sec 9.1 and 9.1.5: Added Matrix Spike Duplicate (MSD) to the list of Sample QC. Subsequent sections adjusted accordingly.
  - Sec 11.5.4: Updated the metals data review checklist form number.
  - Sec 15: Removed reference to SOP ED-MTP-002 (method 3005A) in Sec 15.4; Deleted Data review checklist WI# EDS-WI-007 in Sec 15.10, checklist is referenced in the applicable digestion SOP; subsequent sections adjusted accordingly.
  - Sec 15.11: Replaced Data review checklist (EDS-WI-123) with the standardized Data review checklist WI# CA-Q-WI-043.

- Revision 3, dated 19 August 2013
  - Sec 1 & 12: Revised LQM section references to reflect the most current LQM revision.
  - Sec 7.2.11: Added EPA Tune Check stock standard including preparation and analysis instructions.
  - Sec 10.2.2 & Sec 10.2.4: Added the Operating Manual and Tuning & Application Handbook calibration information for the Mass Hunter Software (ICPMS2)
  - Sec 11.5.4: Added requirement to complete the Initial Calibration Data Review checklist (EDS-WI-123) prior to data submission.
  - Sec 15.11 & 15.12: Added reference information for the Tuning & Application Handbook and MassHunter Workstation Operator's Manual.
  - Sec 15.13: Added reference information for TestAmerica Edison Metals Initial Calibration Data Review checklist.

- Revision 2, dated 23 August 2011
  - Sec 7.2.2 Revised preparation procedure for CAL 1; changed added amount of STLNJ-STD-1 and STLNJ-STD-2 from 2 ml to 4ml

- Revision 1, dated 10 March 2011
  - Sec 1.1 Table 1: Revised RL for soil and water

- Sec 7.2.2: Revised preparation procedure for CAL1; added prep for ME_Se_1ppm.
- Attachment 1: Revised the spiking concentration for the calibration, LLICV/LLCCV and MS concentration.

Attachment 1

STANDARDS AND MATRIX SPIKING CONCENTRATIONS

Name	Mass	Cal-1	Cal-2	Cal-3	Cal-4/CCV	Cal-5	ICV	*Water*	*Soil*
		(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	(ug/L)	*(ug/L)*	*(mg/Kg)*
Be	9	0.8	4	50	100	200	80	*25*	*5*
B	11	80	400	125	250	500	200	*500*	*100*
Na	23	200	1000	12500	25000	50000	20000	*2500*	*500*
Mg	24	200	1000	12500	25000	50000	20000	*2500*	*500*
Al	27	40	200	12500	25000	50000	20000	*2500*	*500*
K	39	200	1000	12500	25000	50000	20000	*2500*	*500*
Ca	40	200	1000	12500	25000	50000	20000	*2500*	*500*
Ti	47	4	20	50	100	200	80	*50*	*10*
V	51	4	20	50	100	200	80	*50*	*10*
Cr	52	4	20	50	100	200	80	*50*	*10*
Mn	55	8	40	50	100	200	80	*250*	*50*
Fe	56	120	600	12500	25000	50000	20000	*2500*	*500*
Co	59	4	20	50	100	200	80	*25*	*5*
Ni	60	4	20	50	100	200	80	*50*	*10*
Cu	63	4	20	50	100	200	80	*50*	*10*
Zn	66	16	80	125	250	500	200	*250*	*50*
As	75	2	10	50	100	200	80	*50*	*10*
Se	78	2.5	12.5	50	100	200	80	*50*	*10*
Sr	88	4	20	50	100	200	80	*50*	*10*
Mo	95	4	20	50	100	200	80	*50*	*10*
Ag	107	2	10	25	50	100	40	*25*	*5*
Cd	111	2	10	50	100	200	80	*25*	*5*
Sn	118	16	80	50	100	200	80	*50*	*10*
Sb	121	2	10	50	100	200	80	*25*	*5*
Ba	137	4	20	50	100	200	80	*50*	*10*
Tl	205	0.8	4	50	100	200	80	*20*	*4*
Pb	208	1.2	6	50	100	200	80	*25*	*5*

Attachment 2

ELEMENT	ICSA (ug/L)
Sodium	100000
Magnesium	100000
Aluminum	100000
Potassium	100000
Calcium	100000
Titanium	2000
Iron	100000
Molybdenum	2000
Chloride	1000000
Carbon	200000
Phosphorus	100000
Sulfur	100000

Title: Mercury Analysis for Solid and Semisolid Waste Samples using the Leeman Mercury Analyzer (Cold Vapor Technique) by SW846 Method 7471B

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Approvals (Signature/Date):

Laura Demone	08/11/22	Donald Evans	08/11/22
Laura Demone Department Manager	Date	Donald Evans Operations Manager	Date
	08/11/22		08/11/22
Carl Armbruster Quality Assurance Manager	Date	Mark Acierno Laboratory Director	Date
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1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

SW846 Method 7471B is applicable to the determination of total mercury (organic and inorganic) in soils, sediments, bottom deposits, and sludge-type materials. All samples must be digested prior to analysis. If this digestion procedure is not sufficient to dissolve a specific matrix type or sample, then this method is not applicable for that matrix.

The typical detection limit using a 0.6 gram sample size is 0.017 mg/Kg Hg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

The sample is digested as described in this SOP and is analyzed using cold vapor atomic absorption. 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 at 253.7-nm is measured as a function of mercury concentration.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Potassium permanganate is added to eliminate possible interferences from sulfide.

4.2 Copper may also be a potential interference although no effect has been observed for samples containing up to 10 mg/Kg Copper.

4.3 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 254 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 hydrochloride reagent (25 mL).

4.4 Certain volatile organic materials that absorb at 253.7 nm may also cause interference. The analysis of the undigested sample should determine if this type of interference is present.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/M^3 Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/M^3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Permanganate	Oxidizer	5 Mg/M^3 for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

6.1.1. Leeman Laboratories Inc. Hydra IIAA Automated Hg Analyzer

6.1.2. Computer and Monitor with Leeman Envoy software.

6.1.3. Top loader balance, 300gm capacity, and minimum sensitivity of ±1.0 mg

6.1.4. Hotblock digestor: Adjustable and capable of maintaining a temperature between 95 ±3°C.

6.2. Supplies

6.2.1 Pipettes and tips in various sizes

6.2.2 100 ml volumetric flasks

6.2.3 15 ml sample cups

6.2.4 Pump tubing:

- Sample, viton, blue tab
- Reductant, red tab
- Drain, blue tab

- Rinse, Black tab

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6.2.5 Drying Tube – Purchased pre-packed with Magnesium Perchlorate from Leeman Labs. Located prior to the optical cell.

6.2.6 Nitrogen or Argon supply - capable of producing 80 PSI.

7.0 Reagents and Standards

Storage requirements: store at room temperature

Life of Reagent: Concentrated acids: refer to manufacturer's instructions
Laboratory prepared reagents and diluted acids: one year

Record reagent preparations in the TALS Reagent Module.

7.1 Reagents

7.1.1 Nitric acid - Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions

7.1.2 Hydrochloric acid-Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.3 Potassium Permanganate (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.4 Sodium Chloride (analytical reagent grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.5 Hydroxylamine Hydrochloride (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.6 Stannous Chloride (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.7 Deionized water - 18 megohm minimum

7.1.8 10% Hydrochloric Acid- Cautiously add 200 ml of concentrated HCl to a container and bring to final volume of 2 liters with deionized water. Store at room temperature; stable for one year.

7.1.9 Stannous chloride solution - Add 50 g of $SnCl_2$ to 500 ml 10% HCl solution. Store at room temperature; stable for one year.

7.1.10 Sodium chloride/Hydroxylamine Hydrochloride solution - Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1 liter using deionized water. Store at room temperature; stable for one year.

7.1.11 Potassium permanganate ($KMnO_4$) 5% solution w/v - Dissolve 100 g of $KMnO_4$ in deionized water and dilute to 2 liters using deionized water. Store at room temperature; stable for one year.

7.1.12 0.15% Nitric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 12mL of concentrated HNO_3 and bring the final volume up to 8 liters with deionized water.

7.2 Standards

Storage requirements: all standards are stored at room temperature

Shelf-life:	Stock standards – refer to manufacturer's instructions Intermediate standards – made fresh daily Working standards – made fresh daily (Note: expiration date must not go beyond the expiration date of the source stock).
Concentration:	see Attachment 1 for example certificates of analysis (COA) of Hg standards listed below. The COA lists the manufacturer's Lot number, certified concentration and shelf life.

Document standard preparation in TALS, see Sec 11.5.3.

7.2.1 Stock Mercury Calibration (10 ppm Hg) - Purchase from SCP Science; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.2 Secondary Stock Mercury Calibration Standard (10 mg/L Hg) - Purchase from Inorganic Ventures; store at room temperature; for stability information, refer to manufacturer's instructions.

7.2.3 Intermediate Calibration Standard (DCAL-Int), 100 ug/L Hg: Dilute 1 ml of Hg calibration stock standard solution (Sec 7.2.1) to 100 ml with 0.15% HNO_3.

7.2.4 Intermediate Initial Calibration Verification Standard (DQCS-Int), 100 ug/L Hg: Dilute 1 ml of Hg stock Calibration Verification standard, 10ppm Hg (Sec 7.2.2) solution to 100 ml with 0.15% HNO_3.

7.2.5 Calibration Standards: Use six 50 ml hotblock cups to prepare the standards. Spike the calibrations standards cups as follows:

Calibration Standard	DCAL-Int Spike Volume (mL)	0.15% HNO_3 (mL)	Final Conc (ug/L)
Std1 (Cal Blk)	0	5	0
Std 2	0.1	4.9	0.2
Std 3	0.5	4.5	1
Std 4	1	4	2
Std 5	2.5	2.5	5
Std 6	5	0	10

Digest the standards following Sample preparation in Section 10.1.

7.2.6 Initial Verification Standard (ICV) 5.0 ppb: Using a hotblock cup, add 2.5 mL of 0.15 HNO_3 and 2.5 ml of DQCS-INT, (Sec 7.2.4). Digest the standard following Section 10.1. After digestion, the ICV will contain 5.0 ppb of Hg.

7.2.7 Continuing Calibration Verification Standard (CCV) 5.0 ppb: Follow the preparation instructions for Std 5 (see Sec 7.2.5)

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Plastic Glass	5 grams	Cool 4 ± 2ºC	28 Days	SW846 Method 7471B

[1] Inclusive of digestion and analysis.

Samples are to be analyzed without drying. A separate procedure is used to determine the percent solids in the sample.

For sample homogenization procedures refer to TestAmerica Edison SOP ED-GEN-007 (Subsampling).

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< RL; 5% of the regulatory limit; 5% of the measured concentration in the sample
Laboratory Control Soil Sample Reference Material (LCSSRM) in soil samples	1 in 20 or fewer samples	Vendor's certified limit
Matrix Duplicate (DUP)[1]	1 in 20 or fewer samples	If original sample and dup are both ≥ 5X RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise ±RL.
Matrix Spike (MS)[1]	1 in 20 or fewer samples	80-120%
Matrix Spike Duplicate (MSD)[2]	1 in 20 or fewer samples	80-120%; If original sample and dup are both ≥ 5X RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise ±RL.
Serial Dilution (SD)	1 in 20 or fewer samples	±10%

[1] The sample for DUP and MS are randomly selected, unless specifically requested by a client; Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.
[2] A MSD is prepared and analyzed when requested by the client.

9.1.1. Method Blank: One laboratory method blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). The method blank is used to identify possible contamination during acid digestion. Results must be less than the RL, 5% of the regulatory limit for that analyte, or 5% of the measured concentration in the sample. If the analyte concentration in the method blank is above this control limit, the batch must be prepared again and the samples reanalyzed.

9.1.2. Laboratory Control Sample Soil Reference Material (LCSSRM: A laboratory control sample must be analyzed with each group of samples digested. For solid matrices, a vendor supplied solid matrix with certified values is carried through the same preparation procedure as the samples. The results of the solid LCS must fall within the 'QC Performance acceptance limits' of the reference material used for that sample. If not, all samples prepared in association with the LCS must be redigested and reanalyzed.

9.1.3 Matrix Duplicate (DUP): A duplicate is analyzed for each batch of samples digested. If original sample and duplicate are both ≥ RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise, ±RL.

9.1.4 Matrix Spike (MS): A matrix spike is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.5 mL of DCAL-Int (Sec 7.2.3). This is equivalent to 1.0 ppb Hg (on instrument). A recovery of 80-120% is required.

9.1.5 Matrix Spike Duplicate (MSD): A matrix spike duplicate is prepared and analyzed when requested by the client. A portion of sample is spiked with 0.5 mL of DCAL-Int (Sec 7.2.3). This is equivalent to 1.0 ppb Hg (on instrument). A recovery of 80-120% is required. If matrix spike sample and matrix spike duplicate sample are both ≥ RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise, ±RL

9.1.6 Serial Dilution (SD): A five fold serial dilution must be performed on one sample per batch. The sample should contain a sufficiently high concentration; minimally a factor of 25 times the estimated detection limit. Dilute the sample by a minimum of five fold (1+4) and reanalyze. The results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

9.2. Instrument QC

9.2.1 Initial Calibration Verification (ICV): Initial calibration is verified after calibration using an independent check standard at a concentration near

the mid-point of the calibration (5.0ppb); see Sec 7.2.6 for preparation instructions. The results must be within 10% of the true value. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

9.2.2 **Continuing Calibration Verification (CCV):** Calibration verification is performed after the calibration, after every 10 samples, and at the end of the run. Use a concentration of mercury at the midpoint of the calibration range (5.0 ppb). See Sec. 7.2.7 for preparation instructions. The value obtained must be within 10% of the true value. If not, stop the analysis and recalibrate. Re-analyze the previous ten samples following the last good calibration verification. .

9.2.3 **Initial and Continuing Calibration Blank (ICB/CCB):** ICB/ CCB must be analyzed after the calibration curve, every 10 samples, and at the end of the analytical run. The absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

10 Procedure

10.1. Sample Preparation (includes all samples, standards, and blanks)

10.1.1 Mix the sample well and weigh 0.5 - 0.6 grams of sample (including the LCSSRM) and place in the bottom of an appropriately identified 50 mL hotblock cup. For QA samples, weigh three portions of 0.5 - 0.6 grams of sample and place in the bottoms of three hotblock cups labeled as SAMPLE, DUP, and MS. Before adding any reagents, spike the MS sample with 0.5 mL of DCAL-Int standard.

10.1.2 Except the calibration standards, add 5 mL of deionized water to all hotblock cups (i.e., all field samples, MB, LCSSRM, Dup, and MS).

10.1.3 Add 1.5 ml concentrated HNO_3 and 4.5 ml concentrated HCl. Heat 2 min on the hotblock at 95 $\pm$3°C. Cool.

10.1.4 Add 15 mL deionized water and 15 mL potassium permanganate solution (Sec 7.1.11) to each hotblock cup. Mix well. The same amount of $KMnO_4$ must be added to the standards and samples.

10.1.5 Cap the hotblock cups loosely enough so that pressure does not build up but also tight enough so that the caps stay on and that volume loss due to heating is minimized. Heat 30 min on the hotblock at 95 $\pm$3°C. Cool.

10.1.6 To each hotblock cup, add 6 ml Sodium chloride - Hydroxylamine hydrochloride solution to reduce excess permanganate.

10.1.7 Using deionized water, bring to a 50 mL final volume. Cap and mix well.

10.2. Calibration

10.2.1 The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated according to the manufacturer's specifications and must contain at least four standards and a blank. The laboratory currently uses five standards and a blank. The correlation coefficient of the calibration curve must be ≥0.995. If it does not, the problem must be corrected, and the instrument must be recalibrated. Standard preparations must be documented in the in TALS reagent module.

10.2.2 Prepare the calibration standards and Calibration Verification Standards as stated in Sections 7.2.5, 7.2.6., & 7.2.7.

10.2.3. Calibration Curve Read-Back:

10.2.3.1. Low-Level Readback (at the RL) – evaluate the 0.2 ug/L calibration standard. The %RE (relative error) must be +/- 20% of the true value (see Sec 11.4). If %RE is outside of the criteria limits, stop the analysis and recalibrate.

10.2.3.2. Mid- Level Readback – evaluate the 5.0 ug/L calibration standard (the mid-level calibration standard). The %RE (relative error) must be +/- 10% of the true value (see Sec 11.4). If %RE is outside of the criteria limits, stop the analysis and recalibrate

10.3. Sample Analysis

10.3.1 Following a sample digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data.

10.3.2 The following analytical run sequence is currently used for samples analyzed under Method 7471B:

Instrument Calibration (Blank and five standards)
ICV
ICB
CCV
CCB

10 Samples
CCV
CCB
10 Samples
CCV
CCB
Repeat until run is complete
CCV
CCB

10.3.3 Instrument Operation:

10.3.3.1 Turn on instrument, computer and monitor.

10.3.4 Plumbing the Reagent Lines:

10.3.4.1 One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic tab is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and click the clamp, lever up. The tab end of the tube should be located at the front of the pump head.

10.3.4.2 Reductant (Red); Connect tab end of tube to the reductant bottle and the other end to the bottom of the mixing tee.

10.3.4.3 Sample (Blue); Connect tab end of tube to the autosampler probe and the other end to the top of the mixing tee.

10.3.4.4 Drain (Blue) Connect the tab end of tube to the sample discharge tube connected on the Liquid/Gas separator and the other end to the waste line.

10.3.4.5 Rinse (Black): Connect tab end of tube to rinse tubing that is connected to the rinse bottle. Connect the other end to the rinse tubing leading to the rinse cup.

10.3.5 Preparation of Reagents:

10.3.5.1 Pour the $SnCl_2$ solution into the reductant bottle and connect to the red reductant tube connector.

10.3.5.2 Pour the ten percent HCl solution into the Rinse reservoir bottle.

10.3.6 Starting Program:

10.3.6.1 Click the Envoy icon on the computer desktop

10.3.6.2 Click Method, select 7471_7471B, and select OK
10.3.6.3 On the main screen, click the StartUp icon. Wait 15 minutes before analyzing calibrating.
10.3.6.4 Click Sequence Tab on bottom of screen
10.3.6.5 Click Sequence on top of screen
10.3.6.6 Select Open, then select New
10.3.6.7 Using the Prep Batch Sheet and hand scanner, enter the sample barcodes into the Sample ID column. Include the Serial Dilution and all needed sample dilutions
10.3.6.8 Pour out the digested calibration standards and samples into the proper locations on the autosampler.
10.3.6.9 Click the Run Sequence icon to begin calibration and to run samples.

10.3.7 Creating the Raw Data PDF:

10.3.7.1 Click the Analysis Tab, select Detailed

10.3.7.2 Click Load, select Use This To Make PDF

10.3.7.3 Click Report, Click Printer

10.3.7.4 Include the calibration curve graph:

10.3.7.4.1 Go to Methods Tab and click Calibration, click Print

10.3.7.5 Combine both documents using the PDF Creator program. The new document is located in the Documents folder on the C:\ drive. Add this document to the "Doc's" location in the analytical batch.

10.3.8 Shutting Down the Instrument:

10.3.8.1 Click the Stop icon on the main screen

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2. Precision (RPD):

Matrix Duplicate (MD) = $\frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$

11.3. Concentration: (mg/Kg) = $\frac{C \times V1 \times D}{W}$

Where: C= Element concentration from instrument (ppb)
V1= Final volume of sample digested (in liters)
D= Dilution performed on sample
W= Initial weight of sample digested (in gram)

NOTE: All dry weight corrections are made in LIMS at the time the final report is prepared.

11.4. Relative Error (%RE):

% Relative Error = (100) (MC-TC)/TC

MC = Measured Concentration of the calibration standard
TC = True Concentration for the calibration standard

11.5. Data Processing:

11.5.1. All data is recorded directly in TALS' Analyst Desktop II program.

11.5.2. Import Data to TALS

11.5.2.1. Click the Analysis tab
11.5.2.2. Select the analytical run that needs to be imported
11.5.2.3. Select Statistics
11.5.2.4. Click Load and select TALS Import, click OK
11.5.2.5. Click Report, click CSV File
11.5.2.6. Name the import file (e.g., batch name, today's date)
11.5.2.7. The newly created import file is in the Import Folder on the desktop. Send to TALS Import Folder

11.5.3. Sample and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers, creation and expiration dates.

11.5.4. All reagents must be recorded in TALS Reagent Module.

11.5.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable preparation batch.

11.5.5.1. Open the preparation batch and click on the Edit tab above to enter the Edit Mode.

11.5.5.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.5.3. Acknowledge by filling in responses to all unacknowledged findings.

11.5.5.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.5.3.2. Fill in appropriate comments in the response box, then hit 'OK.'

11.5.5.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

11.5.6. Record the following reagents and the volume used for sample preparation in the batch information page under "batch comments" in TALS Analyst Desktop II: concentrated sulfuric acid, concentrated nitric acid, potassium permanganate, potassium persulfate and sodium chloride-hydroxylamine hydrochloride. Record reagents in TALS by opening the prep batch, click on "edit" and then right click to choose "view batch information." Enter the information in the "batch comments" section.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2 Instrument Detection Limit

The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined annually or if the instrument is adjusted in any way that may affect the IDL. The IDL is determined by multiplying the average of the standard deviations obtained from the analysis of seven reagent blanks by 3.14.

12.3 Demonstration of Capabilities

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.4 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

12.5. Lower Limit of Quantitation (LLOQ)

12.5.1. LLOQ verification: prior to analyzing samples under method 7470A, after any change that may affect the LLOQ, and quarterly, the LLOQ must be verified. The LLOQ is initially verified by the analysis of 7 replicate samples spiked at the LLOQ (0.2 ppb). The seven replicates must be digested prior to analysis. The LLOQ is verified when the mean recovery of the 7 replicates is +/-30% of the true value and the RSD < 20%.

12.5.2. LLOQ on-going verification: the LLOQ is re-verified quarterly by the analysis of a method blank (MB) and 1 blank sample (deionized water spiked at the LLOQ (0.2 ppb). The MB and LLOQ samples must be digested prior to analysis. The LLOQ is re-verified if the MB result is +/- 0.5 times the reporting limit for each analyte and when the recovery of the LLOQ sample is +/-30% of the .true value.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention.

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed.,U.S. Environmental Protection Agency, SW846 Manual, Method 7471B, Revision 2, January 1998.

15.2. Leeman Hydra IIAA Operating Manual.

15.3. TestAmerica Edison Document ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. Corporate Environmental Health and Safety Manual CW-E-M-001, most current revision.

15.6. TestAmerica Edison Subsampling SOP, *ED-GEN-007,* most current revision.

15.7. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

16.0. Method Modifications:

Item	Method #	Modification
1 (Sec 10.3.5.1)	7471B	Stannous Chloride is automatically added via the instrument versus the manual addition of Stannous Chloride as stated in the method. This is an instrument manufacturer's

		improvement that will reduce error due to loss of Mercury.
Sample preparation	7471B	The amount of DI water added to the sample at digestion is decreased to 15 ml from 50 ml (water bath procedure), while keeping the volume of reagents added during sample digestion the same. Sample final volume for hotblock procedure is modified to 50 ml (100 ml – water bath procedure) which allows for lesser sample volume hence less sample waste produced; hotblock digestion cup has maximum volume of 50 ml.

17.0. Attachments

Attachment 1: Certificate of Analysis of stock standards

18.0. Revision History

- Revision 5, dated 11 August 2022
  - Sec 12.5: Added the lower limit of quantitation (LLOQ), associated instructions and recovery limits.
- Revision 4, dated 03 August 2020
  - Updated SOP header with Eurofin logo.
  - Sec 6.1.1 & 15.2: Replaced Hydra AA with Hydra IIAA.
  - Sec 9.1 Table and 9.1.1: Revised MB control limits to <RL.
  - Sec 9.1, 9.1.4 and 9.1.5: Revised MS and MSD acceptance limits to 80-120%.
  - Sec 9.2.2: Revised CCV acceptance limits to +/-10%.
  - Sec 10.2.3: Added calibration curve read-back criteria.
  - Sec 11.4: Added Relative % error calculation.
  - Sec 11.5: Added Relative % error calculation.
  - Sec 11.5.6.: Added requirements to record reagent volumes in the batch information page of TALS ADII; subsequent sections adjusted accordingly.
- Revision 3, dated 12 October 2018
  - Sec 6.1.2: Removed WinHg instrument software reference and replaced with current instrument software, Envoy.
  - Sec 9.1.2: Revised to clarify the acceptance criteria used in the COA when evaluating recoveries for LCS in soil matrix
  - Sec 10.3.6: Updated the Starting Program instructions to reflect the new instrument software, Envoy.
  - Sec 10.3.7 & 10.3.8: Updated instructions for creating raw data pdf and shutting down instrument.
  - Sec 11.4.2: Updated instructions for importing data to TALS.
  - Sec 11.4.5: Replaced Metals Data Review Checklist with Data Review Checker (DRC) in TALS.

- Sec 15.8 & 15.9: Removed reference to EDS-WI-007 & EDS-WI-125; WI not applicable.

- Revision 2, dated 29 Nov 2016
  - Sec 6.1.4, Sec 10.1.3, & 10.1.5: updated the hotblock temperature range per method 7471B requirements
  - Sec 6.2.7: added Argon gas
  - Sec 7.2.4: the name of the intermediate ICV standard has been changed from DICV-Int to DQCS-Int
  - Sec 7.2.6: removed the CCV preparation instructions using the second source standard. The name of the intermediate ICV standard has been changed from DICV-Int to DQCS-Int
  - Sec 7.2.7: added preparation instructions for the CCV using the primary source standard
  - Sec 9.1: added the quality control sample MSD to the table
  - Sec 9.1.5: added quality control information for the MSD
  - Sec 11.4.5: Metals Data Review Checklist control # has been updated from ED-WI-007 to CA-Q-WI-042

- Revision 1, dated 12 May 2014
  - Sec 1.1: Revised the typical detection limit from 0.033 mg/kg to 0.017 mg/kg to reflect the 50mL final volume – hotblock procedure.
  - Sec 1 & 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 6.1.4: Added hotblock digestor to the list of equipment; removed autoclave from the list.
  - Sec 6.2: Added 50 mL hotblock digestion cups to replace the BOD bottles. Removed supplies which are no longer applicable (BOD bottles, graduated cylinders).
  - Sec 7.0 & 11.4.4: Removed Hg Reagent Logbook and replaced with TALS Reagent Module
  - Sec 7.1. Deleted Sulfuric Acid (previously referenced in Sec 7.1.1). Added 0.15% HNO_3 to list of reagents.
  - Sec 7.2.5: Updated Calibration standards' spiking instructions to reflect the 50 ml final volume.
  - Sec 7.2.6: Updated ICV/CCV spiking instructions to reflect the 50 ml final volume.
  - Sec 8.1: Added plastic container to the list of acceptable sample containers.
  - Sec 9.1 & 9.1.3: Clarified the acceptance limits for Matrix duplicate to reflect actual laboratory practices.
  - Sec 9.1.4: Revised the spiking amount added to the MS sample.
  - Replaced LCSS reference with LCSSRM in various sections of the SOP to reflect TALS QC type reference.
  - Sec 10.1: Revised sample preparation procedure to include hotblock digestor procedure; deleted all reference to autoclave digestion procedure.

- Sec 11.4.5 & 15.9: Added Work instructions EDS-WI-125 (TestAmerica Edison Metals Initial Calibration Data Review for Mercury).
- Sec 16: Added Sample prep method modification (i.e. final volume).

- Revision 0, dated 04 February 2011
  New

Attachment 1

Certificate of Analysis

Catalogue Number :	141-110-111/141-110-112/141-110-115
Description :	*Plasma*CAL - **ICP-MS Verification Standard 1 Solution B**
Lot Number :	**SC8298812**
Expiration Date :	**January 2010**

Analysis of Solution Standard by Inductively Coupled Plasma Spectroscopy (ICP-AES) traceable to NIST Standard Reference Materials : 3133

Actual Concentrations

Hg : 9.98 µg/ml

Matrix : **10% HNO_3**
Density : **1.040 g/ml @ 22.0°C**

MS 0882
Rec'd
1/14/09

Certified by : _____
Thomas Znoj, Chemistry Manager

Certification Date : **October 28, 2008**

This ICP-AES & ICP-MS Standard is guaranteed to be stable and accurate to within plus or minus 1.0% of the actual concentration up to the expiry date, provided the solution is kept tightly capped and stored under normal laboratory conditions. For these solutions, 18 megohm/cm double deionized water, high-purity acids, Class A glassware and acid-cleaned bottles are used. The Material Safety Data Sheet and this Certificate of Analysis are available on our web site. (Également disponible en Français)

Manufactured according to an ISO 9001:2000 Quality System and ISO 17025 (in-process)

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CERTIFICATE OF ANALYSIS

MS0879
Rec'd 1-12-09

1.0 **INORGANIC VENTURES** is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001:2000 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."

2.0 **DESCRIPTION OF CRM** **10 µg/mL Mercury in 10% (v/v) HCL**

Catalog Number:	MSHG-10PPM
Lot Number:	**B2-HG02061**
Starting Material:	Hg metal
Starting Material Purity (%):	99.999549
Starting Material Lot No:	05214TX
Matrix:	10% (v/v) HCL

3.0 **CERTIFIED VALUES AND UNCERTAINTIES**

Certified Concentration: 10.027 ± 0.020 µg/mL

Certified Density: 1.019 g/mL (measured at 22° C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

$$\text{Certified Value } (\bar{x}) = \frac{\Sigma x_i}{n}$$

$$\text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{1/2}}{(n)^{1/2}}$$

$(\bar{x})$ = mean
x_i = individual results
n = number of measurements
Σs_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume, and the fixed error reported on the NIST SRM certificate of analysis.)

4.0 **TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS**

- "Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)
- This product is Traceable to NIST via an unbroken chain of comparisons to the following NIST SRMs:

Title: Digestion of Water and Wastewater Samples for Analysis by ICP and ICP-MS, SW846 Method 3005A

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

Laura Demone — 02/10/22
Laura Demone — Date
Department Manager

Dan Helfrich — 02/03/22
Dan Helfrich — Date
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Carl Armbruster — 02/03/22
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1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

Method SW846 3005A covers the preparation procedures for the determination of elemental constituents in water samples, wastewater samples by inductively coupled plasma (SW846 Method 6010D) and inductively coupled plasma – mass spectrometry (SW846 Method 6020B). Use Method 3010A for the acid digestion of leachate samples.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

The elements analyzed for by this method and the laboratory's reporting limits (RLs) are summarized below:

Element	Method 6010D Water (ug/L)	Method 6020B Water (ug/L)
Aluminum	200	40
Barium	200	4
Beryllium	2	0.8
Boron	50	80
Cadmium	4	2
Calcium	5000	500
Chromium	10	4
Cobalt	50	4
Copper	25	4
Iron	150	120
Manganese	15	8
Magnesium	5000	200
Molybdenum	20	4
Nickel	40	4
Potassium	5000	200
Silver	10	2
Sodium	5000	500
Strontium	20	4
Tin	50	16
Titanium	20	4
Vanadium	50	4
Zinc	30	16
Antimony	20	2
Arsenic	15	2
Lead	10	1.2
Selenium	20	2.5
Thallium	20	0.8
Silicon	500	n/a
Sulfur	200	n/a

2.0 Summary of Method

A volume of sample is heated with nitric and hydrochloric acids. After the sample is reduced in volume, it is filtered if necessary, diluted to volume, and is then ready for analysis by inductively coupled plasma (ICP) by SW846 Method 6010D or inductively coupled plasma-mass spectrometry (ICP-MS) analysis by SW846 Method 6020B.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Whenever reflux is required or sample is to be reduced, NEVER BOIL THE SAMPLE. Boiling could result in a significant loss of sample constituents.

4.2 See the current analytical SOPs for further information related to interferences: TestAmerica SOP Nos. ED-MT-004 *Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy by SW846 Method 6010D* and ED-MT-034, *SW-846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS*.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material [1]	Hazards	Exposure Limit [2]	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Block Digestor: Adjustable and capable of maintaining a temperature of 90ºC-95ºC (Environmental Express or equivalent).
- AutoBlock: Environmental Express

6.2. Supplies

- watch glasses
- 75 mm funnels
- 50 ml and 100 ml Hot Block Digestion Cups
- Whatman # 41 filter paper or equivalent
- 0.45 micron filter units and vacuum pump
- Pipettes varying volumes: Eppendorfs & Fisher
- Reflux Caps, p/n SC506 Environmental Express or equivalent

7.0 Reagents and Standards

7.1. Reagents

7.1.1. Nitric Acid, Concentrated - Trace Grade or equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.2. Hydrochloric Acid, Concentrated - Trace Grade or equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.

7.1.3. Reagent Grade Water 18 Megohm Minimum

7.2. Standards

7.2.1 Stock ICP Spike Standards:

7.2.1.1 Solutions A, B, C, and D (ICP-Spk), Part No. 4400-100419DD01: commercially purchased from CPI International. Store at room temperature; for stability information, refer to manufacturer's instructions. See Attachment 1 for the standards certified concentrations and catalog numbers. 1000ppm each of Silicon and Sulfur purchased from CPI International or equivalent.

7.2.2 Stock ICP-MS Spike Standards:

7.2.2.1 ICPMS LCS/SPK, Part No. SM-606-111: commercially purchased from High-Purity Standards. Store at room temperature; for stability information, refer to manufacturer's instructions. This stock standard doesn't require any pre-dilutions and is ready to be spiked directly into the QC samples. See Attachment 1 for the standards certified concentrations and catalog numbers.

7.3 Working Spike Standards:

7.3.1. ME_LCS-int (for ICP prep) - Add the following to a 1000 ml volumetric flask and bring to volume using 5% HNO_3: 50 ml each of Part No. 4400-100419DD01 Solutions A, B, C, and D (Sec 7.2.1.1). Record standard preparations in TALS Reagent module. **Note**: Standard must not exceed the earliest expiration date of any one of its components.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample[4] Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Water	Polyethylene, glass	50 ml	HNO_3 to pH < 2 prior to shipment; if not, acidify upon receipt in lab [2,3]	180 Days	40 CFR Part 136.3

[1] Inclusive of digestion and analysis.
[2] Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 24 hours. Just prior to digestion or direct analysis, verify pH<2. If pH≥ 2, repeat steps (i.e., add acid, hold for 24hrs, verify ph<2).
[3] Aqueous samples may be stored at room temperature.
[4] All containers must be pre-washed with detergents, acids and water.

9.0 Quality Control

9.1. Sample QC - The following quality control samples are prepared with every 20 samples or every batch of samples digested whichever comes first.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	See the Quality Control Section of the referring analytical SOP (i.e., SOP No. ED-MT-004 and ED-MT-034)
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	
Matrix Spike (MS)[1]	1 in 20 or fewer samples	
Sample Duplicate (DUP)[1]	1 in 20 or fewer samples	
Matrix Spike Duplicate (MSD)	When requested by the client	

[1] The sample selection for MS and Sample Duplicate is random unless specifically requested by a client. Quality control samples prepared with each set of samples digested include Matrix Spike, Sample Duplicate, Method Blank, Laboratory Control Sample and Matrix Spike Duplicate (MSD: when requested by the client). Use the same environmental sample for the matrix spike and duplicate sample whenever possible. If insufficient sample volume is available, another environmental sample may be used for the duplicate sample.

9.2 Instrument QC

None

10.0 Procedure

10.1. Sample Preparation

10.1.1. Filtration Procedure for Dissolved Metals not filtered in the Field

10.1.1.1. The unpreserved sample must be filtered through a 0.45um filter unit as soon as practical after collection.

10.1.1.2. Collect the required volume of filtrate by using a 0.45um filter unit and a vacuum pump.

10.1.1.3. Acidify the filtrate with 1:1 HNO_3 to a pH of <2.

10.1.1.4. The method blank (MB) must be filtered and digested under the same conditions as the lab filtered samples.

10.1.2. During digestion, at least 2-3 ml of sample solution must be maintained in the digestion cup. Sample must never be allowed to go to dryness. If sample should go to dryness, discard sample and redigest.

10.1.3. Pour out 50 ml of well-mixed sample into a 50 ml digestion cups *[note:100 mL hot block digestion cups can be used, except use the same sample volume (50 mL) for the entire preparation batch].* Label the cup with the sample number using a permanent marker. Prepare the QC samples as follows.

10.1.3.1. Method Blank (MB): Pour out 50 ml of deionized reagent water into the 50 ml digestion cup. Label the cup MB mm/dd/yy (this being the date the sample was prepared) and batch number. The Method Blank is carried through the complete digestion process.

10.1.3.2. Matrix Duplicate (DU): Label the digestion cup assigned for the duplicate sample with sample number and suffix 'DU'. Pour 50 ml of the well-mixed sample.

10.1.3.3. Matrix Spike Sample (MS): Measure 50 ml of well-mixed sample into the appropriately labeled 50 ml digestion cups (cup is labeled with the sample number and suffix 'MS'). The resulting elemental concentrations are listed in Attachments 2 & 3.

- For ICP preps: spike the ICP Matrix Spike sample with 1 ml of ME_LCS-int (Sec 7.3.1). If Silicon and Sulfur are needed, spike 0.2 mL each of 1000ppm Sulfur and Silicon.

- For ICP-MS preps: spike the sample with 0.25 ml ICPMS LCS/SPK (Sec 7.2.2.1).

10.1.3.4. Laboratory Control Sample (LCS): Pour out 50 ml of deionized reagent water sample into the appropriately labeled 50 ml digestion cups. Label the cup 'LCS' and the batch number. Spike the ICP-LCS and ICPMS- LCS (as appropriate) in the same way as the Matrix Spike Sample, see Sec. 10.1.3.3. Refer to Attachments 2 & 3 for final elemental concentrations.

10.1.4. Using Hot Block (if using AutoBlock, skip to section 10.1.5)

10.1.4.1. Add 1 ml concentrated HNO_3 and 2.5 ml of hydrochloric acid, cover with ribbed watch glass, place digestion cup on hot block at 90-95 ^oC, and reduce volume to approximately 15-20 ml. Do not allow sample to boil or dry.

10.1.4.2. Allow sample to cool down.

10.1.4.3. Wash down watchglass and digestion cup walls with deionized water. If necessary, accurately label a new 50 ml sample cup and filter samples through a #41 Whatman filter paper to remove any silicates and other insoluble material that could clog the nebulizer on the instrument. Bring the sample volume up to 50 ml.

10.1.4.4. Enter all batch/sample information into TALS (see Sec 11.4. for instructions). Print the sample labels from the batch and attach to the corresponding sample cups. These labels contain information such as: Job & sample number, Batch number and container number.

10.1.5. Using AutoBlock

10.1.5.1. Place a Reflux Cap (sec 6.2) on every 50 ml hotblock cup.

10.1.5.2. Turn on AutoBlock then click the *AutoBlock Plus* icon on the computer desktop.

10.1.5.3. Check the waste drum. If needed, properly dump the waste.

10.1.5.4. Check the reagents (i.e., HCL, HNO3, and Deionized Water). Refill if necessary.

10.1.5.5. Click *Operator Test* and click *Select Method.*

10.1.5.6. Select *3005A-Waters.mtd*

10.1.5.7. Under *Cell Position Selection*, click the hotblock locations where you will have the samples.

10.1.5.8. After closing the see-through plastic door, click on *Start Method.*

10.1.5.9. Don't open the door until the method has been completed.

10.1.5.10. When finished, *Method Completed* will be displayed in the software.

10.1.5.11. Wash down the watchglass and digestion cup walls with deionized water. If necessary, accurately label a new 50 ml sample cup and filter samples through a #41 Whatman filter

paper to remove any silicates and other insoluble material that could clog the nebulizer on the instrument. Bring the sample volume up to 50 ml.

10.1.5.12. Enter all batch/sample information into TALS (see Sec 11.4. for instructions). Print the sample labels from the batch and attach to the corresponding sample cups. These labels contain information such as: Job & sample number, Batch number and container number.

10.2. Calibration

10.2.1. The volume of the 50ml and 100 ml hotblock digestion cups are verified for each lot received. The 50 ml and 100 ml volume verification with the appropriate Lot number is documented in the 'Metals Labware Volume Verification Logbook.'

10.2.2. Per manufacturer's instructions, every 30 days, recalibrate the AutoBlock:

10.2.2.1. Place an empty hotblock cup on an analytical balance and 'zero' the balance.
10.2.2.2. Place that empty hotblock cup in the first position, I1, in the AutoBlock.
10.2.2.3. Click the *AutoBlock Plus* icon on the computer desktop
10.2.2.4. Click the *Manuel Screen* tab.
10.2.2.5. Under *Reagent Selection*, click *H2O(6).* This is Port 6 which contains Deionized Water.
10.2.2.6. Under *Cell Position Selection,* click *I1.*
10.2.2.7. Under *System Control*, click *Move to Position.*
10.2.2.8. In the *Injection Amount (ml)* box, type "30" and press *Inject.* The Autoblock will dispense 30 mL of DI Water into the cup.
10.2.2.9. Click *Move to Drain* and press *Exit.*
10.2.2.10. Weigh the cup containing the DI Water on the zeroed balance to determine the weight of the water.
10.2.2.11. Press the *Service Screen* tab.
10.2.2.12. Press the *Pump Cal* tab
10.2.2.13. Under *Pump 2 Actual Injection Amount (ml)*, enter the weight of the water and press *Pump 2 Save Cal.*

10.3. Sample Analysis

10.3.1. Refer to the Analytical SOP, TestAmerica Edison SOP No. ED-MT-004, and SOP No. ED-MT-034.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Concentration:

Refer to TestAmerica SOP No. ED-MT-004 and ED-MT-034.

11.4. Documenting and Reporting

11.4.1. Sample preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS).

11.4.1.1. Double click the TALS icon on the computer desktop. Enter your username and password and select Login.

11.4.1.2. Under TALS Menu, click the 'plus' sign next to Analyst. Double click Analyst Desktop II. Click the 'plus' sign next to Methods.

11.4.1.3. Right click the prep method that you are using to prep the samples. Select Create New Batch-From Scratch.

11.4.1.4. The analyst must enter the following information: Sample names (use scanners), initial and final sample volume, spike name and amount used, and all reagents and their corresponding lot numbers.

11.4.1.5. After saving the batch information in TALS, select Print-Batch Sheets (which must be submitted to metals analytical dept. along with the samples) and select Print-Batch Labels (which must be attached to the corresponding final digestate sample cups in the batch).

11.4.2. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable preparation batch.

11.4.2.1. Open the preparation batch and click on the Edit tab above to enter the Edit Mode.

11.4.2.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.4.2.3. Acknowledge by filling in responses to all unacknowledged findings.

11.4.2.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.4.2.3.2. Fill in the appropriate comments in the response box, then hit 'OK.'

11.4.2.3.3. Acknowledge all finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization. The neutralization system consists of a 55-gallon poly open top container. There is also a local snorkel exhaust for venting any noxious odors. Once the liquid is transferred into the container, the contents are sparged with air and a representative pH is measured and the amount of material is recorded in the discharge log. The pH value is recorded in the discharge log, and the liquid is neutralized to a pH of 6-9 SU using food grade sodium bicarbonate. Once the secondary pH has been taken, confirmed and recorded, the discharge valve is opened to the municipal sewer system and the neutralization process is complete. The date, type of waste stream, and volume discharged are recorded in the discharge log.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  TSDF under Veolia Profile Number: 590598

- Soil Retain Samples - These containers are stored in the main sample receiving laboratory cooler until they are designated for disposal. Once designated for disposal, the samples are transferred to the waste disposal area and placed into cubic-yard disposal containers, e.g., Jupiter-boxes. This waste is shipped directly to the TSDF under Veolia Profile Number 402535 and is subject to incineration.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed.,U.S. Environmental Protection Agency, SW846 Manual, Method 3005A, Revision 1, July 1992.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision

15.4. TestAmerica Edison SOP No. ED-MT-004, *Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy by SW846 Method 6010D*, most current revision.

15.5. TestAmerica Edison SOP No. ED-MT-034, *SW-846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment and Leachate Samples by ICP-MS,*

most current revision.

15.6. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.7. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16.0. Method Modifications:

Sample initial and final volumes are modified to 50 ml which allows for lesser sample volume needed for digestion and less sample waste produced.

17.0. Attachments

Attachment 1: Certificate of Analysis of ICP and ICPMS stock standard.
Attachment 2: ICP: Laboratory Control Sample and Matrix Spike Final Concentration in Soln (ug/L)
Attachment 3: ICP-MS Matrix Spiking Solution, ICPMS LCS/SPK

18.0. Revision History

- Revision 8, dated 10 February 2022
  - SOP reinstated, updated all applicable sections to reflect the most current procedures for QC, Reagents, Standards, Calibration, Reporting and SOP references.

- Revision 7, dated 10 November 2011
  - Sec 7.2.1.1: Updated the stock ICP spike standards to reflect actual lab practices.
  - Sec 7.2.2.1: Updated the ICPMS LCS/SPK which is now purchased from High-Purity Standards.
  - Sec 7.3.1: Updated preparation procedure for ME-LCS-int to reflect actual lab practices.
  - Sec 8: Added footnote #4: 24 hr waiting period after lab acid preservations.
  - Sec 10.1.1.4: Added procedures for filtering Method blanks for dissolved metals.
  - Sec 10.1.8: Added instructions for adding TALS' generated labels to digestion cups
  - Sec 11.4: Expanded procedures for documenting and reporting information via TALS.
  - Attachment 2: Updated the ICPMS final concentration for Boron and Zinc in *LCS & Matrix Spike, Final Concentrations in Solutions* Table.
  - Attachment 3: Updated COA's of stock standards.
  - Sec 15: Added applicable references.

- Revision 6, dated 20 October 2009

  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec1.1: Deleted analysis by FLAA spectroscopy and added analysis by ICP-MS.
  - Sec 1.2: Added reporting limit for all elements analyzed by this method.
  - Sec 6: Replaced hot plate with block digestor. Deleted glasswares, (graduated

cylinders, beakers, volumetric flasks), supplies not applicable.

- Sec. 7.2 & 7.3: Revised the source of the stock spike standards and the preparation instructions for the ICP working spike standards.
- Sec 10: Replaced beakers with hot block digestion cups
- Sec 15: Applicable references added.
- Sec 17: Attachments added.

Attachment 1

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SOLN. A
DE
4-4-11

Expiry: 21-Sep-12

Certificate of Analysis

Date Rec'd
03/22/11 MP

Part Number: **4400-100419DD01**
Lot Number: **11C157**
Shelf Life: **18 Months**

TestAmerica/Edison
Custom Standard
5% HNO3

Concentrations in ug/mL ± 0.5%

Fe	1000	Zn	500
Se	2000	Mn	500
TL	2000	NI	500
Be	50		
Cd	50		
Cr	200		
Co	500		
Cu	250		
Pb	500		
Ag	50		
Sr	500		
V	500		

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com.

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Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution B**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
03/22/11 MP

TestAmerica/Edison
Custom Standard
10% HNO3

Concentrations in ug/mL ± 0.5%

Al	2000
Mg	20000
K	20000
Ca	20000
Na	20000

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution D**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
05/22/11 MH

TestAmerica/Edison
Custom Standard
2% HNO3

Concentrations in ug/mL ± 0.5%

As	2000
Ba	2000
B	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution C**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
03/22/11 MP

TestAmerica/Edison
Custom Standard
5% HNO3 + 2% HF

Concentrations in ug/mL ± 0.5%

Sb	500
Mo	500
Sn	500
Ti	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megachm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.com.

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Certificate of Analysis

SM-606-111
(ICPMS LCS/SPK)
Lot # 1031503

Source	Source Purity	Matrix	Standard Concentration
High Purity Metals, Salts and Oxides	99.98+%	HNO_3, 4% + Tr HF	µg/mL ± 0.5% See element list on reverse

This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodore C. Rains

Theodore C. Rains, Ph.D.
President

Exp Date: NOV 15 2011
MSDS ATTACHED

SM-606-111
(ICPMS LCS/SPK)
Element List
(µg/mL)

Aluminum	500
Antimony	5
Arsenic	10
Barium	10
Beryllium	5
Boron	100
Cadmium	5
Calcium	500
Chromium	10
Cobalt	5
Copper	10
Iron	500
Lead	5
Magnesium	500
Manganese	50
Molybdenum	10
Nickel	10
Potassium	500
Selenium	10
Silver	5
Sodium	500
Strontium	10
Thallium	4
Tin	10
Titanium	10
Vanadium	10
Zinc	50

Attachment 2

ICP: LCS & Matrix Spike Final concentration in solution	
ELEMENT	ug/L
Aluminum	2000
Antimony	500
Arsenic	2000
Barium	2000
Beryllium	50
Cadmium	50
Calcium	20000
Chromium	200
Cobalt	500
Copper	250
Iron	1000
Lead	500
Manganese	500
Magnesium	20000
Nickel	500
Potassium	20000
Selenium	2000
Silver	50
Sodium	20000
Thallium	2000
Vanadium	500
Zinc	500
Boron	500
Molybdenum	500
Tin	500
Titanium	500
Strontium	500
Silicon	4000
Sulfur	4000

Attachment 3

ICPMS Matrix Spiking Solution, ICPMS LCS/SPK	
Element	**Matrix Spike Conc. (ug/L)**
Aluminum (Al)	2500
Antimony (Sb)	25
Arsenic (As)	50
Barium (Ba)	50
Beryllium (Be)	25
Boron (B)	500
Cadmium (Cd)	25
Calcium (Ca)	2500
Chromium (Cr)	50
Cobalt (Co)	25
Copper (Cu)	50
Iron (Fe)	2500
Lead (Pb)	25
Magnesium(Mg)	2500
Manganese (Mn)	250
Molybdenum (Mo)	50
Nickel (Ni)	50
Potassium (K)	2500
Selenium (Se)	50
Silver (Ag)	25
Sodium (Na)	2500
Strontium (Sr)	50
Thallium (Tl)	20
Tin (Sn)	50
Titanium (Ti)	50
Vanadium (V)	50
Zinc (Zn)	250

Title: Hot Block Digestion of Sediments, Sludges, and Soils by USEPA Method No(s). SW846 3050B

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits

This SOP covers the acid digestion procedure (SW846 3050B) for the determination of total recoverable elemental constituents in sediments, soils and sludges by inductively coupled plasma emission spectroscopy (EPA Method 6010D) and Inductively Coupled Plasma – Mass Spectrometry (EPA Method 6020B).

1.2. For a complete list of analytes and Reporting limits, refer to the appropriate analytical TestAmerica SOPs: SOP ED-MT-004, *Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy, Method No. SW846 Method 6010D;* SOP ED-MT-029; *Trace Metals Analysis for Water, Wastewater, Soil,Sediment and Leachate Samples by ICP-MS Method SW-846 6020B and* SOP ED-MT-034, *SW846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment, and Leachate Samples by ICPMS*

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

A portion of sample is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added to the digestate and the sample is refluxed and then brought to a final volume of 100 ml. The resulting digestate is analyzed by ICP-AES or ICP-MS.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences are discussed in the applicable analytical SOPs. Spiked samples are prepared to determine if any interference are present.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the

assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

Hydrogen peroxide (H2O2) is a strong oxidizer and is corrosive. The digestion must be cooled sufficiently before the addition of H2O2 to avoid a reaction and possible violent effervescence, or boiling over of the digestion. A splash/splatter hazard is possible and a face shield should be worn.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm-Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrogen Peroxide	Oxidizer Corrosive	1 ppm-TWA	Vapors are corrosive and irritating to the respiratory tract. Vapors are very corrosive and irritating to the eyes and skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Block digestor: Environmental Express or equivalent
- AutoBlock: Environmental Express
- Weighing balance capable of accurate weighing to 0.01g.

6.2. Supplies

- watch glasses
- 75 mm funnels
- 50 ml Hot Block Digestion Cups
- Whatman # 41 filter paper or equivalent
- Pipettes varying volumes: Eppendorfs & Fisher
- 4oz Snap Cap Cups from Fisher or equivalent
- Reflux Caps, p/n SC506 Environmental Express or equivalent

7.0 Reagents and Standards

7.1. Reagents

7.1.1. Concentrated Nitric Acid- Trace Grade or equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.
7.1.2. Concentrated Hydrochloric Acid- Trace Grade or equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.
7.1.3. Reagent Grade Water 18 Megohm Minimum
7.1.4. Hydrogen peroxide (30%), H_2O_2. Store at room temperature; for stability information, refer to manufacturer's instructions.

7.2. Standards -

Storage requirements: all standards are stored at room temperature
Shelf-life: Stock standards – refer to manufacturer's instructions
Intermediate standards – 12 months
Working standards – 12 month
(Note: expiration date must not go beyond the expiration date of the source stock).
Concentration: see Attachment 3 for example certificates of analysis (COA) for all of the standards mixes listed below. The COA lists the manufacturer's part number, certified concentration and shelf life.

Standards must be prepared every 12 months or sooner if needed or required. "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria.

7.2.1. Stock ICP Spike Standards:

- Solutions A, B, C, and D (ICP-Spk), Part No. 4400-100419DD01: commercially purchased from CPI International. Store at room temperature; for stability information, refer to manufacturer's instructions. See Attachment 1 for the standards certified concentrations and catalog numbers.
- Sulfur 1000 mg/L, single element standard commercially purchased from CPI International. Store at room temperature; for stability information, refer to manufacturer's instructions

7.2.2. Working Spike Standard, ICP Working Spike Solution ME_ LCS-int (for ICP prep) - Add the following to a 1000 ml volumetric flask and bring to volume using 5% HNO_3: 50 ml each of Part No. 4400-100419DD01 Solutions A, B, C, and D (Sec 7.2.1.). Record standard preparations in TALS Reagent module. **Note**: Standard must not exceed the earliest expiration date of any one of its components.

7.2.3. Stock ICPMS Spike Standards (Method 6020B)

- ICPMS LCS/SPK: commercially purchased from High-Purity Standards. Store at room temperature; for stability information, refer to manufacturer's instructions. This stock standard doesn't require any pre-dilutions and is ready to be spiked directly into the QC samples. See Attachment 3 for the standards certified concentrations and catalog numbers.

7.2.4. Laboratory Control Sample Reference Material (LCSSRM), Metals in Soil: obtained from Environmental Resource Associates. Store at room temperature; for stability information, refer to manufacturer's instructions.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container[1]	Min. Sample Size	Preservation	Holding Time[2]	Reference
Soils	Glass, plastic	5 grams	Cool 4 ±2°C	6 months	SW846 Method 3050B

[1] All sample containers must be demonstrated to be free of contaminants below the reporting limit.
[2] Holding time is 6 months, however sample analysis should begin as soon as possible.

9.0 Quality Control

9.1. Sample QC - The following quality control samples are prepared with each batch of samples digested or with every Sample Delivery Group (SDG), whichever comes first.

Quality Control	Frequency	Control Limit: Method 6010D & 6020B
Method Blank (MB)	1 in 20 or fewer samples	See the quality control section of the associated analytical SOP
Laboratory Control Sample(LCSSRM)	1 in 20 or fewer samples	Vendor's certified limits
Matrix Duplicate (DU)	1 in 20 or fewer samples	See the quality control section of the associated analytical SOP
Matrix Spike (MS)[1]	1 in 20 or fewer samples	75-125%
Matrix Spike Duplicate (MSD)[1]	When requested by client	See the quality control section of the associated analytical SOP

[1] The sample for DU/MS/MSD is randomly selected, unless specifically requested by a client; predetermined by the prep lab. Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.
[2] Statistical control limits are updated annually and are updated into LIMS.

9.2. Instrument QC

None

10.0 Procedure

10.1. Sample Preparation

10.1.1. All digestion cups and filtration apparatus related to a sample must be labeled with that sample number.

10.1.2. Whenever reflux is required, sample should never be allowed to boil or "bump" as this could result in significant loss of analyte. Sample must never be allowed to go to dryness or be baked.

10.1.3. Mix the sample thoroughly to achieve homogeneity. Weigh 1.00-1.50

grams of sample to the nearest .01 g into a 50 ml hot block digestion cup. QC samples are prepared as follows:

10.1.3.1. Method Blank (MB): Use a blank 50 ml digestion cup for the Method Blank. Attach the TALS (TestAmerica Laboratory Systems) generated label which contains the sample ID number and batch number. The Method Blank is carried through the complete digestion process.

10.1.3.2. Matrix Duplicate (DU): Weigh 1.00-1.50 gm of a duplicate sample to the nearest 0.01 gram into a 50 ml hot block digestion cup. Attach the TALS generated label for the matrix duplicate sample.

10.1.3.3. Matrix Spike Sample (MS): Weigh 1.00 -1.50 gram of duplicate sample to the nearest 0.01 gram into a 50 ml hot block digestion cup. Attach the TALS generated label for the matrix spike sample.

- For ICP preps: Spike 2 ml of ME_LCS-int solution (ICP predigestion spike, Sec 7.2.2) to the digestion cup. If Sulfur is needed, also spike 0.4 mL of Sulfur 1000 mg/L (sec 7.2.1). The resulting elemental amounts are listed in Table 1 (attachment 1). NOTE: the spike must be added prior to the addition of any acids.
- For ICP-MS preps: spike the ICP-MS QC sample with 1.0 ml ICPMS LCS/SPK (Sec 7.2.3). The resulting elemental concentrations are listed in Attachment 2

10.1.3.4. Laboratory Control Sample Reference Material (LCSSRM): Weigh out 1.00-1.50 gm of LCS (sec 7.2.6) to the nearest 0.01 grams into a 50 ml hot block digestion cup. Attach the TALS generated label for the laboratory control sample.

10.1.3.5. Laboratory Control Sample (LCS) for Sulfur:
Using a 50 mL hot block cup, spike 0.4 mL of 1000 mg/L Sulfur (sec 7.2.1).

10.1.3.6. Matrix Spike Duplicate (MSD): when a MSD is requested by the client, prepare the sample in the same manner as the Matrix spike sample (MS), see Matrix Spike Sample prep instructions (Sec 10.1.3.3)..

10.1.4. Using Hot Block (if using AutoBlock, skip to section 10.1.5)

10.1.4.1. Add 10 ml of 1:1 HNO_3, mix the slurry, and cover with a watchglass. Transfer digestion cup to a hot block with the dial setting so that the contents of digestion cup are at

approximately 95°C ±3
°C and reflux for 10 to 15 minutes without boiling or drying the sample.

10.1.4.2. Allow sample to cool, add 5 ml of concentrated HNO_3, replace the watchglass, and reflux for 30 minutes.

10.1.4.3. Repeat step above until no brown fumes are generated.

10.1.4.4. Using a ribbed watchglass, cover the vessel and allow the solution to either evaporate to approximately 5ml or evaporate for 2 hours without boiling. Do not allow sample to go to dryness.

10.1.4.5. After step 10.1.7 has been completed, cool the sample; add 2 ml of deionized water and 3 ml of 30% H_2O_2. Warm cup, and make certain no losses occur due to vigorous effervescence. Heat until effervescence subsides and cool cup.

10.1.4.6. Continue to add 30% H_2O_2 in 1.0 ml aliquots with warming until the effervescence is minimal until the general sample appearance is unchanged. *Note*: Do not add a total of more than 10 ml of 30% H_2O_2.

10.1.4.7. Using a ribbed watchglass, cover the vessel and allow the solution to either evaporate to approximately 5ml or evaporate for 2 hours without boiling. Do not allow sample to go to dryness.

10.1.4.8. Add 10 ml of concentrated HCL to the sample and reflux for 15 min at 95°C ±3.

10.1.4.9. Filter the sample through Whatman 41 filter paper or equivalent into a 4oz Fisher snap cap cup or equivalent. Rinse the 50ml cup using DI water and filter into the 4oz cup. Using deionized water, bring to a final volume of 100 ml. This digestate is now ready for ICP or ICP-MS analyses.

10.1.5. Using AutoBlock

10.1.5.1. Place a Reflux Cap (sec 6.2) on every 50 ml hotblock cup.

10.1.5.2. Turn on AutoBlock then click the *AutoBlock Plus* icon on the computer desktop.

10.1.5.3. Check the waste drum. If needed, appropriately dump the waste.

10.1.5.4. Check the reagents (i.e., HCL, HNO3, 1:1HNO3, 30% H2O2, Deionized Water). Refill if necessary.

10.1.5.5. In the software, click the *Manuel Screen* tab. Enter 95 in the *Temp Control* box. Click *Enable Heat.*

10.1.5.6. Place a 50ml hotblock cup filled with DI water on the autoblock. Cover with a reflux cap. After the hotblock has reached a stable temperature of 95 deg C (~15min), record the temperature using a calibrated thermometer.

10.1.5.7. In the software, click *Exit.*

10.1.5.8. Click *Operator Test* and click *Select Method.*

10.1.5.9. Select *SOILmethod3050B.*

10.1.5.10. Under *Cell Position Selection*, click the hotblock locations where you will have the samples.

10.1.5.11. After closing the see-through plastic door, click on *Start Method.*

10.1.5.12. Don't open the door until the method has been completed.

10.1.5.13. Watch for brown fumes after Nitric Acid has been added by the autoblock. If brown fumes are generated, make note to discard this sample. This sample must be prepared using a Hot Block (sec 10.1.4).

10.1.5.14. When finished, *Method Completed* will be displayed in the software. Record the temp (sec 10.1.5.5).

10.1.5.15. Filter the sample through Whatman 41 filter paper or equivalent into a 4oz Fisher snap cap cup or equivalent. Rinse the 50ml cup using DI water and filter into the 4oz cup. Using deionized water, bring to a final volume of 100 ml. This digestate is now ready for ICP or ICP-MS analyses.

10.2. Calibration

10.2.1. The volume of the 100ml cups are verified for each lot received. The 100 ml volume verification with the appropriate Lot number is documented in the 'Metals labware volume verification logbook.'

10.2.2. Per manufacturer's instructions, recalibrate the AutoBlock every 30 days.

10.2.2.1. Place an empty hotblock cup on an analytical balance and 'zero' the balance.
10.2.2.2. Place that empty hotblock cup in the first position, I1, in the AutoBlock.
10.2.2.3. Click the *AutoBlock Plus* icon on the computer desktop
10.2.2.4. Click the *Manuel Screen* tab
10.2.2.5. Under *Reagent Selection*, click *H2O(6).* This is Port 6 which contains Deionized Water
10.2.2.6. Under *Cell Position Selection,* click *I1*
10.2.2.7. Under *System Control*, click *Move to Position*
10.2.2.8. In the *Injection Amount (ml)* box, type "30" and press *Inject.* The Autoblock will dispense 30 mL of DI Water into the cup.
10.2.2.9. Click *Move to Drain* and press *Exit*
10.2.2.10. Weigh the cup containing the DI Water on the zero'd balance to determine the weight of the water
10.2.2.11. Press the *Service Screen* tab
10.2.2.12. Press the *Pump Cal* tab
10.2.2.13. Under *Pump 2 Actual Injection Amount (ml)*, enter the weight of the water and press *Pump 2 Save Cal.*
10.2.2.14. Document the calibration on the Autoblock maintenance logbook.

10.3. Sample Analysis

Refer to the appropriate Analytical SOPs listed in Sec 1.2.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Concentration :

Refer to appropriate Analytical SOPs listed in Sec 1.2.

11.4. Documenting and Reporting

11.4.1. Sample preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS).

11.4.1.1. Double click the TALS icon on the computer desktop. Enter your username and password and select Login.

11.4.1.2. Under TALS Menu, click the plus sign ('+') next to Analyst. Double click Analyst Desktop II. Click the plus sign ('+') next to Methods.

11.4.1.3. Right click the prep method that you are using to prep the samples. Select 'Create New Batch' then select 'From Scratch.'

11.4.1.4. The analyst must enter the following information: Sample names (use scanners), initial and final sample volume, spike name and amount used, and all reagents and their corresponding lot numbers.

11.4.1.5. After saving the batch information in TALS, select Print-Batch Sheets (which must be submitted to metals analytical dept. along with the samples) and select Print-Batch Labels (which must be attached to the corresponding final digestate sample cups in the batch).

11.4.2. Complete the Data Review Checker (DRC) in TALS: Prior to data submission (i.e., prior to 1st leveling data in the Analyst Desktop II module in TALS), the analyst must execute the DRC (Data Review Checker) program in the applicable preparation batch.

11.4.2.1. Open the preparation batch and click on the Edit tab above to enter the Edit Mode.

11.4.2.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.4.2.3. Acknowledge by filling in responses to all unacknowledged findings.

11.4.2.3.1. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.4.2.3.2. Fill in the appropriate comments in the response box, then hit 'OK.'

11.4.2.3.3. Acknowledge all Finding items in the 'Manual Batch Checklist' except for the *"2nd Level review complete?"* this is to be completed by the 2nd level reviewer.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the

primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

- Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.
  Onyx Profile WIP Number: 590598
  Teris Profile Number 50016653

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; SW-846, Method 3050B.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-MT-004, Trace Metals Analysis by Inductively Coupled Plasma Emission Spectroscopy via SW846 Method 6010D, most current revision.

15.6. TestAmerica Edison SOP No ED-MT-029, Trace Metals Analysis for Water, Wastewater, Soil,Sediment and Leachate Samples by ICP-MS Method SW-846 6020B, most current revision.

15.7. TestAmerica Edison SOP ED-MT-034, *SW846 Method 6020B, Trace Metals Analysis of Water, Wastewater, Soil, Sediment, and Leachate Samples by ICPMS*, most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Table 1, Matrix Spike concentration for Soil samples by 3050B-ICP
Attachment 2: Table 2, Matrix Spike concentration for Soil samples by 3050B-ICPMS
Attachment 3: Certificate of analysis of each stock standard.

18.0. Revision History

- Revision 15, dated 29 September 2021
  - Sec. 10.1.3.3: Revised the amount of ICPMS LCS/SPK added for ICP-MS preps to 1.0 ml to reflect actual laboratory practices.

- Revision 14, dated 30 December 2019
  - Sec 6.1: Added AutoBlock to the instrument list.
  - Sec 6.2: Added Reflux Caps to the supplies list
  - Sec 7.2.1: Added Sulfur to the stock ICP spike standards list.
  - Sec 10.1.3.3: Added matrix spiking instructions for Sulfur.
  - Sec 10.1.3.5: Added LCS preparation instructions for Sulfur.
  - Sec 10.1.5: Added instructions for using the AutoBlock.
  - Sec 10.2.1: Replaced the 50ml volume verification with a 100ml volume verification.
  - Sec 10.2.2: Added procedure for the monthly recalibration (volume dispenser) of the Autoblock.
  - Table 1: Added spike amount for Sulfur.

- Revision 13, dated 05 August 2019
  - Updated SOP header with Eurofins emblem.
  - Throughout the SOP deleted all references to Methods 6010B, 6010C, 6020 and 6020A.
  - Sec 2.0: Revised the final volume of digestates from 50ml to 100ml to reflect actual lab practices.
  - Sec 6.2: Added 4oz snap seal cups to the list of supplies.
  - Sec 10.1.3.3: Revised the spiking volume to add for ICP-MS LCS from 1ml to 2ml.
  - Sec 10.1.12: Clarified instructions for filtering digestates; added final volume of digestates.

- Revision 12, dated 06 September 2018
  - Throughout the SOP - revised to reference the analytical methods 6010D and 6020B.
  - Sec 11.4.: Updated instructions for documenting and reporting preparation batch information in TALS and instruction for using TALS Data Review Checker (DRC) program.

- Revision 11, dated 13 July 2016

  - Sec 1.1: Added method 6010C and 6020A
  - Sec 1.2: Deleted reference to SOP ED-MT-033; SOP has been combined with SOP ED-MT-004.
  - Sec 9.1: Added MSD to the list of Sample QC including the frequency and control limit requirements.
  - Sec 10.1.3.5: Added preparation instructions for MSD.
  - Sec 15.7: Removed reference to SOP No ED-MT-033, subsequent section adjusted accordingly.

- Revision 10, dated 05 June 2014

  - Sec 1.2: Removed SOP reference ED-MT-031 and added SOP reference ED-MT-033 and ED-MT-034.
  - Sec 2.0: Corrected typo error for the final volume from 100 ml to 50 ml as described in this SOP.
  - Sec 6.2: Removed 125 ml specimen cups.
  - Sec 10.1.1: Deleted the use of glassware – not applicable for this SOP.
  - Sec 15.7 & 15.8: Added TestAmerica Edison SOP No. ED-MT-033 and ED-MT-034 to the list of reference.
  - Attachment 1: Added Boron to the ICP matrix spike list.
  - Attachment 2: Added Zinc to the ICPMS matrix spike list and deleted Mercury from this list; corrected Boron's matrix spike concentration.

- Revision 9, dated 31 May 2012

  - Sec 1.3 and 12: Revised LQM references to reflect the most current LQM revision.
  - Throughout the document: Removed all references to CLP SOW ISM 01.2
  - Sec 6.1: added 'or equivalent'.
  - Sec 7.2.1: Updated the ICP stock standard to reflect current lab practices.
  - Sec 7.2.2: Updated the preparation of the working spike standard.
  - Sec 7.2.3: Replaced the vendor for the ICPMS stock standards to High-Purity Standards (former vendor- CPI International).
  - Deleted Sec 7.2.4 & 7.2.5 (CLP ISM01.2 Standards, not applicable); subsequent sections adjusted accordingly.
  - Sec 9.1 Table: MB and DU control limits have been updated and now reference the associated analytical SOP for the most current QC limits.
  - Sec 10.1.3.1 – 10.1.3.4: Updated the digestion cup labeling procedure using the TALS generated labels
  - Sec 10.1.12: Revised procedure to reflect actual lab practices - *samples are brought to a 50ml final volume and then filtered.* Final digestate volume revised from 100 ml to 50 ml.
  - Sec 10.2.1: Replaced the 125 ml hot-block cups with 50 ml size cups
  - Sec 13 & 14: Updated to comply with TestAmerica Corporate Quality SOP No. CW-QS-002, Writing a Standard Operating Procedure (SOP), most current revision.
  - Attachment 3: Replaced COAs to reflect the updated stock standards.

- Revision 8, dated 30 March 2010

  - Sec 1.1: Deleted Table which listed the 6010B RLs for soils; added Sec 1.2 to include list of analytical SOPs where the appropriate RLs can be found.
  - Sec 7: Added matrix spike and LCS standards for Method 6020 and ISM01.2
  - Sec 8: Added the holding time for soil samples analyzed by Method CLP SOW ISM01.2.
  - Sec 9.1: Added control limits for CLP SOW ISM01.2.; Deleted Blank spike and LCS duplicate.
  - Sec 10: Revised amount of sample to be weighed to 1.00-1.50 grams as per CLP Method ISM01.2
  - Sec 10: Revised hot block temperature to 95± 3 deg, to follow the more stringent setting.
  - Deleted section previously identified 10.1.3.2, Blank spike; subsequent sections adjusted.
  - Sec 10.1.3.3 & Sec 10.1.3.4: Revised to include spike procedures for ICP and ICPMS, Method 6020 and ISM01.2.
  - Sec 10.1.3.5: Deleted LCS duplicate
  - Sec 10.1.9: Section added to reflect actual lab practices; subsequent section numbers adjusted.
  - Sec 10.1.10: Text added: '*Note: For samples which are being digested for CLP SOW ISM01.2 ICP-MS, do not perform this step.'*
  - Sec 15: Added applicable references.
  - Sec 17: Added attachments 2 & 3
  - Attachment 3: Added applicable COAs
  - Added this revision history.

- Revision 7, dated June 13, 2008
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1: Added laboratory's reporting limits in Table format.
  - Section 7 Reagents & Standards: Included storage and stability information of reagents and standards. Updated the source of the stock spike standards and the preparation instructions for the working spike standards.
  - Removed all reference related to GFAA analysis including standard solutions, digestion procedure and Table 2: Spike amount for soil samples by GFAA; clarified the digestion procedure for ICP-AES analysis.
  - Section 10.1 Sample Preparation: Deleted acid rinsing of glassware since 50 ml digestion cups are being used for digesting samples.
  - Section 10.1 Sample Preparation: Replaced hot plate setting of 2.5 to hot block setting of 95°C.
  - Section 15 References: Added applicable references

Attachment 1

TABLE 1

Matrix Spike Concentration for Soil Samples by ICP	
ELEMENT	SPIKE AMOUNT (mg/Kg)
Aluminum	200
Antimony	50
Arsenic	200
Barium	200
Beryllium	5.0
Cadmium	5.0
Boron	50
Calcium	2000
Chromium	20
Cobalt	50
Copper	25
Iron	100
Lead	50
Manganese	50
Magnesium	2000
Molybdenum	50
Nickel	50
Potassium	2000
Selenium	200
Silver	5.0
Sodium	2000
Strontium	50
Thallium	200
Tin	50
Titanium	50
Vanadium	50
Zinc	50
Sulfur	400

Matrix spike sample concentration: Dry weight correction is made at final calculation

Attachment 2

ICPMS Matrix Spike Concentration Method 6020	
Element	**Matrix Spike Conc. (mg/Kg)**
Aluminum (Al)	500
Antimony (Sb)	5
Arsenic (As)	10
Barium (Ba)	10
Beryllium (Be)	5
Boron (B)	100
Cadmium (Cd)	5
Calcium (Ca)	500
Chromium (Cr)	10
Cobalt (Co)	5
Copper (Cu)	10
Iron (Fe)	500
Lead (Pb)	5
Magnesium(Mg)	500
Manganese (Mn)	50
Molybdenum (Mo)	10
Nickel (Ni)	10
Potassium (K)	500
Selenium (Se)	10
Silver (Ag)	5
Sodium (Na)	500
Strontium (Sr)	10
Thallium (Tl)	4
Tin (Sn)	10
Titanium (Ti)	10
Vanadium (V)	10
Zinc (Zn)	50

Note: For Matrix spike sample concentration, dry weight correction is made at final calculation.

Attachment 3

ERA
A Waters Company

Certificate of Analysis

Lot No. D075-540

Metals in Soil

Catalog No. 540
Issue Date: October 11, 2011
Revision Date: Original

Certification

Parameter	Total Concentration [1] (mg/kg)	Certified Value [2] (mg/kg)	Uncertainty [3]	QC PALs™ [4] (mg/kg)	PT PALs™ [5] (mg/kg)
aluminum	68100*	10100	0.8%	5110 - 15100	4560 - 15600
antimony	228	113	0.8%	DL - 252	22.7 - 286
arsenic	266	237	2.9%	197 - 278	169 - 306
barium	678	252	2.4%	212 - 292	186 - 318
beryllium	105	93.3	4.1%	77.7 - 109	69.4 - 117
boron	135	118	1.3%	87.3 - 149	71.9 - 164
cadmium	215	191	4.5%	159 - 223	140 - 242
calcium	25400*	6840	5.3%	5640 - 8040	5080 - 8600
chromium	364	128	3.8%	104 - 151	89.3 - 166
cobalt	208	178	3.5%	149 - 207	132 - 223
copper	145	123	9.0%	103 - 143	92.3 - 154
iron	42900*	13100	4.8%	6650 - 19600	4290 - 22000
lead	105	103	6.7%	85.6 - 120	73.0 - 132
magnesium	9060	2990	4.4%	2280 - 3710	1980 - 4010
manganese	918	333	5.5%	274 - 391	250 - 415
mercury	12.8	12.4	10.1%	8.88 - 15.9	6.36 - 18.4
molybdenum	113	91.1	4.8%	74.2 - 108	63.3 - 122
nickel	152	118	6.0%	96.8 - 139	85.8 - 150
potassium	19700*	2870	4.0%	1950 - 3780	1780 - 3950
selenium	123	110	1.6%	86.6 - 134	72.6 - 148
silver	51.6	47.3	3.8%	31.3 - 63.2	31.4 - 63.2
sodium	17400*	550	3.8%	405 - 694	289 - 811
strontium	252	128	4.0%	104 - 153	90.6 - 166
thallium	177	158	3.7%	128 - 188	108 - 208
tin	121	107	0.8%	84.5 - 130	61.7 - 153
titanium	3280*	282	4.8%	65.4 - 498	0.00 - 606
vanadium	205	119	4.0%	94.9 - 143	81.2 - 157
zinc	197	183	6.9%	150 - 216	127 - 239

Please see footnotes on back

DL = Detection Limit

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SOLN. A
DE
4-4-11

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Recd
03/22/11 MP

TestAmerica/Edison
Custom Standard
5% HNO3

Concentrations in ug/mL ± 0.5%

Fe	1000	Zn	500
Se	2000	Mn	500
TL	2000	Ni	500
Be	50		
Cd	50		
Cr	200		
Co	500		
Cu	250		
Pb	500		
Ag	50		
Sr	500		
V	500		

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution B**
Lot Number: **11C157**
Shelf Life: **18 Months**

TestAmerica/Edison
Custom Standard
10% HNO3

Date Recd
03/22/11 MP

Concentrations in ug/mL ± 0.5%

Al	2000
Mg	20000
K	20000
Ca	20000
Na	20000

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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The Netherlands

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution C**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
03/22/11 MP

TestAmerica/Edison
Custom Standard
5% HNO3 + 2% HF

Concentrations in ug/mL ± 0.5%

Sb	500
Mo	500
Sn	500
Ti	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

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The Netherlands

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: **4400-100419DD01** **Solution D**
Lot Number: **11C157**
Shelf Life: **18 Months**

Date Rec'd
05/22/11 MP

TestAmerica/Edison
Custom Standard
2% HNO3

Concentrations in ug/mL ± 0.5%

As	2000
Ba	2000
B	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

SM-606-111
(ICPMS LCS/SPK)
Element List
(µg/mL)

Aluminum	500
Antimony	5
Arsenic	10
Barium	10
Beryllium	5
Boron	100
Cadmium	5
Calcium	500
Chromium	10
Cobalt	5
Copper	10
Iron	500
Lead	5
Magnesium	500
Manganese	50
Molybdenum	10
Nickel	10
Potassium	500
Selenium	10
Silver	5
Sodium	500
Strontium	10
Thallium	4
Tin	10
Titanium	10
Vanadium	10
Zinc	50

Title: Extraction of Semi-Volatile Organic Compounds in Aqueous Samples and Leachates - Separatory Funnel, SW846 Method 3510C

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits** SW846 Method 3510C describes a procedure for isolating semivolatile organic compounds from aqueous samples and leachates, including concentration techniques suitable for preparing the extract for GC/MS analysis. This SOP is applicable to the isolation and concentration of water-soluble and slightly water-soluble semivolatile organics in preparation for analysis by SW846 Method 8270E.

1.2. For a complete discussion of analytes and reporting limits (RLs) please refer to Eurofins Edison SOP No. ED-MSS-009, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270E*, current revision

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. A measured volume of sample (~250 mL) is serially extracted with methylene chloride at a pH less than 2 and again at a pH greater than 11 using separatory funnel extraction. The methylene chloride extract is dried and concentrated to a volume of approximately 2 mL. Nitrogen blowdown is employed as the final concentration step. The extract is subsequently analyzed by SW846 Method 8270E (GC/MS) by a large volume injection (LVI) technique. This procedure is referred to throughout as Reduced Volume Extraction (RVE) and Large Volume Injection (LVI).

2.2. An option for preparing aqueous samples using a larger initial volume (~1000 ml) is also described. This procedure is referred to as 'large volume' throughout the document.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Solvents, reagents, glassware, and other sample hardware may yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

4.2. Phthalate esters contaminate many products commonly found in the laboratory. Plastics, in particular, must be avoided, because phthalates are often used as plasticizers and are easily extracted from plastic material. Phthalate contamination may result at any time if consistent quality control is not practiced.

4.3. The decomposition of some analytes has been demonstrated under basic extraction conditions. Phthalate esters may exchange and phenols may react to form tannates. These reactions increase with increasing pH, and are decreased by shorter reaction times. Performing the initial extraction at an acid pH will optimize the recovery of phenols

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The following analytes have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3'dichlorobenzindine, benzo(a)pyrene, alpha-BHC, beta-BHC, gamma-BHC, delta-BHC, dibenz(a,h)anthracene, N-nitrosodimethylamine, 4,4'-DDT, and polychlorinated biphenyl compounds. Primary standards of these toxic compounds should be prepared in hood.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

The use of separatory funnels to extract aqueous samples with Methylene Chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted, periodic venting may be necessary during the extraction. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, the use of a face shield over safety glasses or goggles is recommended. Keep the sash on the fume hood as low as reasonably possible.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric Acid	Corrosive Oxidizer Dehydra-dator	1 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of vapors will cause irritation of the nasal and respiratory system.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Methanol (MeOH)	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- Separatory funnel rotator, APR Machine or equivalent
- Analytical Evaporator (N-Evap) Organomation
- Centrifuge, Varifuge F; Hereaus Sepatech
- Six Position Steam Bath, Fisher 15-496 or equivalent

6.2. Supplies

- 250 ml Erlenmeyer Flask, AMK Glass ERL-0252 or equivalent
- 2000 ml or 500 ml Separatory Funnel, AMK Glass SFC or equivalent
- 100 mm o. d. glass funnels, Fisher or equivalent
- 10 ml jacketed, graduated Concentrator Tubes, AMK Glass KD-0018 or equivalent
- 19/22 Ground Glass Stoppers
- 3 Ball Snyder Columns, TEC Glass TG6-03 or equivalent

- 1 ml Gastight Syringe, Hamilton 81317 or equivalent
- 150 ml Centrifuge Tube
- 100 ml Graduated Cylinder
- Pasteur 5 ¾" Disposable Pipets, Fisher 13-678-20B or equivalent
- Kuderna Danish Flask (500 ml), TEC Glass TG7-01 or equivalent
- Vials, 2ml amber screw cap with Teflon liner
- Glass Wool
- Dessicator
- Standard Taper Clamps (Size 19, blue)
- Boiling Stones, Troemner P/N 133-B or equivalent, rinsed with Methylene Chloride
- pH Paper
- Watch Glass
- Wax Pencil
- 1 Liter Graduated Cylinder
- Marking Tags

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride, Acetone, Methanol and Sulfuric Acid is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.1.1 Methylene Chloride – JT Baker Ultra-Resi 9254-03 or equivalent

7.1.2 Acetone, J.T. Baker Ultra-Resi 9264-03 or equivalent

7.1.3 Methanol, J.T. Baker, Pesticide Grade, 9077-02 or equivalent

7.1.4 Concentrated Sulfuric Acid - Baxter 2876-9 or equivalent

7.1.5 Sodium Hydroxide Pellets – Baxter 7708-500NY or equivalent

7.1.6 Sodium Sulfate Crystals – Mallinckrodt MA8024-06 or equivalent (Must be baked in the muffle furnace for four hours at 400°C and serially rinsed with Methylene Chloride prior to use.)

7.1.7 Sodium Hydroxide (10 N) - Fill a precleaned 1000 ml volumetric flask with 500 mls deionized water. Weigh out 400 g NaOH pellets and dispense slowly into the flask. Stir slowly until the pellets dissolve, then add more deionized water until the 1000 ml level is reached. Be careful as this procedure generates heat. Never add water to the reagent that is to be dissolved.

7.2 Standards

7.2.1 Most stock target analyte standard solutions are purchased as prepared solutions; other standards are prepared in the laboratory using neat compounds (see table below). Most stock solutions are diluted (in volumetric glassware) to working concentration using methylene chloride as the diluent as described below. Stock standards of similar quality from other suppliers may be substituted as required.

NOTE: The standards listed here are used as calibration standards and spiking standards. Separate source calibration verification standards are addressed in the analytical SOPs.

Standard Name	Concentration	Vendor	Catalog #
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	RESTEK	570666
8270 List 1/ Std#10Benzoic Acid	2000ppm	RESTEK	569731
8270 List 1/ Std#9	2000ppm	RESTEK	569730
Custom SVO Mix	2000ppm	SPEX	SVO-TANJ-16-5
Bisphenol A	1000ppm	SPEX	S-509-MC
8270 List 1/ Std#11	2000ppm	RESTEK	569732
8270 Surrogate Standard	5000 ppm	RESTEK	567685
Aromatic Amines Custom Mix	2000 ppm	Supelco	21467482

7.2.1.1. **Spiking Standard:** For use in spiking aqueous samples including TCLP/SPLP leach being prepared for BNA analysis by SW846 Method 8270. Prepare the second source spiking solution for MS/MSD/Blank Spike (LCS) as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	20 ml (methanol)	50/100/200 ppm
8270 List 1/ Std#10	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/ Std#9	2000ppm	10 ml (methanol)	100 ppm
Custom SVO Mix-SPEX	2000ppm	10 ml (methanol)	100 ppm
Bisphenol A-SPEX	1000ppm	10ml (methanol)	50ppm

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
8270 List 1/ Std#11	2000ppm	20ul (methanol) 100ul(methanol)	Used neat for LVI Used neat for non-LVI
Aromatic Amines Custom Mix**	2000ppm	10 ml (methylene chloride)	100 ppm

** *As needed based on clients requirement.*

Note: Neat spiking/surrogate standards are stored per vendor requirements (either room temp or 4 deg C, as appropriate). All prepared standard solutions are refrigerated at 4 deg C.

7.2.2 **8270 Surrogate Standard Spiking Solution**: For use in spiking all blanks, samples and associated QC prior to extraction. Prepare as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 1000 ml methanol	Final Concentration
8270 Surrogate Standard	5000 ppm	20 ml	100 ppm

7.2.3 Internal standard is prepared added by the analytical department. For details see analytical SOP No.. ED-MSS-009 (SW846 8270E).

7.2.4 The preparation of all standards must be documented in Eurofins LIMS (TALS) or a standard preparation logbook. Information such as standard supplier, lot number, original concentration, and a description of how standard was prepared are required along with a laboratory lot number, analyst's initials, date prepared and verification signature. Standards must be made every 6 months or sooner if signs of degradation appear.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

8.1 All samples must be stored at 4ºC (± 2ºC) upon receipt.

8.2 Sample Extract Storage. Samples extracts must be protected from light and refrigerated at 4ºC (± 2ºC) from time of extraction until analysis.

8.3 Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water or Leachate	250ml Amber (RVE)/ Amber glass, 1L	250ml-RVE/ 1000 ml	Cool 4 ± 2ºC	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270C

9.0 Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples. Refer to Eurofins Edison analytical SOP No. ED-MSS-009 (SW846 8270E) for details on analysis and evaluation of these QC elements:

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.
[3] Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)
[4] Statistical control limits are updated annually and are updated into lab reporting software.

9.1.1. **Method blanks** are extracted with every sample batch on each day that samples are extracted.

9.1.2. **Matrix Spike (MS)/Matrix Spike Duplicate (MSD):** A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. (Note: an LCS/LCSD may be substituted for the MS/MSD if insufficient client environmental sample volume is available).

9.1.3. **Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD):** A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples.

9.1.3.1 A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client

sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.2).

10.0 Procedure

NOTE: The sample preparation procedure for aqueous samples (Section 10.1) contains two options: reduced volume extraction (RVE) (250ml) for large volume injection (LVI) analysis and large volume extraction (1000ml),

10.1. Sample Preparation for Aqueous/ Leachates Samples

10.1.1. Rinse the required number of 500-ml separatory funnels (when RVE is required) or 2000-ml separatory funnels (when large volume extraction is required) and 250-ml Erlenmeyer flasks twice with a 1:1 mixture of Methylene Chloride:Acetone and once with Methylene Chloride.

10.1.2. Place a small amount of glass wool into a 100-mm funnel and fill with pre-baked sodium sulfate crystals. Rinse three (3) times with Methylene Chloride. Also rinse the outside of the funnel stem three (3) times with Methylene Chloride (since the stem is likely to come into contact with the extract). Allow time for all of the rinsate to drain out of the funnel into a waste container.

10.1.3. Record the lab sample numbers on the separatory funnels with red wax pencil.

10.1.4. Make up tags with the following information and place on a 250ml Erlenmeyer flask:

BNAs		**BNs**	**AEs**
Acid Fraction	BN Fraction		
Sample Number	Sample Number	Sample Number	Sample Number
Fraction-Matrix	Fraction-Matrix	Fraction-Matrix	Fraction-Matrix
Date of Extraction	Date of Extraction	Date of Extraction	Date of Extraction

10.1.5. Place the 100ml funnel containing rinsed sodium sulfate crystals onto the flask.

10.1.6. Mark the fluid level on the sample bottles with a black Sharpie. Pour each sample into its corresponding separatory funnel. Fill each sample bottle to the black line with tap water. Pour this into the graduated cylinder used for measuring sample volumes. Note the volume for each sample on the Organic Extraction Data Sheet.

10.1.7. Rinse out a graduated cylinder with lab reagent water two to three times. Using the graduated cylinder obtain 250 ml when RVE is used (or 1000-ml when large volume is required) of lab reagent water from the Millipore filtering apparatus located in the Wet Chemistry laboratory for each of the method blank and the laboratory control sample (LCS) (aka blank spike).

10.1.8. Pour each the reagent water for the method blank and LCS into the corresponding separatory funnels.

10.1.9. Rinse syringes eight (8) to ten (10) times with Methylene Chloride.

10.1.10. If you are performing BNAs add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.11. If you are performing *BN only* extraction, add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.20. If you are performing *AE only* extraction add 0.2 ml when RVE is used (or 1.0 ml when large volume is required) of Surrogate Standard Spiking Solution (see Section 7.2.2) to each sample/QC sample and proceed to Section 10.1.11.

10.1.10.1. If extracting QC samples (MS, MSD, LCS or LCSD), add 0.2 ml when RVE is used (or 1.0 ml when large volume is required of the Spiking (see Section 7.2.1.1) to the appropriate separatory funnel. Note: When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Do not touch the tip of the syringe to the liquid or the side of the separatory funnel.

10.1.11. Add concentrated sulfuric acid to each sample to adjust the pH to <2. (Usually you only need to add 1ml using small disposable Pasteur pipette). Note: pH adjustments must be documented in the extraction log.

10.1.12. Shake each sample for a short time and check pH using pH paper. The pH must be 2 or less. If the pH has not been lowered sufficiently, add more acid.

10.1.13. Add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required) to each sample bottle.

10.1.14. Swirl the bottle and add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required) to its corresponding separatory funnel.

10.1.15. After making sure the funnels are properly secured, start the rotators. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.1.16. Stop the rotation and allow the sample to settle.

10.1.17. Drain the bottom (organic) layer from the separatory funnel into the funnel/Erlenmeyer apparatus.

10.1.18. Repeat steps 10.1.13 through 10.1.17 twice, adding the Methylene Chloride directly to separatory, rather than rinsing the sample container as in 10.1.15.

10.1.18.1. If an emulsion forms during extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride on the bottom. A 1 ml disposable pipette should be used to transfer the methylene chloride (bottom) layer from the centrifuge tube to the appropriate Erlenmeyer flask. With this method, care must be taken not to transfer the water (top) layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.1.19. If you are preparing the sample for acid extractables analysis only, you are finished and you can now discard the remaining liquid in each separatory funnel and proceed to Section 10.1.27. If you are required to extract base/neutrals, proceed with Section 10.1.20.

10.1.20. Adjust the pH of the sample to >11 by adding 10N sodium hydroxide (NaOH) to the sample in each separatory funnel. NOTE: pH adjustment must be documented in the extraction logbook.

10.1.21. Shake each separatory funnel for a short time and check pH. It should be basic, >pH 11. If the pH is not as high as it should be, add more 10N sodium hydroxide (NaOH).

10.1.22. Add 15 ml of Methylene Chloride when RVE is used (or 60 ml when large volume is required)

10.1.23. After making sure the funnels are properly secured, start the rotators. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.1.24. Stop the rotation and allow the sample to settle.

10.1.25. Drain the bottom (organic) layer from the separatory funnel into the funnel/Erlenmeyer apparatus.

10.1.26. Repeat steps 10.1.22 through 10.1.25 twice.

10.1.26.1. If an emulsion forms during extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride on the bottom. A 1 ml disposable pipette should be used to transfer the methylene chloride (bottom) layer from the centrifuge tube to the appropriate Erlenmeyer flask. With this method, care must be taken not to transfer the water (top) layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.1.27. Pour the entire methylene chloride extract from the Erlenmeyer flask into a KD concentration tube apparatus (pre-rinsed three times with acetone). Rinse the Erlenmeyer flask from which the sample came twice with methylene chloride and pour both rinsates into the KD apparatus.

10.1.28. Attach Snyder column (pre-rinsed three times with acetone) to the top of the KD apparatus.

10.1.29. For RVE extractions blow the extract directly down to a final volume of 2-ml on the N-Evap. Remove the Snyder column from the top of the KD flask. Remove the blue taper clamp from the ground glass joint and dry the exterior with a Kimwipe. Transfer the concentrator tube with the 5ml extract to the N-Evap and “blow down” the extract until the volume is 2.0 ml.

10.1.30. For large volume (1000 ml) extractions concentrate the extract to approximately 5-ml in Steam Bath Remove the Snyder column from the top of the KD flask. Remove the blue taper clamp from the ground glass joint and dry the exterior with a Kimwipe. Transfer the concentrator tube with the 5ml extract to the N-Evap and “blow down” the extract until the volume is 1.0 ml. Bring the volume of each extract up to 2ml with methylene chloride

10.1.31. Split each extract into two (2) -1ml aliquots and transfer each aliquot to a separate 2ml amber screw cap vial with Teflon liner. Label one vial as ‘SIM” and the second vial as ‘Total’. Transfer custody of the vials to the Semivolatile GC/MS laboratory for analysis (see Eurofins Edison analytical SOP ED-MSS-009 (SW846 8270E).

10.2. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.2.1. The Standards Prep Logbook or TALS Reagent Database must be completed in full with the required information whenever standards are logged and/or prepared.

10.2.2. Each time an extraction is performed, the applicable TALS data record must be completed and reviewed the Organic Prep Supervisor or designee. Lot numbers of all reagents and solvents used or added to samples during preparation must be documented in the database.

10.2.3. Each sample extracted must be included in a batch and be recorded in the TALS database.

10.2.4. Following the extraction procedure, the technician must complete all TALS data fields pertaining to the samples extracted.

11.0. Calculations/Data Reduction

n/a

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For demonstration of capability procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1 It is Eurofins's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½ inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for

neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1 United States Environmental Protection Agency, "Method SW3510C, Separatory Funnel Extraction", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2 Eurofins Edison SOP No. ED-MSS-009, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS) SW846 Method 8270E*, current revision

15.3 Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.4 Eurofins Environmental Health and Safety Manual, CW-E-M-001.

15.5 Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*), current revision.

15.6 Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), current revision.

15.7 Eurofins Edison SOP No. ED-GEN-022, *Training,* current revision

15.8 Eurofins Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)*

15.9 Eurofins Edison SOP No. ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).*

16.0. Method Modifications:

N/A

17.0. Attachments

N/A

18.0. Revision History

Revision 12, effective 07/02/2024
- Updated throughout Eurofins branding.
- Updated references to remove obsolete methods 8270C and 8270D.

Revision 11, effective 03/26/2018
- Updated throughout to clarify that the lab's standard procdure is to use Reduced Volume Extraction (~250 ml initial volume) with an option to use large volume extraction (~1000 ml initial volume).

Revision 10, effective 11/29/2016:
- Sections 7.2.1 and 7.2.1.1 : updated current sources of standards.
- Section 7.2.1.1: added standards storage information as note at end of section.
- Section 8.0: added option for 250 ml amber sample containers (LVI option)

Revision 9, effective 11/21/2014:
- Section 7.2: updated current sources of all standards.
- Throughout document as required: added option for preparation of leachates by LVI.
- Section 10.1.29 through 10.1.31: clarified the concentration techniques for both full volume and LVI extracts.

Revision 8, effective 11/28/2012
- Throughout document: updated references to Lab Quality Manual section numbers.
- Added references as necessary throughout to Eurofins Edison SOP No. ED-MSS-009 (*Semivolatile Organic Compounds by GC/MS, SW846 Method 8270C*D, current revision.
- Section 2.2 added describing option for analysis of lower initial volume for subsequent analysis using large volume injection (LVI) technique.
- Section 6.2: added 500 ml separatory funnel.
- Sections 7.2.1 and 7.2.1.1: added '5 Compound BNA Custom Mix' and 'Aromatic Amine Custom Mix' to list of standards and prep instructions table.
- Section 10.1 (Sample Prep for Aqueous Samples): revised throughout to include option for extraction of reduced aqueous volume (250ml) for subsequent analysis by LVI technique. Added note as preface to Section 10.1 alerting analyst to the two available extraction volume options (1000ml and 250ml).

Revision 7, effective 12/6/10
- Section 3: revised to reference new location for definitions
- Sections 7.2.1 and 7.2.1.1: Added 4 compounds and additional details to the description of standard preparation of the Spiking Standard.
- Section 7.2.4: added option to document standards preparation within TALS rather than a laboratory notebook.
- Section 10.3: revised to include TALS as the main repository for raw data associated with sample prep.

Revision 6, effective November, 2008

- Revised SOP format in accordance with Eurofins Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
- Revised title to include 'Leachates'.
- Section 1.3: Added reference to Quality Assurance Manual for method modifications.
- Section 3: revised to reference new location for definitions.
- Section 5: Revised to include most up to date corporate health and safety references and information.
- Section 7: added details of the solvent testing and approval program.
- Section 7.2.1: Added additional details to the description of standards and the preparation of the Spiking Standards and Surrogate Standards. Removed references to the Internal Standard which is now added by the analytical group and is discussed in Eurofins Edison SOP No. ED-MSS-002, *Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), SW846 Method 8270C*, current revision.
- Section 9: Quality Control: added additional details to the discussion of the various QC sample types
- Section 10: Revised and clarified to reflect current procedures. Removed reference to internal standard addition (now completed by analytical group).
- Section 11: Removed reference to Organic Calculation SOP.
- Section 12: updated and revised the MDL requirements to reflect text in the current revision of the Eurofins Edison Laboratory Quality Manual (LQM).
- Section 15: References: Expanded to include more specific SOP references
- Section 16: Added Section 16 (Method Modifications).
- Section 18: Added this Revision History section

Title: Extraction of Pesticides and PCBs in Water by Separatory Funnel using SW846 Method 3510C

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Approvals (Signature/Date):

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1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits:** SW846 Method 3510C describes a procedure for isolating organic compounds from aqueous samples, including concentration techniques suitable for preparing the extract for GC and/or GC/MS analysis. This SOP is specifically applicable to the isolation and concentration of water-soluble and slightly water-soluble Pesticides and PCBs using Method 3510C in preparation for analysis GC methods SW846 8081B, 8082A and GC/MS method EPA 680.

1.2. For a complete discussion of analytes and reporting limits (RLs) refer to the SOP for the applicable analytical method:.

- TestAmerica Edison SOP No. ED-GCS-016, *Analysis of Organochlorine Pesticides by SW846 Method 8081B,* current revision.
- TestAmerica Edison SOP No. ED-GCS-017, *Analysis of Polychlorinated Biphenyls by SW846 Method 8082A,* current revision.
- TestAmerica Edison SOP No. ED-MSS-010, *Determination of PCBs in Water and Soil/Sediment by GC/MS, EPA Method 680*, current revision.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1 A measured volume of aqueous sample is spiked with surrogates and serially extracted with methylene chloride using a separatory funnel. The extract is dried by passing it through activated sodium sulfate, concentrated using a nitrogen blowdown technique and exchanged into hexane prior to analysis by Methods 8081B, 8082A or 680. Cleanup of extracts may be required prior to analysis.

2.2 The preferred option for preparing aqueous samples for analysis by SW8081B/8082A includes use of a reduced initial volume (~250ml) for analysis by a large volume injection (LVI). A technique for extraction of a larger volume (1000 ml) is also described.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Phthalate esters contaminate many products commonly found in the laboratory. Plastics, in particular, must be avoided, because phthalates are often used as

plasticizers and are easily extracted from plastic material. Phthalate contamination may result at any time if consistent quality control is not practiced.

Method interference 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 interference under the conditions of the analysis, by analyzing method and reagent blanks.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used.

The use of separatory funnels to extract aqueous samples with methylene chloride creates excessive pressure very rapidly. Initial venting should be done immediately after the sample container has been sealed and inverted. Vent the funnel into the hood away from people and other samples. This is considered a high-risk activity, and a face shield must be worn over safety glasses or goggle when it is performed.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions (if applicable).			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Equipment

- Steam Bath - Fisher Scientific 66738 or equivalent.
- Muffle Furnace - Thermolyne Type 6000 or equivalent.
- N-Evap - Meyer Analytical Evaporator Model No. 112 or equivalent
- Separatory Funnel Rotator, APR Machine or equivalent
- Centrifuge, Varifuge F; Hereaus Sepatech

6.2. Supplies

- 400 ml Clear Glass Jar
- 500 ml Separatory Funnel, AMK Glass SFC-0095 or equivalent
- 100 mm o. d. glass funnels, Fisher or equivalent
- 10 ml jacketed, graduated Concentrator Tubes, AMK Glass KD-0018 or equivalent
- 19/22 Ground Glass Stoppers

- 50 ul Gastight Syringe, Hamilton 80900 or equivalent
- 100 ul Gastight Syringe, Hamilton 81000 or equivalent
- 150 ml Centrifuge Tube
- 100 ml Graduated Cylinder
- Pasteur 5¾" Disposable Pipettes, Fisher 13-678-20B or equivalent
- Glass Wool
- Desiccators
- Standard Taper Clamps (Size 19, blue)
- Wax Pencil
- 1 Liter Graduated Cylinder
- Marking Tags
- pH paper

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride and Acetone, is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

- Methylene Chloride - JT Baker Ultra-Resi 9254-03 or equivalent
- Sodium Sulfate Crystals – Mallinckrodt MA8024-06 or equivalent
- Acetone, J. T. Baker Ultra-Resi 9264-03 or equivalent
- Hexane, Pesticide Grade, Baxter 217-4or equivalent
- Organic free reagent water - Reagent water is defined as a water in which an interference is not observed at the MDL of each parameter of interest (ASTM Specification D1193, Type ii). At TestAmerica Edison this water is generated by the Barnstead/Thermolyne Water System (Model # D11991 Serial # 1191020210415).

7.1.1 Reagent preparation

7.1.1.1 Anhydrous sodium sulfate crystals (Mallinckrodt MA8024-06 or equivalent) must be baked in the muffle furnace for four hours at 400^{o}C and serially rinsed with methylene chloride prior to use.

7.2 Standards

7.2.1 Standards are purchased as concentrated solutions (see Section 7.2.2). Most stock solutions are diluted (in volumetric glassware) to working concentration using acetone as the diluent as described in Section 7.3.

7.2.2 **Standard mixes and sources:** Table 1 lists the standard concentrate mix sources. Table 2 details the individual components of these mixes.

Table 1a:
Pesticide/PCB Standard Mixes and Sources (8081/8082)*

Standard Name ("Lab Name")	Concentration in ug/ml (each component)	Source - Catalog #
Pesticide Surrogate Spike Mix	200	RESTEK-32000
Aroclor Spike Mix (1660 Aroclor)	1000	RESTEK-32039
Organochlorine Pesticides Mix ("Pest Spike")	2000	RESTEK-32415
Chlordane (technical) ("Technical Chlordane Spike")	5000	RESTEK-32072
Toxaphene	5000	RESTEK-32071

*May be substituted with equivalent standards from alternate sources.

Table 1b:
PCB Homologue Standard Mixes and Sources (EPA 680)*

Standard Name ("Lab Name")	Concentration in ug/ml (each component)	Source - Catalog #
Retention Time Calibration Standard Mixture	Varies	Ultra-CB682-MN
Concentration Calibration Standard Mix	Varies	Ultra-CB681-MN
$^{13}C_{12}$-Decachlorobiphenyl Surrogate	40	Cambridge-EC1410-3
Lindane13C6	100	Cambridge-CL1282-S

Table 2a:
Components of Pesticide/PCB Standard Mixes (8081/8082)

Parameter	Catalog Nos.	Lab Standard Name	Conc (ug/ml)
Decachlorobiphenyl (DCB)	Pesticide Surrogate Spike Mix	RESTEK-32000	200
2,4,5,6-Tetrachloro-m-xylene (TCMX)	Pesticide Surrogate Spike Mix	RESTEK-32000	200
Aroclor 1016	Aroclor Spike Mix	RESTEK-32039	1000
Aroclor 1260	Aroclor Spike Mix	RESTEK-32039	1000
Aldrin	Organochlorine Pesticides Mix	RESTEK-32415	2000
alpha-BHC	Organochlorine Pesticides Mix	RESTEK-32415	2000
beta-BHC	Organochlorine Pesticides Mix	RESTEK-32415	2000
Lindane	Organochlorine Pesticides Mix	RESTEK-32415	2000
g-BHC	Organochlorine Pesticides Mix	RESTEK-32415	2000
DDD	Organochlorine Pesticides Mix	RESTEK-32415	2000
4,4'-DDE	Organochlorine Pesticides Mix	RESTEK-32415	2000

Table 2a: Components of Pesticide/PCB Standard Mixes (8081/8082)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Conc (ug/ml)**
4,4'-DDT	Organochlorine Pesticides Mix	RESTEK-32415	2000
Dieldrin	Organochlorine Pesticides Mix	RESTEK-32415	2000
alpha-Endosulfan	Organochlorine Pesticides Mix	RESTEK-32415	2000
beta-Endosulfan	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endosulfan Sulfate	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endrin	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endrin aldehylde	Organochlorine Pesticides Mix	RESTEK-32415	2000
Endrin ketone	Organochlorine Pesticides Mix	RESTEK-32415	2000
Heptachlor	Organochlorine Pesticides Mix	RESTEK-32415	2000
Hetptachlor epoxide	Organochlorine Pesticides Mix	RESTEK-32415	2000
Methoxychlor	Organochlorine Pesticides Mix	RESTEK-32415	2000
Chlordane (technical)	Technical Chlordane Spike	RESTEK-32072	5000
Toxaphene	Toxaphene Spike	RESTEK-32071	5000

Table 2b: Components of PCB Homologue Standard Mixes (680)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Conc (ug/ml)**
PCB-104	Ultra-CB682-MN	Retention Time Calibration Standard Mix	100
PCB-208	Ultra-CB682-MN	Retention Time Calibration Standard Mix	200
PCB-77	Ultra-CB682-MN	Retention Time Calibration Standard Mix	100
Total Nonachlorobiphenyls	Ultra-CB682-MN	Retention Time Calibration Standard Mix	200
DCB Decachlorobiphenyl	Ultra-CB681-MN	Concentration Calibration Standard Mix	250
Total Dichlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	50
Total Heptachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	150
Total Hexachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	100
Total Monochlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	50
Total Octachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	150
Total Pentachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	100
Total Tetrachlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	100
Total Trichlorobiphenyls	Ultra-CB681-MN	Concentration Calibration Standard Mix	50
Decachlorobiphenyl-13C12	Cambridge-EC1410-3	$^{13}C_{12}$-Decachlorobiphenyl Surrogate	40
Lindane13C6	Cambridge-CL1282-S	Lindane13C6	100

7. 3. Standards Preparation

7.3.1. All standard stock solutions are diluted to the working concentrations with hexane or acetone (as indicated) using Class A volumetric glassware. **Note:** septa on all surrogate and spike vials are to be replaced immediately after use. Additionally, all surrogate and spike vials are to be returned to the standards refrigerator immediately after use.

7.3.2. **Pesticide Spiking Standard (LCS/MS/MSD): For the reduced volume extraction option:** prepare a 4 ug/ml spiking solution by diluting 10 ml of the 20ug/ml standard prepared above to a final volume of 50ml in acetone. For spiking instructions refer to Section 10.

7.3.3. **8082 PCB Spiking Standard (LCS/MS/MSD):**. **For the reduced volume extraction option:** prepare a 20 ug/ml spiking solution by diluting 10 ml of the 100 ug/ml stock standard prepared above to a final volume of 50ml in acetone. For spiking instructions refer to Section 10.

7.3.4. **680 PCB Homologue Spiking Standard (LCS/MS/MSD):** The EPA Method 680 PCB homologue spiking standard is prepared by diluting 1.0 ml of the Retention Time Calibration Standard Mix (Ultra-CB682-MN) and 1.0 ml of the Concentration Calibration Standard Mix (Ultra-CB681-MN) to 100mL of hexane using volumetric glassware. For spiking instructions refer to Section 10.

7.3.5. **Technical Chlordane Spiking Standard (LCS/MS/MSD):** The technical Chlordane spiking solution is prepared at a final concentration of 100 ug/ml by diluting 0.2 ml of the 5000 ug/ml Technical Chlordane Spike solution (RESTEK-32072, see Tables 1 and 2) to a 10 ml final volume using acetone. For spiking instructions refer to Section 10.

7.3.6. **Toxaphene Spiking Standard (LCS/MS/MSD):** A Toxaphene spiking solution is prepared at a final concentration of 100 ug/ml by diluting 0.2 ml of the 5000 ug/ml Toxaphene Spiking Standard (RESTEK-32071, see Tables 1 and 2) to a 10 ml final volume with acetone. For spiking instructions refer to Section 10.

7.3.7. **Pesticide Surrogate Spiking Standard: For the reduced volume extraction option:** prepare a 2 ug/ml spiking solution by diluting 10 ml of the 10 ug/ml stock standard described above to a final volume of 50ml in acetone. For spiking instructions refer to Section 10.

7.3.8. **680 PCB Homologue Surrogate Spiking Standard:** The EPA Method 680 surrogate spiking standard is prepared by diluting 3.125 ml of Decachlorobiphenyl-13C12 Surrogate (Cambridge-EC1410-3) and 1.0 ml of Lindane13C6 (Cambridge-CL1282-S) to 50.0 ml hexane using volumetric glassware. For spiking instructions refer to Section 10.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Water	Amber Glass	250 ml (8 oz)	Cool 4 ± 2°C	7 days to extraction; 40 days to analysis	USEPA SW846
Water	Amber glass	1000 ml	Cool 4 ± 2°C	7 days to extraction; 40 days to analysis	USEPA Method 680

8.1. Extracts must be stored under refrigeration (Cool 4 ± 2°C) in the dark and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample used for MS/MSD is randomly selected by the organic prep lab, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into TALS (LIMS).

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to ensure that the analytical system is in control. It is also used to assess performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated acceptance criteria. If the spiked sample recovery results fall outside the laboratory generated limits (refer to the current active TALS

method limit group database), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QC batch must be re-extracted and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to refer to the current active TALS method limit group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in the applicable analytical SOP (see Section 15.0, References).

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with surrogate standard solution containing DCB and TCMS (see Section 7.3.6). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to refer to the current active TALS method limit group database). If the surrogate recovery limits are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

10.0. Procedure

10.1. Rinse a 500 ml separatory funnels twice each with a 1:1 methylene chloride/acetone mix and once with methylene chloride. Drain the solvents before adding sample to the funnels.

10.2. Place a small amount of glass wool into a 100-mm funnel and fill with pre-baked sodium sulfate crystals. Rinse three times with methylene chloride. Also rinse the outside of the funnel stem three times with methylene chloride, as it is likely to come into contact with the extract. Allow time for all of the rinsate to drain out of the funnel into a waste container.

10.3. Write the lab sample id number for each sample on the separatory funnels using a red wax pencil.

10.4. Make up hangtags for each sample as follows and hang on the corresponding

PCBs	PST
Sample Number: xxx	Sample Number: xxx
Fraction-Matrix:xxx	Fraction-Matrix:xxx
Date of Extraction:xx/xx/xx	Date of Extraction:xx/xx/xx

10.5. Place a 100 ml funnel containing rinsed sodium sulfate crystals onto each flasks

10.6. Mark the fluid level on each sample bottle with a black magic marker.

10.7. Rinse out a 1000ml graduated cylinder two to three times with organic free reagent water (for TCLP/SPLP leachates or reduced volume extraction use a 250 ml graduated cylinder). Obtain 1000ml of organic free reagent water (100 ml for TCLP/SPLP leachates or 250ml for reduced volume extraction) from the Millipore filtering apparatus for each of the method blank and the LCS.

10.8. Measure the initial pH of each sample with wide range pH paper and record the observed value in TALS prep batch as 'Received/initial pH'. If the initial pH of the sample is not between 5-9 adjust the pH to within 5-9 using 1:1(v/v) sulfuric acid or 10N Sodium hydroxide, as appropriate. Record the final adjusted pH value in the TALS batch comment section. Lesser strengths of acid or base solution may be employed, provided that they do not results in a significant change (<1%) in the volume of sample.

10.9. Pour each sample (including the method blank and LCS water) into its corresponding separatory funnel.

10.10. Rinse the spiking syringes 8 to 10 times with Acetone.

10.11. **For methods 8081/8081**: Add 50 ul of Pesticide Surrogate Spike Mix (see Section 7.2) to each sample and QC sample. When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Don't touch the tip of the syringe to the liquid or the side of the separatory funnel

10.12. **For method 680**: add 1.0 ml of the PCB Homologue Surrogate Spiking Standard (see Section 7.3.8) to each sample and QC sample. When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Don't touch the tip of the syringe to the liquid or the side of the separatory funnel

10.13. Depending upon the fraction and requested target analytes (Refer to Section 7.3 for information of spiking solutions).

- **PCBs by 8082**: add 50 ul of the 20ug/ml PCB Spike solution (see Section 7.3.3) to each LCS and designated MS/MSD
- **Pesticides (total) and TCLP/SPLP Pesticides by 8081**: 50 ul of the 4 ug/ml Pest Spike solution (see Section 7.3.2) to each LCS and designated MS/MSD
- **Technical Chlordane by 8081:** 20 ul of the 100 ug/ml Technical Chlordane Spike mix (see Section 7.3.5) to each LCS and designated MS/MSD.
- **Toxaphene by 8081:** 20 ul of the 100 ug/ml Toxaphene Spike mix (see Section 7.3.6) to each LCS and designated MS/MSD.

- **PCB Homologues by 680:** 1ml of the 680 PCB Homologue Spiking Standard (see Section 7.3.4) to each LSC and designated MS/MSD).

When spiking the samples, make sure to get all bubbles out of the syringe. In addition, hold the syringe just above the level of the liquid when adding the spike. Don't touch the tip of the syringe to the liquid or the side of the separatory funnel. Add 15 ml of methylene chloride for the reduced volume extraction option.

10.14. Add 15 ml of methylene chloride to each sample bottle. Swirl the solvent in the bottle and add the 15 ml for of methylene chloride to its corresponding separatory funnel.

10.15. Secure the separatory funnels to the rotators. Start the rotators.

10.16. Stop the rotator and vent the funnels after about 10 seconds. Resume rotating for 2 minutes.

10.17. After the rotator stops let the sample settle.

10.18. Drain bottom layer (organic layer) of the sample into the funnel/Concentrator apparatus.

10.19. Repeat steps 10.12 through 10.16 twice, now adding the methylene chloride directly to separatory funnel, rather than rinsing the sample container as in 10.12.

10.20. If an emulsion forms during the extraction, rinse a centrifuge tube well with methylene chloride and then drain the lower layer from the large separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the methylene chloride with the desired sample extract on the bottom. A 1-ml disposable pipette should be used to transfer the bottom layer from the centrifuge tube to the appropriate concentration tube. With this method, care must be taken not to transfer any of the top layer. The top layer that remains is poured back into the separatory funnel with the rest of the original sample.

10.21. Find the empty sample bottles previously marked with a black marker with tap water up to the black line. Pour this into the graduated cylinder used for measuring sample volumes. Record each sample volume into the TALs extraction batch database.

10.22. Rinse funnel/sodium sulfate attached to concentration tube, twice with about 20ml of methylene chloride.

10.23. Sample can be concentrated between 30^{o}C-40^{o}C in the N-Vap to 2 ml final volume in the methylene chloride

10.24. Add about 10ml of Hexane to extract to exchange

10.25. Concentrate the hexane extract to 1 ml on the steam bath (temperature set between 30°C-40°C) using the N-EVAP apparatus.

10.26. The extract is now ready for the appropriate clean up.

10.27. If an emulsion forms during extraction, rinse a centrifuge tube well with Methylene Chloride and then drain the lower layer from the large separatory funnel into the tube. Centrifuge for 3 to 5 minutes at 2000 rpm. Make sure the levels (and weights) of the samples directly opposite one another in the centrifuge are approximately equal. After the centrifuging process is finished, there will be two layers: water on top and the Methylene Chloride with the desired sample extract on the bottom. A 1-ml disposable pipette should be used to transfer the bottom layer from the centrifuge tube to the appropriate concentration tube. With this method, care must be taken not to transfer any of the top layer. The top layer that remains is poured back into the 2000-ml separatory funnel with the rest of the original sample.

10.28. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.28.1. The TALS reagent database must be completed in full with all required information whenever standards are logged and/or prepared.

10.28.2. All required data types (sample volumes, reagent and standard volumes, reagent and standard lots, spike witness, etc…) must be entered into the TALS batch database each time an extraction is performed. The department manager is responsible for ensuring that the data is reviewed for completeness and accuracy.

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11.0. Calculations / Data Reduction

None

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a

calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to the current revision of TestAmerica Edison SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½

inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method 3510C: Separatory Funnel Liquid-Liquid Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2. *EPA Method 680: Determination of Pesticides and PCBs in Water and Soils/Sediment by Gas Chromatography/Mass Spectrometry*. Physical and Chemical Methods Branch, Environmental Monitoring and Support Laboratory, Office of Research and Development, USEPA, Cincinnati, OH, November 1985.

15.3. TestAmerica Edison SOP No. ED-GCS-016, *Analysis of Organochlorine Pesticides by SW846 Method 8081B,* current revision.

15.4. TestAmerica Edison SOP No. ED-GCS-017, *Analysis of Polychlorinated Biphenyls by SW846 Method 8082A,* current revision.

15.5. TestAmerica Edison SOP No. ED-MSS-010, *Determination of PCBs in Water and Soil/Sediment by GC/MS ,EPA Method 680*, current revision.

15.6. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.7. TestAmerica Corporate Document No. CW-E-M-001, *Environmental Health and Safety Manual,* current revision.

15.8. TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.9. TestAmerica Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.10. TestAmerica Edison SOP No. ED-GEN-013, *Glassware Cleaning*, most current revision

15.11. TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision.

16.0. Method Modifications:

NONE

17.0. Attachments

NONE

18.0. Revision History

- Revison 13, dated Jun 27, 2019:
    - Sections 1 and 15 (and throughout document as applicable): Deleted references to obsolete methods SW8082 and SW8081A as well as references to associated SOPs.
    - Section 2.2: revised wording to clarify that the RVE/LVI technique is the standard procedure.
    - Throughout document: removed references to equipment no longer used in the procedure: Synder columns, Kuderna-Danish concentrators, Erlenmyer flasks.

- Revision 12, dated May 16, 2017:
    - Updated throughout to include aqueous preps for EPA method 680.
- Revision 11, dated Dec 13, 2016:
    - Section 6.2: added pH paper to list of supplies.
    - Section 10.8: added instruction for measuring, adjusting and documenting sample pH values.
- Revison 10, dated Jun 15, 2015:
    - Section 2.1 and throughout document: Revised standard initial volume for Reduced Volume Extraction (RVE) to 250 ml.
    - Section 7.2: Tables 1 and 2 updated with new Restek standards.
    - Sections 7.3: updated spiking standards preparation instructions as necessitated by switch to Restek standards.
    - Section 8.0: updated minimum volume to 250ml.
- Revison 9, dated May 3, 2013:
    - Section 7.2.2, Table 1: corrected concentration of the Chlordane (technical) standard solution to 1000 ug/ml. Deleted 'TCLP Pest Mix 1' and 'TCLP Pest Mix 2'. Added Toxaphene (Supleco 48103-U).
    - Section 7.2.2, Table 2: corrected concentration of the Chlordane (technical) standard solution to 1000 ug/ml. Deleted all TCLP Pest Spike standards. Added Toxaphene (Supleco 48103-U).
    - Deleted Section 7.3.4, TCLP Pesticide Spiking Standard (LSC/MS/MSD). Remaining sections renumbered accordingly.
    - Section 10.11. Deleted last bullet (TCLP Pesticides). Added '…and TCLP/SPLP Pesticides' to the second bullet (Pesticides). Added '(or 20 ul of the 100ug/ml mix for reduced volume extraction)' to the Toxaphene bullet.
- Revision 8, dated February 26, 2013:
    - Throughout document: updated Lab Quality Manual section number references as required.
    - Section 1.2: added references to all applicable analytical SOPs.
    - Section 2.0: added Section 2.1 which describes option for Reduced Volume Extraction.
    - Section 6.2: added a 500 ml separatory funnel.
    - Section 7.0: updated throughout to include additional standards preps for reduced volume extraction.
    - Section 8.0: updated to include smaller sample container for the reduced volume extraction option.
    - Section 10.0: updated to include spiking and concentration instructions for the reduced volume extraction option.

- Revision 7, dated October 31, 2011

    - Section 7.0: added 'Reagent Water' and details concerning it's preparation to the list of reagents.

- Revision 6, dated October 30, 2009

    - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
    - Section 1.1 Added reference to Quality Assurance Manual for method modifications.
    - Section 1.1: Expanded to include references to applicable analytical SOPs.
    - Section 3: revised to reference new location for definitions.
    - Section 5: Revised to include most up to date corporate health and safety references and information.
    - Section 7.2.2: Added tables detailing components found in the various spiking standards mixes.
    - Section 7.3: Updated the instructions for preparation of spiking standards.
    - Section 7.3: Added tables with spiking standards prep details.
    - Section 8: Updated with additional details including a table outlining containers, preservation and holding times.
    - Section 9.1: Expanded QC sample preparation details.
    - References: Expanded to include more specific SOP references
    - Section 18: Added this Revision History section
    - Throughout document: added references to TestAmerica LIMS (TALS).
    - Throughout document: deleted references to organophosphorus pesticides as the Edison lab no longer analyzes for these.

Title: Extraction of Organochlorine Herbicides in Water by SW846 Method 8151A

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):

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1.0 Scope and Application

1.1. **Analytes, Matrix(s), and Reporting Limits:** SW846 Method 8151A provides extraction and derivatization procedures for the gas chromatographic analysis of chlorinated acid herbicides in water, soil, and waste samples. An option for the hydrolysis of esters is also described. This SOP details the extraction of water samples with diethyl ether followed by esterification with trimethylsilyl diazomethane (an option for the use of diazald is also included). The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

1.2. For a complete discussion of analytes and reporting limits (RLs) please refer to Eurofins Edison SOP No. ED-GCS-005, *Analysis of Organochlorine Herbicides by SW846 Method 8151A,* current revision.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work*) and 19 (*Test Methods and Method Validation*) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

This SOP provides extraction and derivatization procedures for the determination of Chlorinated acid herbicides and their esters in water and aqueous waste samples.

Water samples are hydrolyzed, cleaned with methylene chloride, acidified, extracted with diethyl ether and then esterified with TMSD (trimethylsilyl diazomethane). And optional esterification using diazald is also described. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD).

The sensitivity of Method 8151A depends on the level of interferences in addition to Instrumental limitations.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Method interference 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 interference under the conditions of the analysis, by analyzing reagent blanks.

Matrix interference may be caused by contaminants that are co-extracted from the sample. Organic acids, especially chlorinated acids, cause the most direct

interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure.

The acid forms of the analytes are strong organic acids that react readily with alkaline substances and can be lost during sample preparation. Glassware and glass wool must be acid-rinsed with 1 N hydrochloric acid and the sodium sulfate must be acidified with sulfuric acid prior to use to avoid analyte losses due to adsorption.

Sample extracts must be dry prior to methylation or else poor recoveries will result.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Ethyl Ether is an extremely flammable solvent. If a mechanical device is used for sample extraction, the device should be equipped with an explosion proof motor and placed in a hood.

Ethyl Ether must be free of peroxides as determined by use of EM Quant Peroxide Test Strips as described in Sections 6 and 7 below.

Diazomethane can explode under certain conditions therefore take the following precautions:

- Use a well-ventilated hood.
- Use a safety screen.
- Do not heat above 90°C.
- Avoid grinding surfaces, ground glass joints, sleeve bearings, and glass stoppers.
- Store away from alkali metals.
- Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

Ether creates excessive pressure very rapidly. Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Ethyl Ether	Flammable Irritant Peroxide Former	400 ppm-TWA	General anesthesia by inhalation can occur. Continued exposure may lead to respiratory failure or death. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. May cause irritation, redness and pain to the eyes. Irritating to the skin and mucous membranes by drying effect. Can cause dermatitis on prolonged exposure. May be absorbed through skin. **May form explosive peroxides on long standing or after exposure to air or light. This material must be disposed of within six months.**

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Equipment

- Top loading Balance - Sargent Welch S2642-25 or equivalent
- Automatic separatory funnel rotator
- Furnace - Thermolyne Type 6000 or equivalent.
- Vacuum Pump - General Electric Model No. 0322-V48-G180X or equivalent.
- N-Evap - Meyer Analytical Evaporator Model No. 112 or equivalent

6.2. Supplies

- 400 ml Clear Glass Jar
- 1 L Erlenmeyer Flask
- 500 ml Erlenmeyer Flask
- 2 L Separatory Funnel
- 500 ml Separatory Funnel
- 500 ml Centrifuge Bottle - Pyrex 1260 or equivalent.
- 10 ml graduated Concentrator Tube - Kontes K570050-1025 or equivalent.
- 10 ml glass vials with Teflon lined screw caps.
- 20 ml glass vials with Teflon lined screw caps.
- 5 ml to 1000 ml Class A Volumetric flasks
- Funnel - 75 mm diameter
- 1 L Graduated Cylinder

- 50 ul Gastight Syringe - VWR 60376-241 or equivalent.
- 100 ul Gastight Syringe - Baxter S9662-5 or equivalent.
- 100 ml Graduated Cylinder
- 1 ml Pasteur Disposable Pipet
- 10 ml Disposable Pipette
- Glass stopper - size 24/40
- Teflon stopper - size 19/22
- Filter paper - 15-cm diameter, Whatman No. 1 or equivalent.
- Phosphoric Acid Washed Glass Wool - Supelco 2-0383 or equivalent.
- Desiccator - Baxter D1420-2 or equivalent.
- Septa, 12 mm - Supelco 2-3273 or equivalent.
- Septa, 8 mm - Supelco 2-3272 or equivalent.
- Standard Taper Clamp (size 19, blue) - Wheaton 297749 or equivalent.
- Silicon Carbide Boiling Stones - Thomas 1590-D33 or equivalent.
- pH paper, Fisher 9590
- EM Quant Peroxide Test Strips, EMD No. 10011-1 or equivalent

Note: All glassware must be pre-rinsed with a 25% Sulfuric Acid: reagent water solution.

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride, Acetone, Methanol and Sulfuric Acid is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

- Organic-free reagent water.
- Trimethylsilyldiazomethane (TMSD) , 2.0 molar solution in hexane, Acros 429211000 or equivalent

- Diethyl Ether (Residue Analysis Grade with 2% Ethanol preservative) - Baker 9259-03 or equivalent (tested for peroxide formation upon opening and monthly thereafter by use of EM Quant Peroxide Test Strips)
- Methylene Chloride (High Purity Grade) - Baker 9266 or equivalent.
- Acetone (High Purity Grade) – Baker 9254 or equivalent.
- Sodium Sulfate Anhydrous Crystals (Reagent Grade) – Malinckrodt 8024 or equivalent. (Baked at 400°C for 4 hours)
- Hexane (High Purity Grade) – Baker 9262 or equivalent.
- Carbitol (diethylene glycol monoethyl ether)
- Potassium Hydroxide (Analytical Reagent Grade) – Aldrich 306568 or equivalent
- Sulfuric Acid (Analytical Reagent Grade) - Mallinckrodt 2876 or equivalent.
- Silicic Acid, Aldrich 30, 636-3 or equivalent

7.1.1 Reagent preparation

7.1.1.1 Anhydrous sodium sulfate crystals must be baked in the muffle furnace at 400° C for 4 hours prior to use. After baking, the sodium sulfate must be acidified. Place 500 grams of anhydrous sodium sulfate into a 500 ml wide mouth Erlenmeyer flask. Add enough diethyl ether to cover the sodium sulfate. Add 0.5 ml concentrated sulfuric acid. Shake vigorously for 1 minute. Evaporate the ether by placing the flask on steam bath. Mix 1 g of the resulting solid with 5 ml of organic-free reagent water and measure the pH of the mixture. It must be below a pH of 4. Prepare fresh daily.

7.1.1.2 6N NaOH Solution: Rinse a 1 liter Pyrex flask three times with organic free reagent water. Weigh 240 g NaOH into flask and add 400 ml organic free reagent water. Mix until the NaOH is completely dissolved and the flask has cooled to room temperature. Transfer the NaOH solution to a clean 1 L bottle and fill up to 1 L with DI water.

7.1.1.3 12N H_2SO_4 : Rinse a 1 liter Pyrex flask three times with organic free reagent water. Add 200 ml organic free reagent water to the flask. Slowly, and while mixing, add 100-ml concentrated H_2SO_4 to flask. Continue mixing until flask has cooled to room temperature. Transfer the H_2SO_4 solution to a clean 500-ml amber bottle and label.

7.1.1.4 1:1 H_2SO_4: Rinse a 1 liter Pyrex flask three times with organic free reagent water. Add 100 ml organic free reagent water to the flask. Slowly, and while mixing, add 100-ml concentrated H_2SO_4 to flask. Continue mixing

until flask has cooled to room temperature. Transfer the H2SO4 solution to a clean 500-ml amber bottle and label.

7.1.1.5 25% H2SO4 solution (1:3 H2SO4 solution): 25 ml Acid and 75 ml reagent water. Add 75 ml organic free reagent water to a graduated cylinder. Slowly, and while mixing, add 25-ml concentrated H2SO4 to cylinder. Continue mixing until cylinder has cooled to room temperature. Transfer the H2SO4 solution to a clean amber bottle and label.

7.2 Standards

7.2.1 Standards are purchased as concentrated solutions (see Section 7.2.2). The surrogate stock solution is diluted (in volumetric glassware) to working concentration using acetone as the diluent as described in Section 7.3.

7.2.2 **Standard mixes and sources:** Table 1 lists the standard concentrate mix sources. Attachment 1 details the individual components of these mixes.

Table 1:
Herbicide Standard Mixes and Sources*

Standard Name ("Lab Name")	Concentration (ug/ml)	Source
Acid Herbicide LCS RTS ("Spiking Mix")	varies (in Methanol)	Restek Catalog # 567950
DCAA standard (Surrogate Spiking Solution)	1000 (in methanol)	Restek Catalog #567804

*May be substituted with equivalent standards from alternate sources.

Table 2:
Components of Herbicide Standard Mixes

Parameter	Catalog Nos.	Lab Standard Name	Concentration (ug/ml)
2,4-dichlorophenoxyacetic acid (2,4-D)	Restek Catalog # 567950	Spiking Mix	20
2-(2,4,5-Trichlorophenoxy)propionic acid (2,4,5-TP aka Silvex)	Restek Catalog # 567950	Spiking Mix	5
2,4,5-T	Restek Catalog # 567950	Spiking Mix	5
2,4-DB	Restek Catalog # 567950	Spiking Mix	20
Dalapon	Restek Catalog # 567950	Spiking Mix	20
Dicamba	Restek Catalog # 567950	Spiking Mix	10
Dichloroprop	Restek Catalog # 567950	Spiking Mix	20
Dinoseb	Restek Catalog #	Spiking Mix	20

Table 2: Components of Herbicide Standard Mixes			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
	567950		
MCPA acid	Restek Catalog # 567950	Spiking Mix	2000
MCPP acid	Restek Catalog # 567950	Spiking Mix	2000
Pentachlorophenol	Restek Catalog # 567950	Spiking Mix	5
Picloram (4-amino-3,5,6-trichloropicolinic acid)	Restek Catalog # 567950	Spiking Mix	20
2,4-Dichlorophenylacetic Acid	Restek Catalog # 567804	Surrogate Spiking Solution	1000

7. 3. Standards Preparation

7.3.1. The surrogate stock solutions are diluted to the working concentrations with acetone as indicated in Attachment1 and 2.

7.3.2. Herbicide Spiking Standard (LCS/MS/MSD):

7.3.2.1 Herbicide QC Spiking Standard Mix (12 compound analysis): The 12 compound spiking standard is ready to spike and used when analyzing for the standard list of herbicides The spiking standard mix is a 25 ml solution (Restek Catalog # 567950; see Tables 1 and 2) of the 12 compounds at the specified concentrations as free acids. The concentrate is used without further dilution prior to the spiking of the following quality control sample types: MS, MSD, and LCS. Spiking instructions are found in Section 10.

7.3.3. Herbicide Surrogate Spiking Standard (2, 4-DCAA Acid Spike Mix**):** The herbicide surrogate spiking standard is prepared as detailed in Attachment 1 using volumetric glassware and the concentrated free acid herbicide standards listed in Table 1 above.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific

contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Glass	1L amber/ 8oz amber	Cool 4 ± 2ºC	7 days to extraction; 40 days to analysis	USEPA SW846

8.1. Extracts must be stored under refrigeration (Cool 4 ± 2ºC) in the dark and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD is randomly selected by the organic prep lab, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into TALS (LIMS).

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to ensure that the analytical system is in control. It is also used to assess performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated acceptance criteria. If the spiked sample recovery results fall outside the laboratory generated limits (refer to the current active TALS method limit group database), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are

attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QC batch must be re-extracted and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to refer to the current active TALS method limit group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in Eurofins Edison SOP No. ED-GCS-00, *Analysis of Organochlorine Herbicides by SW846 Method 8151A,* current revision.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with surrogate standard solution containing 2,4-DCAA (see Section 7.3.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to refer to the current active TALS method limit group database). If the surrogate recovery limits are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as "estimated concentration".

10.0. Procedure

10.1. Sample Extraction (Aqueous Samples)

Note: For TCLP or SPLP leachate extraction procedure proceed to Section 10.6

10.1.1. Using a graduated cylinder, transfer a 250 ml sample aliquot to a 500 ml separatory funnel. Spike the sample with surrogate compound(s).

10.1.2. Add 50 ul of 100 ppm 2, 4-Dichlorophenylacetic Acid (DCAA) Surrogate Spiking Solution (see Attachment 2) to each sample and QC sample. NOTE: when spiking the samples with either surrogates or target spiking compounds, ensure that there are no bubbles in the syringe. In addition, hold the syringe just above the level of the water when adding the spike. Do not touch the tip of the syringe to the water or to the side of the separatory funnel. Swirl the separatory funnel after adding surrogate spike. All spikes must be witnessed.

10.1.3. When preparing QC spike samples (MS/MSD/LCS), spike 100ul of 12 compound mix (see Tables 1 and 2).

10.1.4. Add 20 g of NaCl to the sample, seal, and shake to dissolve the salt.

10.1.5. Use this step only if herbicide esters, in addition to herbicide acids, are to be determined.

10.1.6. Add 5 mL of 6 N NaOH to the sample, seal, and shake. Check the pH of the sample with pH paper. If the sample does not have a pH greater than or equal to 12, adjust the pH by adding more 6 N NaOH. Let the sample sit at room temperature until the hydrolysis step is completed (usually 1 - 2 hours), shaking the separatory funnel and contents periodically.

10.1.7. Add 15 mL of methylene chloride to the sample bottle and rinse both the bottle and the graduated cylinder. Transfer the methylene chloride to the separatory funnel and extract the sample by vigorously shaking the funnel for 2 minutes, with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 minutes. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation, or other physical methods. Discard the methylene chloride phase.

10.1.8. Add a second 30-mL volume of methylene chloride to the separatory funnel and repeat the extraction procedure a second time, discarding the methylene chloride layer. Perform a third extraction in the same manner.

10.1.9. Add 5 mL of 12 N sulfuric acid to the sample, and shake to mix. Check the pH of the sample with pH paper. If the sample does not have a pH less than or equal to 2, adjust the pH by adding more acid.

10.1.10. Add 30 mL diethyl ether to the sample, seal, and extract the sample by vigorously shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum techniques to complete the phase separation depends upon the sample, but may include stirring, filtration through glass wool, centrifugation, or other physical methods. Remove the aqueous phase to a 1-L Erlenmeyer flask and collect the ether phase in a 100-mL Erlenmeyer flask containing approximately 10 g of acidified anhydrous sodium sulfate. Periodically, vigorously shake the extract and drying agent.

10.1.11. Return the aqueous phase to the separatory funnel, add 15 mL of diethyl ether to the sample, and repeat the extraction procedure a second time, combining the extracts in the 100-mL Erlenmeyer

flask. Perform a third extraction with 15 mL diethyl ether in the same manner. Allow the extract to remain in contact with the sodium sulfate for approximately 2 hours.

10.1.12. NOTE: The drying step is very critical to ensuring complete esterification. Any moisture remaining in the ether will result in low herbicide recoveries. The amount of sodium sulfate is adequate if some free flowing crystals are visible when swirling the flask. If all of the sodium sulfate solidifies in a cake, add a few additional grams of acidified sodium sulfate and again test by swirling. The 2 hour drying time is a minimum, however, the extracts may be held in contact with the sodium sulfate overnight.

10.2. Esterification

Esterification By TMSD

10.2.1. Add approximately 200uL of methanol to the extract followed by approximately 200uL of 2.0M Trimethylsilyldiazomethane (TMSD) solution. The extract will turn a yellow color. If this does not occur, add additional (approximately) 200uL aliquots of 2.0M Trimethylsilyldiazomethane (TMSD) solution until the yellow color persists. Check the sample every 15 minutes for yellow color. If the yellow disappears in this time frame, continue adding an additional aliquot of 2.0M Trimethylsilyldiazomethane (TMSD) solution until the yellow color persists.

10.2.2. Allow the extract to stand for at least one hour at room temperature to allow the methylation reaction to occur. The reaction is halted with the addition of approximately 0.05g (one scoop using a disposable spatula) of silica gel. The extract is then brought up to a final volume of 10 mL with hexane by visually comparing it to a calibrated collection tube.

10.2.3. Adjust the final extract volumes to 10ml with hexane. The extracts ready to be analyzed by GC. Transfer some volume to GC autosampler vials.

10.2.4. Samples must be analyzed as soon as possible (less than 24 hrs. preferably) after esterification. This is to minimize the trans-esterification and other potential reactions that may occur.

10.2.5. Store refrigerated if further processing will not be performed immediately. Extracts should be stored at 4°C away from light.

10.3. Esterification By Diazald

10.3.1. Add 10 ml of DI water and 15 ml of Carbitol (diethylene glycol monoethyel ether) to a 250-ml Erlenmeyer flask. Dissolve in 6 g of KOH

10.3.2. Prepare a 2-hole rubber stopper holding a 60 ml separatory funnel in one hole, and a length of U-shaped tubing in the other hole. Attach the 2-hole rubber stopper to the Erlenmeyer flask.

10.3.3. Prepare a diazald solution by adding 10 ml of ether to the 60 ml separatory funnel and dissolving in 2 g of diazald.

10.3.4. Place the Erlenmeyer flask/separatory funnel apparatus on a stirring hot plate. Allow the diazald to slowly drip into the Erlenmeyer. (The hot plate setting should never exceed setting of 2.)

10.3.5. Rinse the end of the U-shaped tubing 3 times with hexane, then place into extract allowing the diazomethane gas to bubble through the extract. The extract should turn a yellow color signaling that esterification is complete.

10.3.6. After esterification, remove excess diazomethane gas by adding a few grams of silicic acid to the extract and bubbling nitrogen gas through the extract on the N-Evap for several minutes.

10.3.7. Adjust the final extract volume of 10 ml with Hexane (5 ml for TCLP). The extracts ready to be analyzed by GC.

10.3.8. Samples must be analyzed as soon as possible (less than 24 hrs. preferably) after esterification. This is to minimize the trans-esterification and other potential reactions that may occur.

10.3.9. Store refrigerated if further processing will not be performed immediately.

10.3.10. Extracts should be stored at 4°C away from light.

10.4. Extract Concentration

10.4.1. Concentrate samples in an N-Evap apparatus making sure the control the temperature below 40°C such that the concentrator tube is partially immersed in the hot water and the entire lower surface of the tube is bathed with hot water.

10.4.2. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-20 minutes.

10.4.3. Evaporate samples until the sample volume is approximately 2 ml.

10.4.4. Add 15 ml of hexane and concentrate further to a final volume of approximately 3 ml.

10.4.5. Remove the sample from the Evaporator and cool for about 5 minutes.

10.5. Sample Extraction (Leachate Samples)

10.5.1. Rinse enough 60-ml separatory funnels and 500-ml Erlenmeyer flasks to accommodate the number of samples to be prepped. Each funnel and flask must be rinsed three times: once with a 50:50 mixture of methylene chloride: acetone, once with methylene chloride and once with a 25% sulfuric acid: reagent water solution.

10.5.2. Label the sample on the corresponding separatory funnels and the corresponding 500 ml Erlenmeyer flasks.

10.5.3. Pipette 15 ml of each sample into its corresponding separatory funnel.

10.5.4. Rinse a 100 ml graduated cylinder once with methylene chloride and two to three times with lab reagent water. Using the graduated cylinder obtain 15 ml of lab reagent water from the Millipore filtering apparatus for each of the method blank and the laboratory control sample (LCS) (aka blank spike).

10.5.5. Rinse each syringe to be used for adding surrogates and spikes, two to three times with acetone.

10.5.6. Add 50 ul of 100 ppm 2, 4-Dichlorophenylacetic Acid (DCAA) Surrogate Spiking Solution (see Attachment 1) to each sample and QC sample. NOTE: when spiking the samples with either surrogates or target spiking compounds, ensure that there are no bubbles in the syringe. In addition, hold the syringe just above the level of the water when adding the spike. Do not touch the tip of the syringe to the water or to the side of the separatory funnel. Swirl the separatory funnel after adding surrogate spike. All spikes must be witnessed.

10.5.7. When preparing QC spike samples (MS/MSD/LCS), spike 100uL of 12 compound mix (see Tables1 and 2). .Swirl the separatory funnel after adding the spike solution(s).

10.5.8. Add 2 ml of 6N NaOH solution to each sample.

10.5.9. Add 20 ml of diethyl ether.

10.5.10. Extract the samples by vigorously shaking the separatory funnels for 2 minutes, with periodic venting to release excess pressure.

10.5.11. Allow the organic layer to separate from the water phase for a minimum of 10 minutes.

10.5.12. If the emulsion interface between the layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration through glass wool, centrifugation or other physical methods.

10.5.13. Collect the diethyl ether layer for each sample into the corresponding 250-ml Erlenmeyer flask.

10.5.14. Repeat steps 10.6.10 through 10.14 twice, using 10-ml aliquots of diethyl ether each time.

10.5.15. Discard the ether phase after each extraction.

10.5.16. Adjust the pH to $\leq$ 2 by adding approximately 2 ml of 1:3 Sulfuric Acid/Water to the samples. Check pH with pH paper.

10.5.17. Extract three times for two minutes each with ether, using 20 ml the first time, and 10 ml each the second and third times.

10.5.18. Collect all three ether phases into a screw top 125-ml glass jar containing acidified sodium sulfate. Let the sample dry for 2 hours.

10.5.19. Pour the dried extract through a funnel plugged with acid washed glass wool, and collect the extract into the N-Evap tubes.

10.5.20. Rinse the Erlenmeyer flask and funnel with 20 to 30 ml of diethyl ether to complete the quantitative transfer.

10.5.21. Proceed to Section 10.2.1.

10.6. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.6.1. The TALS reagent database must be completed in full with the required information whenever standards are logged and/or prepared.

10.6.2. All required data types (sample volumes, reagent and standard volumes, reagent and standard lots, etc…) must be entered into the TALS batch database each time an extraction is performed.

.

11.0. Calculations / Data Reduction

None

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

A Method Detection Limit (MDL) study, as described in the Eurofins corporate Detection and Quantitation Limits SOP, CA-Q-S-006, must be performed initially and whenever a significant change affecting sensitivity is made to the analytical system. The MDL must be re-evaluated from quarterly MDL points at least every 12 months..

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to the current revision of Eurofins Edison SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section

13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½ inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-1824-03), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method 8151A: Organochlorine Herbicides by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2. Eurofins Edison SOP No. ED- ORP-023, *Extraction of Organochlorine Herbicides in Soil by SW846 Method 8151A,* current revision.

15.3. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.4. Eurofins Corporate Document No. CW-E-M-001, *Environmental Health and Safety Manual,* current revision.

15.5. Eurofins Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.

15.6. Eurofins Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.7. Eurofins Edison SOP No. ED-GEN-013, *Glassware Cleaning*, most current revision

15.8. Eurofins Edison SOP No. ED-GEN-022, *Training,* current revision.

16.0. Method Modifications:

NONE

17.0. Attachments

Attachment 1 – Surrogate Solution Prep

18.0. Revision History

Revision 16, 09/11/2024

- Updated to Eurofins branding throughout.
- Updated sec. 10.0 Procedure to help with better the herbicide recoveries.
- Added section 10.3 the use of Diazald esterification in case of TMSD shortage.
- Updated equipment with the use of N-Evap in place of the Kuderna Danish apparatus.

Revision 15, 10/5/2021

- Updated Lab Quality Manual section references as needed throughout document.
- Updated throughout to reflect used of TMSD for esterification (replacing diazald)
- Section 12: updated to reflect current MDL procedures.

Revision 14, April 26, 2019

- Section 10.4.11: methanol was added to extract to help with better herbicide recoveries.

- Revision 13, dated February 22, 2016:

  - Updated Table 2 with the corrected spelling of Picloram(4-amino-3,5,6-trichloropicolinic acid.
  - Section 7.1.1.3: corrected the preparation instructions for 12N sulfuric acid. The correct volume of water is 200ml (was 300 ml). 100ml concentrated sulfuric diluted into 200 ml water is 12N.
  - Section 7.1.1.5: clarified to indicate that a 25% H2SO4 solution is a 1:3 solution.

- Revision 12, dated January 28, 2016:

  - Sections 7.2.1 and 7.3.1: updated info to reflect prep using new standard solutions.
  - Tables 1 and 2 updated with new standard mixes and sources.
  - Attachment 1: deleted. Attachment 2 now becomes Attachment 1.
  - Section 7.3.2.1: revised to reflect new standards and procedures.
  - Section 7.3.3: updated to revise from Attachment 2 to Attachment 1.
  - Section 8.0: added 8 oz amber glass container as option.
  - Section 10.1.1: added 500 ml separatory funnel as option.
  - Section 10.1.9 and 10.6.8: updated spiking instructions.
  - Section 10.4.11: updated and clarified final extract volumes.

- Revision 11, dated January 27, 2014::

  - Minor editorial changes throughout document.
  - Section 7.2.2: Update standard sources and mixes (Table 1).
  - Section 7.3.2 and 7.3.3: Updated standards prep information.
  - Section 10.1.9: updated QC spiking procedures (LCS/MS/MSD).
  - Section 10.6.8: updated leachate QC spiking procedures.
  - Added Attachment 1 which details the individual target compounds and their concentrations in the new standards.
  - Added Attachment 2 which details the individual surrogate compounds and their concentrations in the new standards.

- Revision 10, dated January 6, 2012:

  - Section 7.2.2, Table 2: updated concentration of MCPP spiking mix.
  - Section 7.2.2, Tables 1 and 2: Updated source of Picloram spiking standard.
  - Section 7.3.2.4: revised instructions for preparation of Picloram spiking solution.
  - Corrected number of Sections 7.3.2.1 thru 7.3.2.4.
  - Revised Section 10.6.8: corrected typos and added detailed procedures dependent upon spiking scenarios.

- Revision 9, dated December 21 2011:

    - Revised the following sections to reflect the requirement that all glassware must be rinsed with sulfuric acid prior to use in the extraction of herbicides: Section 6.2, Section 10.1.1 and Section 10.6.1.
    - Added Section 7.1.1.5 which describes the preparation of a 25% sulfuric acid: reagent water solution for use in acid-rinsing glassware.
    - Throughout document: updated LQM section references as appropriate as some have changed with the latest LQM revision

- Revision 8, dated August 26 2011:

    - Section 5.1: revised to include following statement: “Ethyl Ether must be free of peroxides as determined by use of EM Quant Peroxide Test Strips as described in Sections 6 and 7 below.”
    - Section 6.2: added EM Quant Peroxide Test Strips to supplies list.
    - Section 7.1: added requirement to test Ether for peroxide formation.
    - Revised Section 10.3.4 to include a requirement to pour extract through a funnel plugged with acid washed glass wool. Also revised collection container (was Erlenmeyer flask; now K-D concentrator).
    - Section 10.3.9 was revised to clarify that the ether extracts from each sample are collected into a single container (not multiple separate containers).
    - Section 10.6.8: corrected the spike volume used for the 10 Compound Spike mix. Reworded the section to make the instructions clearer.

- Revision 7, dated October 30, 2009
    - Revised SOP format in accordance with Eurofins Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
    - Section 1.1 Added reference to Quality Assurance Manual for method modifications.
    - Section 1.1: Expanded to include references to applicable prep and cleanup SOPs.
    - Section 3: revised to reference new location for definitions.
    - Section 5: Revised to include most up to date corporate health and safety references and information.
    - Section 7.2.2: Added tables detailing components found in the various standards mixes.
    - Section 7.3: Updated the instructions for preparation of standards.
    - Section 7.3: Added tables with calibration standards prep details.
    - Section 8: Updated with additional details including a table outlining containers, preservation and holding times for waters and soils.
    - Section 9.1: Expanded QC sample preparation details.
    - References: Expanded to include more specific SOP references
    - Section 18: Added this Revision History section
    - Throughout document: added references to Eurofins LIMS (TALS).

Attachment 1 – Surrogate Standards Preparation

TestAmerica
THE LEADER IN ENVIRONMENTAL TESTING

Reagent ID: OP_HERB_SU_00020

		Expiration Date:	06/10/2014
Description:	HERBICIDE Surrogate	Laboratory:	TestAmerica Edison
No. of Bottles:	10	Prepared By:	Patel, Hemex
Storage Location:	ORP-STDS & SPIKES	Solvent:	Acetone
Reagent Volume:	100.000 mL	Solvent Lot:	33214
Creation Date:	12/10/2013		
Container(s):	2644797, 2644798, 2644799, 2644800, 2644801, 2644802, 2644803, 2644804, 2644805, 2644806		
Comment:			

Reagent Analyte Information

Analyte	Source ID	Source Exp. Date	Source Conc.	Source Conc. Units	Final Conc.	Final Conc. Units
2,4-Dichlorophenylacetic acid	OP_2,4DCAA_00016	09/30/2014	1000.00000	ug/mL	100.00000	ug/mL

Source Reagents

Reagent	Description	Type	Expiration	Vendor	Vendor Lot #	Vendor Cat Lot #	Volume Used	Volume Units
OP_2,4DCAA_00016	2,4 - DCAA Acid Spike Mix	ASTD	09/30/14	RESTEK	A093820	567804	10.00000	mL

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Title: Extraction of Organochlorine Herbicides in Soil by SW846 Method 8151A

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Approvals (Signature/Date):

06/05/2024		06/05/2024	
Mamdou Leye	Date	Dan Helfrich	Date
Organic Prep Department Manager		Health & Safety Manager / Coordinator	
06/05/2024		06/05/2024	
Carl Armbruster	Date	Mark Acierno	Date
Quality Assurance Manager		Laboratory Director	

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1.0 Scope and Application

1.1. Analytes, Matrix(s), and Reporting Limits: SW846 Method 8151A provides extraction and derivatization procedures for the gas chromatographic analysis of chlorinated acid herbicides in water, soil, and waste samples. An option for the hydrolysis of esters is also described. This SOP details the extraction of soil samples with diethyl ether follower by esterification with diazomethane (or diazald) The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The results are reported as acid equivalents.

1.2. For a complete discussion of analytes and reporting limits (RLs) please refer to Eurofins Edison SOP No. ED-GCS-005, Analysis of Organochlorine Herbicides by SW846 Method 8151A, current revision.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 9 (Service to the Client) and 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

This SOP provides extraction and derivatization procedures for the determination of chlorinated acid herbicides and their esters in solid samples. Soil samples are serially extracted with diethyl ether. The extracts are hydrolyzed, dried and concentrated and then esterified with Trimethylsilyl diazomethane. The derivatives are determined by gas chromatography with an electron capture detector (GC/ECD). The sensitivity of Method 8151 depends on the level of interferences in addition to instrumental limitations.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

Method interference 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 interference under the conditions of the analysis, by analyzing reagent blanks.

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.

Matrix interference may be caused by contaminants that are co-extracted from the sample. Organic acids, especially chlorinated acids, cause the most direct interference with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure.

The acid forms of the analytes are strong organic acids that react readily with alkaline substances and can be lost during sample preparation. Glassware and glass wool must be acid-rinsed with 1 N hydrochloric acid and the sodium sulfate must be acidified with sulfuric acid prior to use to avoid analyte losses due to adsorption.

Sample extracts must be dry prior to methylation or else poor recoveries will result.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Ethyl Ether is an extremely flammable solvent. If a mechanical device is used for sample extraction, the device should be equipped with an explosion proof motor and placed in a hood.

Ethyl Ether must be free of peroxides as determined by use of EM Quant Peroxide Test Strips as described in Sections 6 and 7 below.

Diazomethane can explode under certain conditions therefore take the following precautions:

- Use a well-ventilated hood.
- Use a safety screen.
- Do not heat above 90 degree Celsius.
- Store away from alkali metals.
- Solutions of diazomethane decompose rapidly in the presence of solid materials such as copper powder, calcium chloride, and boiling chips.

The use of Kevlar gloves is required for the assembly/disassembly of ground glass joints in addition to those tasks that present the potential risk for injury.

Ether creates excessive pressure very rapidly. Therefore, initial venting should be done immediately after the separatory funnel has been sealed and inverted. Vent into hood away from analysts and other samples.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sodium Hydroxide	Corrosive Poison	2 ppm, 5 mg/m^3	This material will cause burns if comes into contact with the skin or eyes. Inhalation of Sodium Hydroxide dust will cause irritation of the nasal and respiratory system.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Ethyl Ether	Flammable Irritant Peroxide Former	400 ppm TWA	General anesthesia by inhalation can occur. Continued exposure may lead to respiratory failure or death. Early symptoms include irritation of nose and throat, vomiting, and irregular respiration, followed by dizziness, drowsiness, and unconsciousness. May cause irritation, redness and pain to the eyes. Irritating to the skin and mucous membranes by drying effect. Can cause dermatitis on prolonged exposure. May be absorbed through skin. May form explosive peroxides on long standing or after exposure to air or light. This material must be disposed of within six months.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Equipment

- Top loading Balance - Sargent Welch S2642-25 or equivalent
- N-Evap - Meyer Analytical Evaporator Model No. 112 or equivalent
- Explosion Proof Hood

6.2. Supplies

- 250 ml Clear Glass Jar
- 500 ml Erlenmeyer Flask
- 125 ml Erlenmeyer Flask
- 125 ml Separatory Funnel
- 10 ml graduated Concentrator Tube - Kontes K570050-1025 or equivalent.
- 10 ml glass vials with Teflon lined screw caps.

- 20 ml glass vials with Teflon lined screw caps.
- 5 ml to 1000 ml Class A Volumetric flasks
- Funnel - 75 mm diameter
- 50 ul Gastight Syringe - VWR 60376-241 or equivalent.
- 100 ul Gastight Syringe - Baxter S9662-5 or equivalent.
- 100 ml Graduated Cylinder
- 1 ml Pasteur Disposable Pipette
- 10 ml Disposable Pipette
- Acid Washed Glass Wool – Fisher cat # B0773KMDPK or equivalent.
- pH Paper
- Tongue Depressors
- EM Quant Peroxide Test Strips, EMD No. 10011-1 or equivalent

7.0 Reagents and Standards

7.1 Reagents

Note: Each lot of Methylene Chloride, Acetone, Methanol and Sulfuric Acid is screened for contaminants before being used for analysis as detailed in Eurofins Quality SOP No. CA-Q-S-001 (Solvent & Acid Lot Testing & Approval) and Eurofins Edison SOP No. ED-GEN-023 (Bulk Solvent Testing and Approval).

- Trimethylsilyl diazomethane (TMSD), 2.0 molar solution in hexane, Acros 429211000 or equivalent.
- Diethyl Ether (Residue Analysis Grade with 2% Ethanol preservative) - Baker 9259-03 or equivalent (tested for peroxide formation upon opening and monthly thereafter by use of EM Quant Peroxide Test Strips)
- Methylene Chloride (High Purity Grade) - Baker 9266 or equivalent.
- Acetone (High Purity Grade) – Baker 9254 or equivalent.

- Sodium Sulfate Anhydrous Crystals (Reagent Grade) – Fisher or equivalent. (Baked at 400□C for 4 hours)
- Hexane (High Purity Grade) – Baker 9262 or equivalent.
- Potassium Hydroxide (Analytical Reagent Grade) – Aldrich 306568 or equivalent
- Sulfuric Acid (Analytical Reagent Grade) – FisherA300S212 or equivalent.
- Peroxide Test Strips - Aldrich Z10, 168-0 or equivalent
- Organic free reagent water - Reagent water is defined as a water in which an interference is not observed at the MDL of each parameter of interest (ASTM Specification D1193, Type ii). At Eurofins Edison this water is generated by the Barnstead/Thermolyne Water System (Model # D11991 Serial # 1191020210415).
- Diazald - Aldrich D2, 800-0 or equivalent.

7.1.1 Reagent preparation

7.1.1.1 Anhydrous sodium sulfate crystals must be baked in the muffle furnace at 400 degrees C for 4 hours prior to use. After baking, the sodium sulfate must be acidified. Place 500 grams of anhydrous sodium sulfate into a 500 ml wide mouth Erlenmeyer flask. Add enough diethyl ether to cover the sodium sulfate. Add 0.5 ml concentrated sulfuric acid. Shake vigorously for 1 minute. Evaporate the ether by placing the flask on steam bath. Mix 1 g of the resulting solid with 5 ml of organic-free reagent water and measure the pH of the mixture. It must be below a pH of 4. Prepare fresh daily.

7.1.1.2 37% KOH Solution: Rinse a 1 liter Pyrex flask three times with organic free reagent water. Weigh 148 g KOH into flask and add 400 ml organic free reagent water. Mix until the KOH is completely dissolved and the flask has cooled to room temperature. Transfer the KOH solution to a clean 500-ml amber bottle and label.

7.1.1.3 12N H2SO4: Rinse a 1 liter Pyrex flask three times with organic free reagent water. Add 200 ml organic free

reagent water to the flask. Slowly, and while mixing, add 100-ml concentrated H2SO4 to flask. Continue mixing until flask has cooled to room temperature. Transfer the H2SO4 solution to a clean 500-ml amber bottle and label.

7.1.1.4 25% H2SO4 solution: 25 ml Acid and 75 ml reagent water. Add 75 ml organic free reagent water to a graduated cylinder. Slowly, and while mixing, add 25-ml concentrated H2SO4 to cylinder. Continue mixing until cylinder has cooled to room temperature. Transfer the H2SO4 solution to a clean amber bottle and label.

7.2 Standards

7.2.1 Standards are purchased as concentrated solutions (see Section 7.2.2). Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent as described in Section 7.3.

7.2.2 Standard mixes and sources: Table 1 lists the standard concentrate mix sources. Table 2 details the individual components of these mixes.

Table 1:
Herbicide Standard Mixes and Sources*

Standard Name ("Lab Name")	Concentration (ug/ml)	Source
Acid Herbicide LCS RTS ("Spiking Mix")	varies (in Methanol)	Restek Catalog # 567950
DCAA standard	1000 (in methanol)	Restek Catalog #567804

*May be substituted with equivalent standards from alternate sources.

Table 2:
Components of Herbicide Standard Mixes

Parameter	Catalog Nos.	Lab Standard Name	Concentration (ug/ml)
2,4-dichlorophenoxyacetic acid (2,4-D)	Restek Catalog # 567950	Spiking Mix	20
2-(2,4,5-Trichlorophenoxy)propionic acid (2,4,5-TP aka Silvex)	Restek Catalog # 567950	Spiking Mix	5
2,4,5-T	Restek Catalog # 567950	Spiking Mix	5
2,4-DB	Restek Catalog # 567950	Spiking Mix	20
Dalapon	Restek Catalog # 567950	Spiking Mix	20

Table 2: Components of Herbicide Standard Mixes			
Parameter	Catalog Nos.	Lab Standard Name	Concentration (ug/ml)
Dicamba	Restek Catalog # 567950	Spiking Mix	10
Dichloroprop	Restek Catalog # 567950	Spiking Mix	20
Dinoseb	Restek Catalog # 567950	Spiking Mix – 10 compound	20
MCPA acid	Restek Catalog # 567950	Spiking Mix – 10 compound	2000
MCPP acid	Restek Catalog # 567950	Spiking Mix – 10 compound	2000
Pentachlorophenol	Restek Catalog # 567950	Pentachlorophenol spike	5
Picloram (4-amino-3,5,6trichloropicolinic acid)	Restek Catalog # 567950	Picloram Acid Spiking Standard	20
2,4-Dichlorophenylacetic Acid	Restek Catalog # 567804	Surrogate Spiking Solution	1000

7. 3. Standards Preparation

7.3.1. All standard stock solutions are diluted to the working concentrations as required with acetone (as indicated) using Class A volumetric glassware.

7.3.2. Herbicide Spiking Standard (LCS/MS/MSD):

7.3.3.1 Herbicide QC Spiking Standard Mix (12 compound analysis): The (12) compound spiking standard is ready to spike and used when analyzing for the standard list of herbicides The spiking standard mix is a 25 ml solution (Restek Catalog # 567950; see Tables 1 and 2) of the 12 compounds at the specified concentrations as free acids (see Table 2). The concentrate is used without further dilution prior to the spiking of the following quality control sample types: MS, MSD, and LCS. Spiking instructions are found in Section 10.

7.3.3. Herbicide Surrogate Spiking Standard (2,4-DCAA Acid Spike Mix): The herbicide surrogate spiking solution is a 1 ml solution of 2,4-DCAA at a concentration of 1000 ug/ml. (Restek Catalog # 867804,

see Tables 1 and 2). The concentrate is used after 10X dilution in acetone prior to the spiking of samples (final concentration of spiking standard is 100 ug/ml).

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Soil	Glass	8oz clear glass	Cool 4 ± 2°C	14 days to extraction; 40 days to analysis	USEPA SW846

8.1. Extracts must be stored under refrigeration (Cool 4 ± 2°C) in the dark and analyzed within 40 days of extraction.

9.0 Quality Control

9.1 Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS) [1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS) [2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD) [2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]

[1] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.
[2] The sample selection for MS/MSD is randomly selected by the organic prep lab, unless specifically requested by a client.
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into TALS (LIMS).

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank

must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to ensure that the analytical system is in control. It is also used to assess the performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated acceptance criteria. If the spiked sample recovery results (MS/MSD) fall outside the laboratory generated limits (refer to the current active TALS method limit group database), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QC batch must be re-extracted and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to refer to the current active TALS method limit group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in Eurofins Edison SOP No. ED-GCS-005, Analysis of Organochlorine Herbicides by SW846 Method 8151A, current revision.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with surrogate standard solution containing 2,4-DCAA (see Section 7.3.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to refer to the current active TALS method limit group database). If the surrogate recovery limits are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

10.0. Procedure

10.1. Sample Extraction and hydrolysis (Soil Samples)

10.1.1. Rinse enough graduated cylinders and flasks once with acetone, twice with diethyl ether and once with 25% sulfuric acid: reagent water.

10.1.2. Weigh out 30g (+/- 0.1g) of soil sample into a clean clear glass 8oz sample container. Mark the lab sample identification numbers on the corresponding sample containers with red wax pencil.

10.1.3. Make up labels for each sample with the following information and place on the corresponding 500 ml Erlenmeyer flasks.

10.1.4. Weigh out approximately 30 g of sodium sulfate into two clean glass jars. These will serve as the blank and LCS samples respectively.

10.1.5. Rinse each syringe to be used for adding surrogates and spikes with acetone.

10.1.6. Add 50 ul of 100 ppm 2,4-Dichlorophenylacetic Acid (DCAA) Surrogate Spiking Solution (see Table 1) to each sample and QC sample. NOTE: Hold the syringe just above the level of the soil when adding the spike. Do not touch the tip of the syringe to the soil or to the side of the glass jar. Swirl the separatory funnel after adding surrogate spike. All spikes must be witnessed.

10.1.7. When preparing QC spike samples (MS/MSD/LCS/LCSD), spike with 250ul of the Herbicide QC Spiking Standard Mix (Restek 569750, see Section 7.3.3.1). Swirl the glass jar after adding the spike. All spikes must be witnessed.

10.1.8. Adjust the pH of each sample and QC sample to 2 with concentrated HCl (about 5ml) and monitor the pH for 15 minutes with occasional stirring. If necessary, add additional HCl until the pH remains at 2.

10.1.9. Add 20 ml of acetone to each sample. Mix each sample with the wrist shaker for 20 minutes.

10.1.10. Add 60 ml of diethyl ether to the each sample and shake again for 20 minutes. Decant the extract and measure the volume of solvent recovered.

10.1.11. Extract the sample twice more using 20 ml of acetone followed by 60 ml of diethyl ether. After addition of each solvent, the mixture should be shaken with the wrist shaker for 10 minutes and the acetone-ether extract decanted.

10.1.12. After the third extraction, the volume of extract recovered should be at least 75% of the total volume of solvent added. If solvent recovery is less than 75%, extract once more with 20 ml of diethyl ether.

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10.1.13. Check the pH of the extract. If it is not at or below pH 2, add more concentrated HCl until stabilized at pH of 2.

10.1.14. Into a pre-rinsed 500ml separatory funnel add 250ml of organic free reagent water.

10.1.15. Add 5ml of H2SO4 to the reagent water and check with pH paper to insure pH is <2.

10.1.16. Add the solvent extract to separatory funnel and gently mix the contents of the separatory funnel for 1 minute and allow the layers to separate.

10.2. Partitioning

10.2.1. Add 2.0 ml of the 37% KOH solution, 15 ml of reagent water.

10.2.1. Allow it to cool for 5-10 minutes.

10.2.2. Pour the extract into a 500-ml separatory funnel pre-rinsed with diethyl ether.

10.2.3. Add 40 ml of diethyl ether to the separatory funnel and shake for two minutes.

10.2.4. Pour the dried diethyl ether extract through a funnel plugged with acid washed glass wool, collecting the extract in the K-D concentrator. Use a glass rod to crush any caked sodium sulfate during the transfer.

10.2.5. Repeat twice using 20-ml aliquots of diethyl ether.

10.2.6. Discard the ether phase.

10.2.7. Adjust the pH of the extract remaining in the 500-ml separatory funnel by adding 5 ml of 12N Sulfuric Acid. Check pH with pH paper.

10.2.8. Add 40 ml of diethyl ether to the separatory funnel and shake for 2 minutes with periodic venting to release pressure.

10.2.9. Collect each diethyl ether phase into one pre-cleaned screw top glass jars containing approximately 10 g of acidified sodium sulfate crystals and mix well.

10.2.10. Repeat sections10.2.8 and 10.2.9 twice using 20-ml aliquots of diethyl ether.

10.2.11. Allow the diethyl ether extract to remain in contact with the sodium sulfate for approximately 2 hours.

10.2.12. Pour the dried diethyl ether extract through a funnel plugged with acid washed glass wool, collecting the extract in the Evaporator concentrator. Use a glass rod to crush any caked sodium sulfate during the transfer.

10.2.13. Place the white identification tag on the Evaporator apparatus.

10.2.14. Rinse the Erlenmeyer flask and funnel with 20 to 30 ml of diethyl ether to complete the quantitative transfer.

10.4. Extract Concentration

10.4.1. Blow down the sample in the concentrator until the sample volume is approximately 1 ml.

10.4.2. Add 30 ml of hexane and concentrate further on the evaporator to a final volume of approximately 10 ml.

10.4.3. Remove the samples from the evaporator and allow it to drain and cool for at least 10 minutes.

10.4.4 Extracts will be esterified either by TMSD or by Diazald

10.5. Esterification by TMSD

10.5.1. Add approximately 200 uL of methanol to the extract followed by approximately 200 uL of 2.0M Trimethylsilyl diazomethane (TMSD) solution. The extract will turn a yellow color. If this does not occur, add additional (approximately) 200 uL aliquots of 2.0M Trimethylsilyl diazomethane (TMSD) solution until the yellow color persists. Check the sample every 15 minutes for yellow color. If the yellow disappears in this time frame, continue adding an additional aliquot of 2.0M Trimethylsilyldiazomethane (TMSD) solution until the yellow color persists.

10.5.2. Allow the extract to stand for at least one hour at room temperature to allow the methylation reaction to occur. The reaction is halted with the addition of approximately 0.05g (one scoop using a disposable spatula) of silica gel. The extract is then brought up to a final volume of 10 mL with hexane by visually comparing it to a calibrated collection tube.

10.5.3. Adjust the final extract volumes to 10ml with hexane. The extracts ready to be analyzed by GC. Transfer some volume to GC autosampler vials.

10.5.4. Samples must be analyzed as soon as possible (less than 24 hrs. preferably) after esterification. This is to minimize the transesterification and other potential reactions that may occur.

10.5.5. Store refrigerated if further processing will not be performed immediately.

10.5.6. Extracts should be stored at 4 deg C away from light.

10.6 Esterification by Diazald

10.6.1 Add 10 ml of DI water and 15 ml of Carbitol (diethylene glycol monoethyel ether) to a 250-ml Erlenmeyer flask. Dissolve in 6 g of KOH

10.6.2 Prepare a 2-hole rubber stopper holding a 60 ml separatory funnel in one hole, and a length of U-shaped tubing in the other hole. Attach the 2-hole rubber stopper to the Erlenmeyer flask.

10.6.3 Prepare a diazald solution by adding 10 ml of ether to the 60 ml separatory funnel and dissolving in 2 g of diazald.

10.6.4 Place the Erlenmeyer flask/separatory funnel apparatus on a stirring hot plate. Allow the diazald to slowly drip into the Erlenmeyer. The hot plate setting should never exceed 2.

10.6.5 Rinse the end of the U-shaped tubing 3 times with hexane, then place into extract allowing the diazomethane gas to bubble through the extract. The extract should turn a yellow color signaling that esterification is complete.

10.6.6 After esterification, remove excess diazomethane gas by adding a few grams of silicic acid to the extract and bubbling nitrogen gas through the extract on the N-Evap for several minutes'

10.6.7 Adjust the final extract volume of 10 ml with Hexane (5 ml for TCLP). The extracts are ready to be analyzed by GC.

10.6.8 Samples must be analyzed as soon as possible (less than 24 hrs. preferably) after esterification. This is to minimize the trans-esterification and other potential reactions that may occur.

10.6.9 Store refrigerated if further processing will not be performed immediately.

10.6.10 Extracts should be stored at 4 deg C away from light.

10.7. Required Documentation:

The organic prep technician is responsible for completing the following items.

10.7.1. The TALS reagent database must be completed in full with the required information whenever standards are logged and/or prepared.

10.7.2. All required data types (sample volumes, reagent and standard volumes, reagent and standard lots, etc…) must be entered into the TALS batch database each time an extraction is performed.

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11.0. Calculations / Data Reduction

None

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The MDL is the minimum measured concentration of a substance that can be reported with 99% confidence that the measured concentration or amount is distinguishable from method blank results. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM) consistent with 40CFR Part 136 Appendix B, August, 2017. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to the current revision of Eurofins Edison SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental

management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."

14.2. The following waste streams are generated as a result of this analysis:

- Extractions Waste water. This material is created when 50% Acetone and 50% Methylene Chloride are added to 1 liter of sample water. The water is shaken with the solvent. The solvent is collected with the compounds of interest and the water is discarded into the Extractions Waste Water drum. This drum is removed to the walk-in hood in the waste room. A ½ inch PVC pipe is inserted into the bung hole of the drum and air is passed through the solution over night. The solution is then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9. This solution is discharged into the municipal sewer system.

- Mixed Solvent Waste. This material is collected from rinsing and other processes into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.
  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Waste sodium sulfate. This material is collected from various methods which require the removal of water from solvent which carries the analyte (s) of interest. The solvent is passed through the sodium sulfate and the sodium sulfate plus the water is disposed of. The sodium sulfate is collected in buckets inside the hoods. The material is air dried and disposed of in the municipal waste dumpster.

- Waste sulfuric acid. This material is generated from clean up of PCB extracts for sulfur compounds. The acid is collected in satellite accumulation in the hood. The container is removed to the waste room for neutralization with 50 % sodium hydroxide (Siedler Chemical SC-182403), water and sodium bicarbonate (Siedler Chemical SC-0219-25). Ice is used to control temperature in the plastic drums of the neutralization system. When neutralization is complete (pH 6 -9) the material is transferred to the municipal sewer system.

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, “Method 8151A: Organochlorine Herbicides by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

15.2. Eurofins Edison SOP No. ED- ORP-015, Extraction of Organochlorine Herbicides in Water by SW846 Method 8151A, current revision.

15.3. Eurofins Edison Document No. ED-QA-LQM, Laboratory Quality Manual, current revision.

15.4. Eurofins Corporate Document No. CW-E-M-001, Environmental Health and Safety Manual, current revision.

15.5. Eurofins Corporate Quality SOP No. CA-Q-S-001, Solvent & Acid Lot Testing & Approval, current revision.

15.6. Eurofins Edison SOP No. ED-GEN-023, Bulk Solvent Testing and Approval, current revision.

15.7. Eurofins Edison SOP No. ED-GEN-013, Glassware Cleaning, most current revision

15.8. Eurofins Edison SOP No. ED-GEN-022, Training, current revision.

16.0. Method Modifications:

NONE

17.0. Attachments

NONE

18.0. Revision History

- Revision 13, dated 05 Jun 2024:
  - Removed all KD apparatus no longer used
  - Added the Diazald esterification procedure in case we run out of TMSD.
  - Removed all equipment, reagents, supplies and glassware no longer used.
  - Updated throughout to reflect Eurofins branding.

- Revision 12, dated Oct 08 2020:
  - Section 7.1: added Trimethylsilyldiazomethane (TMSD) , 2.0 molar solution in hexane, Acros 429211000 or equivalent. Removed Diazald..
  - Section 10.5.1: replaced the Diazald esterification procedure with the TMSD procedure.

- Revision 11, dated April 26, 2019
  - Section 10.4.11: methanol was added to extract to help with better herbicide recoveries.

- Revision 10, dated February 22, 2016:
  - Revised throughout to correct the spelling of Picloram (4-amino-3,5,6trichloropicolinic acid
  - Section 7.1.1.3: corrected the preparation instructions for 12N sulfuric acid. The correct volume of water is 200ml (was 300 ml). 100ml concentrated sulfuric diluted into 200 ml water is 12N.

- Revision 9, dated January 06, 2016:
  - Tables 1 and 2: updated sources and concentrations of standards.
  - Section 7.3.1 revised to specify acetone as the diluent.
  - Section 7.3.3.1 (spiking solution prep): updated to reflect current standards and procedures. Sections 7.3.3.2, 7.3.3.3 and 7.3.3.4 deleted (obsolete).
  - Section 7.3.3 (surrogate spiking standard prep): updated to reflect current standards and procedures.
  - Section 8.0: updated minimum sample size (8 oz. glass container).
  - Section 10.1.7 (spiking procedures): updated to reflect current practice and new standard mixes.

- Revision 8, dated December 21, 2011

- Revised the following sections to reflect the requirement that all glassware must be rinsed with sulfuric acid prior to use in the extraction of herbicides: Section 6.2 and Section 10.1.1.
- Added Section 7.1.1.4 which describes the preparation of a 25% sulfuric acid: reagent water solution for use in acid-rinsing glassware.
- Throughout document: updated LQM section references as appropriate as some have changed with the latest LQM revision

- Revision 7, dated August 26, 2011

  - Section 5.1: revised to include following statement: “Ethyl Ether must be free of peroxides as determined by use of EM Quant Peroxide Test Strips as described in Sections 6 and 7 below.”
  - Section 6.2: added EM Quant Peroxide Test Strips to supplies list.
  - Section 7.1: definition and source of Organic-Free Water added.
  - Section 7.1: added requirement to test Ether for peroxide formation.
  - Section 10.1.2: added detail regarding the initial weight of sample to be measured (i.e. ‘Weigh out 30g +/- 0.1g)
  - Revised Section 10.3.4 to include a requirement to pour extract through a funnel plugged with acid washed glass wool. Also revised collection container (was Erlenmeyer flask; now K-D concentrator).
  - Revised Section 10.3.9 to include a procedure for mixing well after adding ethyl ether phase to sodium sulfate.
  - Section 10.6 deleted. Section 10.6 was essentially a duplicate of Section 10.5 that had mistakenly been incorporated into the SOP text.

- Revision 6, dated October 30, 2009

  - Revised SOP format in accordance with Eurofins Corporate Quality SOP No.CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1 Added reference to Quality Assurance Manual for method modifications.
  - Section 1.1: Expanded to include references to applicable prep and cleanup SOPs.
  - Section 3: revised to reference new location for definitions.
    Section 5: Revised to include most up to date corporate health and safety references and information.
  - Section 7.2.2: Added tables detailing components found in the various standards mixes.
  - Section 7.3: Updated the instructions for preparation of standards.
    Section 7.3: Added tables with calibration standards prep details.
  - Section 8: Updated with additional details including a table outlining containers, preservation and holding times for waters and soils.

- Section 9.1: Expanded QC sample preparation details.
- References: Expanded to include more specific SOP references
  Section 18: Added this Revision History section
- Throughout document: added references to Eurofins LIMS (TALS).

Title: Procedure for the Microwave Extraction of Solids, SW846 3546

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Approvals (Signature/Date):

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1.0 Scope and Application

The procedure uses microwave energy to produce elevated temperature and pressure conditions (i.e., 100° -115°C and 50 - 175 psi) in a closed vessel containing the sample (soil, sediment and sludge) and organic solvent(s) to achieve analyte recoveries equivalent to those from Automated Soxhlet extraction (Method 3541), using less solvent and taking significantly less time than the Automated Soxhlet procedure.

This method is applicable to the extraction of semi-volatile petroleum products in soil, sediment and sludge which may then be analyzed by a variety of chromatographic procedures. The method may also be applicable for the extraction of additional target analytes, provided that the analyst demonstrates adequate performance for the intended application as further described in Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

This method only is applicable to solid samples with small particle sizes. If practical the sample may be air dried and ground to a fine powder prior to extraction.

1.1 **Analytes, Matrix(s), and Reporting Limits:** This standard operating procedure details the procedures used by Eurofins Edison for the microwave extraction of soils, sediments, sludges, and waste solids for semivolatile organic compounds by SW846 Method 3546 for subsequent analysis of:

- Diesel Range Organics (C10-C28 & C8-C44) by SW846 Method 8015D (see Eurofins Edison SOP No. ED-GCS-020);
- Organochlorine Pesticides and PCBs by SW846 8081B and 8082A (see Eurofins Edison SOPs No. ED-GCS-016 and ED-GCS-017)
- Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS) by SW846 8270E (Eurofins Edison SOP No. ED-MSS-009)
- PCBs by GC/MS using EPA Method 680 (Eurofins Edison SOP ED-MSS-010)

1.2 The microwave prep for NJDEP Extractable Petroleum Hydrocarbons (EPH) (10/08), current revision, is addressed separately in Eurofins Edison SOP ED-GCS-012 (current revision).

1.3 For a complete discussion of analytes and reporting limits (RLs) please refer to the applicable Eurofins Edison analytical SOPs referenced in Section 1.1 and in References (Section 14.0).

1.4 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 9 (*Review of Work Request*) and 19 (*Test Methods and Method Validation*) of Eurofins Edison's Quality Assurance Manual.

2.0 Summary of Method

A representative soil, clay, sediment, sludge, solid or waste sample is weighed and placed into a 75mL Teflon vessel and extracted using an appropriate solvent in a microwave extractor. The extraction vessel containing the sample and solvent system is heated to the extraction

temperature and extracted for 10 minutes. The mixture is allowed to cool. The vessel is opened and the contents are filtered. The solid material is rinsed and the various solvent fractions are combined. The extract is then dried, concentrated using a nitrogen blow down device, and as necessary, solvent exchanged for use in clean up, fractionation, or determinative methods.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Solvents, reagents, glassware, and other sample processing hardware may contribute to interferences with sample analysis. All materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing a method blanks.

4.2 Phthalate esters are commonly extracted from laboratory items. Plastics in particular, must be avoided because phthalates are easily extracted from these materials. The presence of these contaminants in extracts is problematic because it will give false positive results for the methods that list phthalates as target compounds or it will interfere with the target compounds of other methods that are co-extracted with them.

4.3 All glassware used must be scrupulously cleaned before use in trace analysis. Refer to Eurofins Edison SOP No. ED-GEN-013 (*Glassware Cleaning*), current revision.

4.4 Use caution if sodium sulfate is used for this method. Salts are known to super heat when used in microwave. Typically samples are analyzed with the addition of 5.0 grams sodium sulfate.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Safety Manual, Radiation Safety Manual and this document.

This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1 Specific Safety Concerns or Requirements

Nitrile gloves should be used when performing this extraction. Latex and vinyl gloves provide no significant protection against the organic solvents used in this SOP, and should not be used. During Kuderna-Danish (KD) concentration, do not allow the extract to boil to dryness. The solvent vapors remaining in the KD apparatus may superheat and create an explosion or fire hazard.

The use of the microwave to extract samples creates excessive pressure and temperature within the digestion vessels very rapidly. Cooling the sample after digestion is extremely important.

5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. NOTE: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Instrumentation

- Zymark Turbovap II concentrator
- Electronic Balance, capable of weighing to 0.01g.
- N-Evap Analytical Evaporator
- Fisher 8 Position Steam Bath
- MARS 5 microwave oven with temperature sensor (CEM Corp) capable of sensing temperature within 2.5°C and adjusting microwave field output within 2 sec of sensing.
- MARS 40 position carousel (CEM Corp)
- Visiprep DL column holder

6.2 Supplies

- 10, 50 and 200 mL Concentrator tubes.
- Vials, - 1ml, 4ml, and 10ml with PTFE lined snapcaps.

- Glass syringes, 25ul, 50ul, 100ul, 500ul, 1000ul
- Pastuer pipets
- Glass Buchner funnels (stainless steel funnels may be substituted) – Fisher Scientific.
- 500 ml Kuderna-Danish (KD) flask
- Stainless steal spatulas
- Glass wool- Contaminant-free (silane treated or oven baked at 400^{O}C)
- 75mL Teflon Express vessels with stopper and cap (CEM Corp.)
- Glass fiber filter paper - #1 185mm (Whatman)
- Weigh boats
- Tongue depressors, wood

7.0 Reagents and Standards

Note: Each solvent lot is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.1. Reagents

7.1.1 Organic free reagent water - Reagent water is defined as a water in which an interference is not observed at the MDL of each parameter of interest (ASTM Specification D1193, Type ii). At Eurofins Edison this water is generated by the Barnstead/Thermolyne Water System (Model # D11991 Serial # 1191020210415).

7.1.2 Methylene Chloride, JT Baker Resi-analyzed, P/N 9254-03 (or equivalent)

7.1.3 Hexane – JT Baker Ultra-Resi 73513-42-5 or equivalent

7.1.4 Acetone, J.T. Baker Ultra- ResiAnalyzed, Catalog No. 9264-03 (or equivalent)

7.1.5 Nitrogen, high-purity

7.1.6 Construction Sand (Home Depot or Lowes), free from organic compounds. (Baked in the muffle furnace for four hours at 400^{O}C and stored in a desiccator prior to use.)

7.1.7 Sodium Sulfate Anhydrous Powder, Mallinckrodt MA8020-06 or equivalent (Must be baked in the muffle furnace for four hours at 400^{O}C and stored in a dessicator prior to use.)

7.2. Standards

7.2.1 Stock target analyte standard spiking solutions are purchased as prepared solutions (see table below). Stock solutions are diluted (in volumetric glassware) to a working concentration using methylene chloride ($MeCl_2$) hexane or Acetone as the diluent as indicated below

below. Stock standards of similar quality from other suppliers may be substituted as required.

NOTE: Second sources (from separate lots) are used for quanitation standards and spiking.

Stock Standards (Methods 8015D DRO)				
Standard Mix	**Concentration**	**Source**	**Catalog #**	**Used in method(s):**
o-Terphenyl	10000 ppm	Sigma Aldrich	47580U	*SW846 8015D DRO
Custom TPH Mixed	1000 ppm	Agilent	CUS-3860	*SW846 8015D DRO
Diesel Fuel #2	50000 ppm	Restek	31259	*SW846 8015D

Stock Standards - Pesticides/PCBs* (Methods SW846 8081B and 8082A)				
Standard Mix	**Concentration**	**Source**	**Catalog #**	**Used in method(s):**
Pesticide Surrogate Spike Mix ("Pest/PCB Surrogate")	10 ug/ml	Supelco	861275	SW846 8081B/8082A
Aroclor Spike Mix ("PCB Spike")	100 ug/ml	Supelco	861274	SW846 8082A
SS CLP Organochlorine Pesticides Mix ("Pest Spike")	2000 ug/ml	Supelco	4S7426-U	SW846 8081B
Chlordane (technical) ("Technical Chlordane Spike")	1000 ug/ml	Supelco	48065-U	SW846 8081B
Toxaphene	1000 ug/ml	Supelco	48065-U	SW846 8081B

*May be substituted with equivalent standards from alternate sources.

Components of Pesticide/PCB Standard Mixes (Methods SW846 8081B and 8082A)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
Decachlorobiphenyl (DCB)	Supelco – 4S8913	Pest/PCB Surrogate	10.0
2,4,5,6-Tetrachloro-m-xylene (TCMX)	Supelco – 4S8913	Pest/PCB Surrogate	10.0
Aroclor 1016	Supelco - 861274	PCB Spike	100
Aroclor 1260	Supelco - 861274	PCB Spike	100
Aldrin	Supelco - 4S7426-U	Pest Spike	2000
Alpha-BHC	Supelco - 4S7426-U	Pest Spike	2000
Alpha-Chlordane	Supelco - 4S7426-U	Pest Spike	2000
Beta-BHC	Supelco - 4S7426-U	Pest Spike	2000
Delta-BHC	Supelco - 4S7426-U	Pest Spike	2000
Dieldrin	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan I (Alpha)	Supelco - 4S7426-U	Pest Spike	2000
Endosulfan II (Beta)	Supelco - 4S7426-U	Pest Spike	2000

Components of Pesticide/PCB Standard Mixes (Methods SW846 8081B and 8082A)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
Endosulfan Sulfate	Supelco - 4S7426-U	Pest Spike	2000
Endrin Aldehyde	Supelco - 4S7426-U	Pest Spike	2000
Endrin Ketone	Supelco - 4S7426-U	Pest Spike	2000
Endrin	Supelco - 4S7426-U	Pest Spike	2000
Gamma-BHC (Lindane)	Supelco - 4S7426-U	Pest Spike	2000
Gamma-Chlordane	Supelco - 4S7426-U	Pest Spike	2000
Heptachlor	Supelco - 4S7426-U	Pest Spike	2000
Heptachlor Epoxide (Isomer B)	Supelco - 4S7426-U	Pest Spike	2000
Methoxychlor	Supelco - 4S7426-U	Pest Spike	2000
4,4'-DDD	Supelco - 4S7426-U	Pest Spike	2000
4,4'DDE	Supelco - 4S7426-U	Pest Spike	2000
4,4'-DDT	Supelco - 4S7426-U	Pest Spike	2000
Chlordane (technical)	Supelco – 48065-U	Technical Chlordane Spike	1000
Toxaphene	Supelco 48103	Toxaphene Spike	1000

Stock Standards – BNAs (Method 8270E)			
Standard Mix	**Concentration (ug/ml)**	**Lab Vendor**	**Catalog #**
8270 List 1/ Std#1 MegaMix	500/1000/2000	RESTEK	567672
8270 List 1/ Std#5 N-Nitrosodiphenylamine	2000	RESTEK	567676
8270 List 1/ Std#2 Amines	2000	RESTEK	567673
8270 List 1/ Std#3 Benzoic Acid	2000	RESTEK	567674
Custom SVO Mix	2000	SPEX	SVO-TANJ-16-5
Bisphenol A	1000	SPEX	S-509-MC
8270 List 1/ Std#8	2000	RESTEK	568724
8270 Surrogate Standard	5000	RESTEK	567685
Aromatic Amines Custom Mix	2000	Supelco	21467482

Stock Standards – PCB Homologues (Method 680)			
Standard Mix	**Concentration (ug/ml)**	**Lab Vendor**	**Catalog #**
Retention Time Calibration Standard Mixture	Varies by component (100 to 200)	Ultra	CB-682MN
Concentration Calibration Standard Mix	Varies by component (50 to 250)	Ultra	CB-681MN
13C12-Decachlorobiphenyl Surrogate	40	Cambridge	EC-1410-3
Lindane13C6	100	Cambridge	CLM-1282-S

Components of PCB Homologue Mixes (Methods 680)			
Parameter	**Catalog Nos.**	**Lab Standard Name**	**Concentration (ug/ml)**
PCB-104	Ultra CB-682MN	Retention Time Calibration Standard Mixture	100
PCB-208	Ultra CB-682MN	Retention Time Calibration Standard Mixture	200
PCB-77	Ultra CB-682MN	Retention Time Calibration Standard Mixture	100
Total Nonachlorobiphenyls	Ultra CB-682MN	Retention Time Calibration Standard Mixture	200
DCB Decachlorobiphenyl	Ultra CB-681MN	Concentration Calibration Standard Mix	250
Total Dichlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50
Total Heptachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	150
Total Hexachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Monochlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50
Total Octachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	150
Total Pentachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Tetrachlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	100
Total Trichlorobiphenyls	Ultra CB-681MN	Concentration Calibration Standard Mix	50

7.2.2 Spiking Standards Prep for GC FID (DRO): The table below provides instruction in preparation of the various working spiking standards for use in this preparation method using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with the indicated solvent.

Working Spiking Standards Preparation					
Standard Name	**Vendor/ Cat #**	**Initial Conc. (ug/ml)**	**Vol. of Standard (ml)**	**Vol. of solvent ml (solvent)**	**Final Conc. (ug/ml)**
QAM-025 LCS Spike Diesel Fuel #2	Restek/31259	50000	4	100 ($MeCl_2$)	2000

Working Spiking Standards Preparation					
Standard Name	**Vendor/ Cat #**	**Initial Conc. (ug/ml)**	**Vol. of Standard (ml)**	**Vol. of solvent ml (solvent)**	**Final Conc. (ug/ml)**
DRO Surrogate -o-terphenyl	Supelco/47580U	10000	1.0	500 (Acetone)	20
QAM-025 MS/MSD Spike TPH Mix 3	Sigma/861394-U	1000	1.47	10 ($MeCl_2$)	147

7.2.3 Spiking Standards Prep for GC ECD(8081/8082):

7.2.3.1 All standard stock solutions are diluted to the working concentrations with hexane or acetone (as indicated) using Class A volumetric glassware. **Note:** septa on all surrogate and spike vials are to be replaced immediately after use. Additionally, all surrogate and spike vials are to be returned to the standards refrigerator immediately after use.

7.2.3.2 Pesticide Spiking Standard (LCS/MS/MSD): The Pesticide Mix BS/MS/MSD solution with 20 single component pesticides is prepared at a final concentration of 20 ug/ml by diluting 500 ul (0.50 ml) of the 2000 ppm Pest Spike stock solution (Supelco 4S7426-U, see tables in Section 7.2.1) to a 50 ml final volume with acetone. For spiking instructions refer to Section 10.

7.2.3.3 PCB Spiking Standard (LCS/MS/MSD): The PCB spiking solution (Supelco – 861274, see Tables 1 and 2) is a custom mix received from the vendor at its working concentration of 100 ug/ml in Acetone. For spiking instructions refer to Section 10.

7.2.3.4 Technical Chlordane Spiking Standard (LCS/MS/MSD): A technical Chlordane spiking solution is prepared at a final concentration of 100 ug/ml by diluting 1 ml of the 1000 ug/ml Technical Chlordane Spike solution (Supelco 48065-U, see tables in Section 7.2.1) to a 10 ml final volume using acetone. For spiking instructions refer to Section 10.

7.2.3.5 Toxaphene Spiking Standard (LCS/MS/MSD): A Toxaphene spiking solution is prepared at a final concentration of 100 ug/ml by diluting 1 ml of the 1000 ug/ml Toxaphene Spike solution (Supelco 48103, see tables in Section 7.2.1) to a final volume 10 ml. using acetone. For spiking instructions refer to Section 10.

7.2.3.6 Pesticide/PCB Surrogate Spiking Standard: The Pesticide/PCB surrogate spiking solution is a concentrated 10 ml solution of DCB/TCMX at a concentration of 10 ug/ml. (Supelco Catalog # 861275, see tables in Section 7.2.1). The concentrate is used without further dilution prior to the spiking of samples.

7.2.4 Spiking Standard for BNA: For use in spiking Soils samples prepared for BNA analysis by SW846 Method 8270. Prepare the spiking solution for MS/MSD/Blank Spike (LCS) as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol

Standard Name	Concentration	Volume of Standard added to final volume of 200ml (solvent)	Final Concentration
8270 List 1/ Std#1 MegaMix	500/1000/2000pmm	20 ml (methanol)	50/100/200 ppm
8270 List 1/ Std#5 N-Nitrosodiphenylamine	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/ Std#2 Amines	2000ppm	10 ml (methanol)	100 ppm
8270 List 1/ Std#3 Benzoic Acid	2000ppm	10 ml (methanol)	100 ppm
Custom SVO Mix	2000ppm	10 ml (methanol)	100 ppm
Bisphenol A	1000ppm	10ml (methanol)	50ppm
8270 List 1/ Std#8	2000ppm	20ul (methanol) 100ul(methanol)	Used neat for LVI Used neat for non-LVI
Aromatic Amines Custom Mix**	2000ppm	10 ml (methylene chloride)	100 ppm

*** As needed based on clients requirement.*

7.2.4.1 BNA Surrogate Standard Spiking Solution: For use in spiking all blanks, samples and associated QC prior to extraction. Prepare as detailed in the following table using the applicable standards listed in 7.2.1. Use volumetric glassware. Dilute to the volume marker with methanol.

Standard Name	Concentration	Volume of Standard added to final volume of 1000 ml methanol	Final Concentration
8270 Surrogate Standard	5000 ppm	20 ml	100 m

7.2.5 Spiking Standard for PCB Homologues (EPA 680): For use in spiking LCS and MS/MSD prior to extraction. Prepare by adding 1.0ml of the Retention Time Calibration Standard and 1.0ml of the Concentration Calibration Standard Mix to hexane in volumetric glassware and dilute to final volume of 100ml. 1.0mL of this solution is spiked into LCS/MS/MSD prior to extraction.

7.2.5.1 PCB Homologue Surrogate Standard Spiking Solution (EPA 680): (2.0ug/mL and 2.5ug/mL) – Add 3.125mL of $^{13}C_{12}$-Decachlorobiphenyl

and 1.0mL of Lindane13C6 and dilute to a final volume of 50mL in hexane in volumetric glassware. 1.0mL of this solution is spiked into all blanks, samples and QC items prior to extraction.

7.2.6 The preparation of all standards must be documented in a standard preparation logbook or in the TALS reagent module. Information such as standard supplier, lot number, original concentration, and a description of how standard was prepared are required along with a laboratory lot number, analyst's initials, date prepared and verification signature. Standards must be made every 6 months or sooner if signs of degradation appear.

8.0 Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Soils	Glass	4 oz	Cool 4 ± 2°C	14 Days	SW846 Method 8000

Samples are collected in amber jars with Teflon lined caps.
[1]All sample extracts must be analyzed within forty (40) days of extraction.

9.0 Quality Control

9.1. Sample QC – The following quality control samples are prepared with each batch of samples.

QC Analysis	Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS/LCSD)[1]	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria
Method Blank (MB)	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria
MS\MSD[2] (Client specified)	1 in 20 or fewer samples	See applicable analytical SOPs	See applicable analytical SOPs for control criteria
Surrogates[3]	All Samples including QC	See applicable analytical SOPs	See applicable analytical SOPs for control criteria

[1] LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract except for NJDEP EPH 10/09 where LCS/LCSD is required.
[2] The sample selections for MS/MSD are randomly selected, unless specifically requested by a client. For NJDEP EPH 10/09, only MS is required (MSD for NJDEP EPH 10/09 is by client request only).
[3] Analytical and QC samples (MB, LCS, MS/MSD)
[4] Statistical control limits are updated annually and are updated into LIMS.

9.2. Instrument QC

N/A

10.0 Procedure

10.1. Sample Preparation

10.1.1 Decant and discard any water layer on a sediment sample. Discard any foreign objects such as rocks, leaves and sticks. Mix sample thoroughly with wooden tongue depressor prior to subsampling

10.1.2 The initial amount and treatment of soil samples is method dependent:

- For DRO (8015): 15 grams of soil is weighed into a 75mL Teflon microwave vessel (the weight is recorded to two significant figures). Where practical samples should be air dried and ground prior to extraction. Drying should be performed in a hood to avoid contamination.
- For 8270: 15 grams of soil is weighed into an aluminum weighing dish (the weight is recorded to two significant figures). 15 ml of acetone is added to the dish and the soil/acetone mixture is stirred well with a wooden tongue depressor to create a slurry. The soil/acetone slurry is transferred to a 75 ml Teflon microwave vessel. The dish is then rinsed with 15 ml of methylene chloride which is also added to the microwave vessel.
- For 8081/8082: 15 grams of Soil is weighed into an aluminum dish. 15 ml of Acetone is added to the dish and the soil / acetone mixture is stirred well with a wooden tongue depressor to create a slurry. Then, the mixture is transferred into a 75ml Teflon vessel. The dish is then rinsed with 15 ml of Hexane which is also added to the microwave vessel.
- For EPA 680: 30 grams of soil is weighed into a 75mL Teflon microwave vessel (the weight is recorded to two significant figures).

10.1.3 For laboratory blanks and control spikes, an equal portion of clean sand is prepped along with samples.

10.1.4 All samples, blanks, and spikes are fortified with an appropriate amount of the applicable method surrogates. All matrix spikes and lab control spikes are fortified with an appropriate amount of the applicable method spiking solution. See Attachment 1 for general spiking instructions. (Refer to Section 7.2 for information on preparation of spiking solutions):

- PCBs (8082): add 50 ul of the PCB Spike solution to each LCS and designated MS/MSD;

- Pesticides (total, 8081): 100 ul of the Pest Spike solution to each LCS and designated MS/MSD;
- Technical Chlordane: 100 ul of the Technical Chlordane Spike mix to each LCS and designated MS/MDS;
- Toxaphene: 100 ul of the Toxaphene Spike mix to each LCS and designated MS/MSD;
- BNA (8270): 500ul of spike/Surrogate solution to each LCS and designated MS/MSD);
- PCB Homologues (EPA 680): 1.0 ml each of Spiking solution (see Sec.7.2.5) and Surrogate solution (see Sec. 7.2.5.1) to each LCS and designated MS/MSD.

  NOTE: When spiking the samples, it is critical to remove all bubbles from the syringe.

10.1.5 Add the following solvent to each sample vessel depending upon the fraction (**note**: for Pesticides and PCBs by methods 8081 and 8082 the solvent addition is addressed in Section 10.1.2 above)

- 30mL of methylene chloride for DRO (8015);
- 30 mL of Methylene Chloride/Acetone blend (50:50) for BNA (8270)

30 mL of Hexane for PCB homologues by EPA 680

10.1.6 A stopper is placed over the opening along with the vessel cap. The vessel is then tightened using the MARS capping wrench. This capping wrench tightens the cap to the appropriate torque.

10.1.7 The sample vessels are placed into the 40-position carousel. Each position has an insulator sleeve that the vessel slides into – slide the vessel into the appropriate sleeve – and press firmly down to ensure a complete connection.

10.1.8 Place the carousel (with samples and vessels) into the microwave unit.

Start Run: (load method – using key pad on face of unit)
Press: Home
Press Select: load method
Press Select: from user directory
Press Select: 3546 Microwave – Xpress method
Press Start.

Microwave Operating Parameters				
Power Max	**Power %**	**Oven Ramp**	**Degrees Celsius**	**Hold Time**
1200W	100	10.0 minutes	110	10.0 minutes
1600W[1]	100	20.0 minutes	115	10.0 minutes

[1] For pesticide/PCB and BNA samples only

10.1.9 A 5-minute cool down will close out the run.

10.1.10 Remove vessels from carousel and allow cooling (either at room temperature or in a cold water bath).

10.1.11 When cool down is complete, remove carousel from microwave.

10.1.12 Pre-wash a funnel filled with sodium sulfate with three successive washings of 30 ml acetone. Pour the extract carefully through the sodium sulfate funnel (to remove any moisture) into the 200mL concentrator tube. Rinse the 75mL vessel with 10 mL methylene chloride three times to ensure quantitative transfer.

10.1.13 Set the water temperature in the concentrator to 35°C and adjust the pressure to around 5psi of nitrogen.

10.1.14 Place the tube into the apparatus and slowly raise the pressure to 20psi. If splashing occurs, start with a lower pressure and raise it when the solvent gets lower.

10.1.15 Concentrate to just under the 1mL mark on the nipple of the tube. Remove from the bath and transfer to an auto sampler vial with a glass Pasteur pipette..

10.1.16 Add a few drops of clean methylene chloride to the tube, rinsing the narrow part and add to the vial to adjust to 1mL or other method specific final volume. If solvent exchange to hexane is required, add approximately 30mL (mix well) and return to concentrator set at 35°C. See Attachment 2.

10.1.17 Total concentration time should be approximately 20 minutes for methylene chloride. If a sample takes a longer than usual amount of time to concentrate or if the extract becomes viscous the final volume should be adjusted to a larger volume and documented.

10.1.18 The extract is now ready for the appropriate clean up and or analysis.

10.2. Calibration

10.2.1 The balance calibration is checked daily prior to use. Refer to Eurofins Edison SOPs ED-GEN-010, *Calibration of Analytical Balances*, current revision and ED-GEN-006, *Standard Operating Procedure for Preventive Maintenance and Calibration Procedures for All Analytical Instruments and Ancillary Equipment*, current revision.

11.0 Calculations / Data Reduction

N/A

12.0 Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For demonstration of capability procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0 Pollution Control

13.1. It is Eurofins's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0 Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are

disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins Edison SOPs No. ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision).* The following waste streams are produced when this method is carried out:

- *Mixed Solvent Waste*: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- *Auto sampler vials and expired standards*: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

15.0 References / Cross-References

15.1 United States Environmental Protection Agency, “Method SW3546, Microwave Extraction”, Test Methods for Evaluating Solid Wastes, SW846 Laboratory Manual, Physical/Chemical Methods, February 2007.

15.2 United States Environmental Protection Agency, Method SW8015D, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 4, June 2003.

15.3 United States Environmental Protection Agency, “Method 8081B, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, February 2007.

15.4 United States Environmental Protection Agency, “Method 8082A, Organochlorine Pesticide by Gas Chromatography”, Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 1, February 2007.

15.5 United States Environmental Protection Agency, “Method SW8270E, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry”, Test Methods for Evaluating Solid Wastes, SW846 Update VI, Revision 6, June 2018.

15.6 Eurofins Edison SOP No. ED-GCS-020, *Preparation of Water Samples by SW846 Methods 3510C (Separatory Funnel) and Analysis of Diesel Range Organics in Soil and Water Samples by SW846 Method 8015D (GC/FID)*current revision.

15.7 Eurofins Edison SOP No. ED-GCS-016, *Analysis of Organochlorine Pesticides by Gas Chromatograph, SW846 Method 8081B*, current revision.

15.8 Eurofins Edison SOP No. ED-GCS-017, *Analysis of Polychlorinated Biphenyls by Gas Chromatograph, SW846 Method 8082A*, current revision

15.9 Eurofins Edison SOP No. ED-MSS-010, *Determination of PCBs in Water and Soil/Sediment by GC/MS, EPA Method 680,* current revision.

15.10 Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.

15.11 Eurofins Environmental Health and Safety Manual, CW-E-M-001.

15.12 Eurofins Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing& Approval*, current revision.

15.13 Eurofins Edison SOP No. ED-GEN-023, *Bulk Solvent Testing and Approval*, current revision.

15.14 Eurofins Edison SOP No. ED-GEN-013, *Glassware Cleaning,* current revision

15.15 Eurofins Edison SOP No. ED-GEN-022, *Training,* current revision

15.16 Eurofins Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures,* current revision.

15.17 Eurofins Edison SOP No. ED-GEN-006, *Standard Operating Procedure for Preventive Maintenance and Calibration Procedures for All Analytical Instruments and Ancillary Equipment,* current revision.

15.18 Eurofins Edison SOP No. ED-GEN-010, *Calibration of Analytical Balances,* current revision.

15.19 Eurofins Edison SOP No. ED-GEN-007, *Subsampling*, current revision.

15.20 Operation Manuals for the Zymark Turbo Vap II Concentrator.

15.21 Operating manual for the MARS 5 Microwave unit.

16.0 Method Modifications:

N/A

17.0 Attachments

Attachment 1: Spiking information
Attachment 2: Specific Extraction Conditions

18.0 Revision History

Rev 12, July 2, 2024

- Updated to reflect Eurofins branding and formatting.
- Section 1.1: updated method/SOP references as needed.
- Throughout document: updated Lab Quality Manual section references as needed.
- Section 14 (References): updated to reflect current documents.
- Section 7.2.1: updated DRO spiking solution.
- Section 10.1.2: updated procedure for 8081/8082 such that acetone: hexane is now the extraction solvent (methylene chloride eliminated).

Rev 11, March 12, 2018

- Updated Section 10.1 to revise procedures for the microwave extraction of solids for subsequent analysis by EPA methods 8081, 8082 and 8270.
- Updated to remove all references to addition of sodium sulfate to samples prior to microwave extraction.
- Fixed section numbering and formatting errors.

Rev 10, May 16, 2017

- Updated throughout to add procedures for microwave extractions of solids for subsequent analysis by EPA Method 680 (PCBs by GC/MS).

Rev 9, Dec 15, 2016

- Updated throughout to remove procedures for microwave extraction of solids for NJ EPH method. Those procedures are fully covered in Eurofins Edison SOP ED-GCS-012, Preparation and Analysis of Extractable Petroleum Hydrocarbons (EPH) in Solid and Water Samples using NJDEP EPH Method 10/08, August 2010 (Rev. 3): Analysis of Extractable Petroleum Hydrocarbon Compounds (EPH) in Aqueous and Soil/Sediment/Sludge Matrices (current revision)
- Added following text to Section 1.2: The microwave prep for NJDEP Extractable Petroleum Hydrocarbons (EPH) (10/08), current revision, is addressed separately in Eurofins Edison SOP ED-GCS-012 (current revision).
- All references to NJDEP EPH methodology removed.

Rev 8, Jan 22, 2015

- Updated to include procedures for microwave extraction of solids for analysis my SW846 Methods 8270C and 8270D (Semivolatile Organic Compounds by Gas Chromatography/ Mass Spectrometry (GC/MS). Specifically
- Added 8270 method references and SOP numbers to Sections 1.1 and 15.0
- Added a table to Section 7.2.1 detailing the BNA standard mixes.
- Added new Section 7.2.4 detailing the preparation of BNA spiking standards
- Added Section 7.2.4.1 detailing the preparation of BNA surrogate spiking standards.
- Added details of 8270 spiking and extraction to Section 10.0 as appropriate.
- Added BNA Spiking details to Attachments 1 and 2. COAs for BNA spiking not included in SOP but are on-file in the department.

Rev 7, June 14, 2013

- Section 1.1: updated the DRO method references to include SW8015C and SW8015D. Also update the SOP references for the DRO analytical methods.
- Section 7.2.1: updated stock standards table to include SW8015C and 8015D. Revised source for o-Terphenyl stock. Removed TPH Mix 1 from list. Removed 8015x DRO from the list of methods for which TPH Mix 3 is used. Added 8015x DRO to the list of methods for which Diesel Fuel #2 is used.
- Section 7.2.2.1: Revised DRO surrogate spike source and prep instructions. Revised DRO and QAM-025 spiking standard source and prep instructions.
- Section 15.0: References: added references for methods SW8015C and 8015D.
- Attachment 1: updated spiking information for DRO.

Rev 6, October 10, 2012

- Throughout document:: Revised LQM reference sections to reflect the most current revision
- Throughout document: updated as detailed below to include prep instructions for solid samples undergoing analysis by SW846 8081A/B and 8082/A.
- Section 1.1: added references to the 8081/8082 SOPs: Eurofins Edison SOPs No.ED-GCS-003, ED-GCS-004, ED-GCS-016 and ED-GCS-017.
- Section 6.2: added weigh boats and wooden tongue depressors.
- Section 7.2.1: added acetone as a diluent and added tables detailing the Pestcide and PCB standards.
- Section 7.2.2: added “GCFID (DRO/EPH/QAM)” to section header.
- Section 7.2.3: added instructions for prep of Pest/PCB spiking standards.
- Section10.1.4: Added Pest/PCB spiking instructions.

- Section 10.1.5: added instructions for addition of extraction solvent (Methylene Chloride:Acetone for Pest/PCB).
- Section 10.1.8: updated table to include Microwave Parameters for Pest/PCB prep.
- Section 10.1.12: added a methylene chloride rinse.
- Section 15: added method and SOP references for SW846 8081A/8081B and SW846 8082/8082A.
- Attachments: updated to include Pest/PCB information.

Rev 5, August 26, 2011

- Section .6.2: added vendor and part number details for silica gel columns (Phenomenex Part No:8L-S012-JCH)
- Section 10.1.2: Added details for the pre-washing of sodium sulfate funnels with three successive 30 ml volumes of acetone. Added details for the rinsing of the 75 ml vessel with three successive volumes of methylene chloride.

Rev 4, April 21, 2010

- Section 7.2.1: Corrected the footnotes in the tables 'Stock Standards – Aliphatics (Method NJDEP EPH 10/08)' and 'Stock Standards-Aromatics (Method NDJEP EPH 10.08)'.
- Attachment 3: Added this attachment which consists of select certificates of analysis for Method NJDEP EPH 10/08.
- Section 7.2.2.1: Table 'Working Spiking Standards Preparation'" replaced asterisks (*) with dashes (-) in order to clarify that these are simply bullets rather than referencing footnotes.

Rev 3, April 19, 2010

- Section 7.2.2.1: added procedure for preparation of a 500 ppm Aliphatic Neat Spike Mix.
- Section 7.2.2: Spiking Standards Prep: revised the procedure for prep of the EPH Aliphatic Spike in the 'Working Spiking Standards Preparation' table as follows:
    - Replaced 'MA EPH-n-Hydrocarbons (Absolute/91488) with the 'Florida TRPH Standard (Restek/31266)'
    - Included used of the intermediate 500 Aliphatic Neat Spike Mix.

Rev 2, February 9, 2009

- Section 3.0, Definitions – updated to reflect current location of definitions (Appendix 2 of Lab Quality Manual
- Section 5.2, Primary Materials Used – added 'hexane' to table (used in the NJDEP EPH method).
- Section 7.2.1: added stock standards associated with NJDEP EPH 10/08 method.
- Section 7.2.2: Spiking Standards Prep: updated 'Working Spike Standards' table to include revised NJDEP EPH 10/09 spiking standards; revised other as required.
- Section 9.1, Sample QC: updated with revise NJDEP EPH 10/09 QC requirements.

- Section 10.1, Sample Preparation: revised initial sample weight to 15 grams.
- Section 10.1.8: added Microwave Operating Parameters for the NJDEP EPH 10/09 method.
- Section 10.2.1: changed the specifications of the silica column.
- Section 10.2.5: changed the volume of the hexane rinsate.
- Section 10.2.7: changed the volume of the methylene chloride rinsate.
- Section 10.2.11: Added text describing procedures for NJDEP EPH 10/09 Demonstration of Fractionation Capability.
- Section 14.1 and Section 15.0 : Removed reference to Eurofins Edison SOP No. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision)* (SOP is now retired).
- Section 15.0: updated references to NJDEP EPH method as well as Eurofins Edison SOP for NJDEP EPH analysis.
- Section 17.0, Attachments: corrected descriptions of attachments.

Rev 1, June 29, 2009

- Section 1.0, Scope and Application – corrected typo in the temperature range 100 – 115°C
- Section 5.2, Primary Materials Used – removed ‘sodium hydroxide’ and ‘sulfuric acid’ from table as they are not used in this method.
- Section 6.1, Instrumentation – added Visiprep DL column holder to the instrumentation list
- Section 6.2, Supplies – added 5 g silica gel column (20 ml capacity)
- Sections 7.2.1, 7.2.2, Attachment 1 and Attachment 2: corrected a typo in the abbreviation for Methylene Chloride ($MeCl_2$).
- Section 10.1.1.2 - sentence correction to show actual process and typo error correction. Phrase ‘may also’ changed to ‘shall’.
- Section 10.1.12 – replaced the word ‘salt’ with ‘Sodium Sulfate’ to show actual reagent being used.
- Section 10.1.17 – typo error correction to ’20 minutes’
- Section 10.2.5 – Added the following text to the end of the section: “The amount on hexane will need to be adjusted with every lot of silica columns. The Hexane amount should never exceed 20 mls.”
- Corrected carbon ranges for the reference DRO SOPs throughout the document.

Rev 0, March 27, 2009: New

Attachment 1

Spiking Information – Microwave Extraction

Method	Surrogate Amount (all samples and QC)	Spike Amount (LCS/MS/ MSD)	Final volume	Initial Solvent	Final Solvent
DRO (8015)	1 mL	1 mL	1 mL	$MeCl_2$	$MeCl_2$
Pest/PCB (8081/8082)	0.05 mL	0.1/0.05 mL	10 mL	MeCl2/Acetone (50:50)	Hexane Exchange from MeCl2/Acetone
BNA (8270)	500uL	500uL	1 mL	MeCl2/Acetone (50:50)	$MeCl_2$
PCB Homologues (EPA 680)	1.0	1.0	1 mL	Hexane	Hexane

Attachment 2

Specific Extraction Conditions

Method	Initial extraction pH	Secondary extraction pH	Final Solvent	Volume of extract required for cleanup(mL)	Final extract volume for analysis (mL)
DRO (8015)	as received	NA	$MeCL_2$	NA	1.0
Pest/PCB (8081/8082)	as received	NA	Hexane	3	10.0
BNA (8270)	as received	NA	$MeCL_2$	NA	1.0
PCBs (EPA 680)	as received	NA	Hexane	NA	1.0

Title: SW846 Method SW8081B, Analysis of Organochlorine Pesticides by Gas Chromatography

Approvals (Signature/Date):

Catalina Dalangin SVOA GC Manager	06/12/2024 Date	Dan Helfrich Health & Safety Manager	06/12/2024 Date
Carl Armbruster Quality Assurance Manager	06/12/2024 Date	Mark Acierno Laboratory Director	06/12/2024 Date
Sylvanus Klusey Operations Manager - Organics	06/12/2024 Date		

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

Method 8081B is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices, using dual fused-silica, open-tubular, capillary columns with electron capture detectors (ECD). The list of analytes and their corresponding reporting limits are as follows:

Parameter		Soil	Water	Leachate
	CAS Registry No.	Reporting Limits (ug/Kg)	Reporting Limits (ug/L)	Reporting Limits (mg/L)
Aldrin	309-00-2	6.7	0.02	---
Alpha-BHC	319-84-6	2.0	0.02	---
Beta-BHC	319-85-7	2.0	0.02	---
Delta-BHC	319-86-8	2.0	0.02	---
Gamma-BHC (Lindane)	58-89-9	6.7	0.02	0.00050
Chlordane	57-74-9	67	0.50	0.0050
4,4' –DDD	72-54-8	6.7	0.02	---
4,4' –DDE	72-55-9	6.7	0.02	---
4.4' –DDT	50-29-3	6.7	0.02	---
Dieldrin	60-57-1	2.0	0.02	---
Endosulfan I	959-98-8	6.7	0.02	---
Endosulfan II	33213-65-9	6.7	0.02	---
Endosulfan sulfate	1031-07-8	6.7	0.02	---
Endrin	72-20-8	6.7	0.02	0.00050
Endrin aldehyde	7421-93-4	6.7	0.02	---
Endrin ketone	53494-70-5	6.7	0.02	---
Heptachlor	76-44-8	6.7	0.02	0.00050
Heptachlor epoxide	1024-57-3	6.7	0.02	0.00050
Methoxychlor	72-42-5	6.7	0.02	0.00050
Toxaphene	8001-35-2	67	.50	0.0050
Gamma- Chlordane	5103-74-2	6.7	0.02	---
Alpha-Chlordane	5103-71-9	6.7	0.02	---
Mirex	2385-85-5	6.7	0.010	---

The most current MDLs and RLs for this method can be found in the active Eurofins LIMS (TALS) SW846 8081B Method Limit Group (MLG) database.

1.2 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and Section 19 (*Test Methods and Method Validation)* of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. Samples undergo a preparation step prior to analysis by SW846 Method 8081B. A measured volume or weight of sample is extracted using the appropriate matrix-specific sample extraction technique. (Reference the applicable Organic Sample Prep SOPs listed below). The effective final volume is usually between 5 and 20 ml in hexane.

2.1.1. Aqueous samples are extracted using either SW846 Method 3510C (SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel*) or SW846 Method 3511 (SOP No. ED-ORP-051: *Extraction of Semivolatile Organic Compounds in Water by Microextraction using SW846 Method 3511*)

2.1.2. Solid samples are extracted using SW846 Method 3550B: Sonication (*SOP No. ED-ORP-018: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction*) or SW846 Method 3546: Microwave (SOP No. ED-ORP-044: *Procedure for the Microwave Extraction of Solids,SW846 3546).*

2.1.3. Organic liquids are prepared using SW846 Method 3580A (*SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs*).

2.1.4. Extract cleanup steps are employed depending on the nature of the matrix interferences. Suggested cleanups include SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*) and SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*).

2.2. After cleanup, a small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds which were recovered during the extraction step by boiling point. The effluent of the gas chromatograph is interfaced to an Electron Capture Detector (ECD) which is used to detect the compounds eluting from the GC.. Quantitation is accomplished by comparing the area response of each target analyte relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Method 8081B.

2.3. For pesticide analysis a system performance check (DDT/Endrin breakdown) and a calibration verification standard must be run prior to analysis. Failure of either will generally indicate the need for injection port/column maintenance and/or recalibration.

2.4. Samples are analyzed after all the necessary checks have been performed. Samples analyzed for pesticides require an additional post analysis Quant Report to be printed and attached to the chromatographic report.

2.5. All samples are then manually reviewed. Secondary column confirmation of target compounds and quantitation are conducted by the analyst as required.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.

4.1.1. Interferences from phthalate esters can be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.

4.2. The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. If sulfur is encountered, employ the sulfur removal procedures detailed in SW846 Method 3660B (*SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts*). Note that the recover of Endrin Aldehyde is adversely affected by the TBA cleanup procedure detailed in this method. Accordingly, this compound must be determined prior to sulfur cleanup.

4.3. Co-eluting chlorophenols are eliminated by using SW846 Method 3620B (*SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*).

4.3.1. Check Florisil prior to use to assure quantitative recovery of targeted analytes. Duplicate checks are required for each new lot or every three hundred samples whichever is more frequent.

4.3.2. Check Florisil by spiking 1ml of the Pest Std Mix A midpoint (Supelco Catalog No. 47977) and 0.5 ml of trichlorophenol (Absolute Standardsl Catalog No. 20024) onto the cartridge and concentrating to final volume of 1 ml. Inject 1 ul onto a capillary column, conducting the elution and analyzing the extract. Recovery is acceptable if all pesticides are recovered at 80 - 110% and the recovery of trichlorophenol is <5% and co-eluting interfering peaks are absent from the extract.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm-TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methanol	Flammable Poison Irritant	200 ppm-TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm-TWA 125 ppm-STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation:

6.1.1. Gas Chromatograph: The system used is an HP and an Agilent Technologies (Avondale, PA) model 5890/6890 Gas Chromatograph (GC). Each GC is equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine pesticides. All operations are as automated as possible with the equipment utilized.

6.1.2. Injection system: Sample injection is accomplished by a single auto injector. The auto injector is serviced by a robot arm that shuttles samples between the sample tray and the injector turret.

6.1.2.1. The samples are injected into a split/splitless injection port equipped with electronic pressure control (EPC). The injection port is normally operated in splitless mode during injection. The EPC is operated in the ramp pressure mode.

6.1.2.2. Liners: The injection port is each fitted with replaceable, heavy-walled siltek-coated glass double gooseneck liner. The liner contains a plug of silanized glass wool approximately 1 cm in length. The glass wool is positioned in the liner between the double gooseneck. The liner is replaced on a regular maintenance schedule.

6.1.2.3. Oven and Columns: Temperature programmable gas chromatograph ovens are required, capable of integrated temperature control between 35^{O}C and 350^{O}C.

6.1.2.3.1. Two dissimilar columns are used for analysis. A Restek RtxCLPesticides, 30m x 0.53mm ID x 0.5um film thickness column is used for sample quantitation. The secondary confirmation column is a Restek RtxCLPesticides II, 30m x 0.53mm ID x 0.42um film thickness column.

6.1.2.4. Detectors: Sample detection is by electron capture. The GC is equipped with dual Electron Capture Detectors (ECD), one for each column.

6.1.2.4.1. Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron plasma. This is in addition to the gas exiting the column. The make-up gas (P-5 & Nitrogen) is fed from a supply other than the injection port.

7. Reagents and Standards

7.1. Reagents

7.1.1. Gases: Hydrogen is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. P-5 and Nitrogen) is used as make-up gas. It is introduced to the GC via the make-up gas adapter at the end of the capillary column. Hydrogen is supplied via a Parker Balston H2 Generator. Nitrogen & P-5 is supplied by Air Gas

7.1.1.1. The gas streams are polished using three traps or filters before introduction to the GC The traps are as follows:

7.1.1.1.1. Hydrocarbon trap
7.1.1.1.2. H_2O trap
7.1.1.1.3. O_2 scrubber

7.1.1.2. Both the moisture trap and the Oxygen scrubber are of the indicating type. They require either replacement or reconditioning upon color change of the active agents. Refer to the instructions for the individual traps to determine if it is still active. The hydrocarbon trap is a simple activated carbon trap. With high quality gas, it should last for an extended period of time (1-yr. minimum).

7.1.2. Solvents used in the extraction and cleanup procedures include n-hexane, methylene chloride, and acetone that are exchanged to n-hexane prior to analysis.

7.1.3. Hexane is required in this procedure. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis as detailed in Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

7.2. Standards

7.2.1. Standards are purchased as concentrated solutions (see Section 7.2.2).

NOTE: Independent sources are used for quantitation standards and spiking standards

7.2.1.1. Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent as described in Section 7.2.2.1.

7.2.2. Standard mixes and sources *.

Standard Name	Source
Organochlorine Pesticide Mix AB #3	Restek Catalog No. 32415
Organochlorine Pesticide Mix AB #3.sec	Restek Catalog No. 32415.sec (second source)
Pesticide Surrogate Mix	Restek Catalog No. 32000
Endrin/DDT	Supelco Catalog No 48282
Chlordane	Restek Catalog No. 32021
Toxaphene	Restek Catalog No. 32005
Toxaphene (different pattern)	Restek Catalog No. 32071
Mirex	Accustandard Cat. No. P-066S-10X
1-Bromo-2-nitrobenzene (internal standard)	Restek Cat. No. 32279

*Suppliers with equivalent standards may be substituted..

The components of each standard mix are as follows (note: 'sec' indicates second source):

Parameter	Supplier	Catalog No.	Concentration of Standard (ug/ml)
Aldrin	Restek	32415 & 32415.sec	2000
Alpha-BHC	Restek	32415 & 32415.sec	2000
Beta-BHC	Restek	32415 & 32415.sec	2000
Delta-BHC	Restek	32415 & 32415.sec	2000
Gamma-BHC (Lindane)	Restek	32415 & 32415.sec	2000
Alpha -Chlordane	Restek	32415 & 32415.sec	2000
Gamma -Chlordane	Restek	32415 & 32415.sec	2000
Technical Chlordane	Restek	32021	1000
4,4' –DDD	Restek	32415 & 32415.sec	2000
4,4' –DDE	Restek	32415 & 32415.sec	2000
4.4' –DDT	Restek	32415 & 32415.sec	2000
Dieldrin	Restek	32415 & 32415.sec	2000
Endosulfan I	Restek	32415 & 32415.sec	2000
Endosulfan II	Restek	32415 & 32415.sec	2000
Endosulfan sulfate	Restek	32415 & 32415.sec	2000
Endrin	Restek	32415 & 32415.sec	2000
Endrin aldehyde	Restek	32415 & 32415.sec	2000
Endrin ketone	Restek	32415 & 32415.sec	2000
Heptachlor	Restek	32415 & 32415.sec	2000
Heptachlor epoxide	Restek	32415 & 32415.sec	2000
Methoxychlor	Restek	32415 & 32415.sec	2000
Toxaphene	Restek	32005	1000
Toxaphene	Restek	32071	5000
Mirex	Accustandard	861428-U	1000
4,4-DDT	Supelco	18282	500
Endrin	Supelco	48282	500
Decachlorobiphenyl (DCB)	Restek	32000	200
Tetrachloro-m-xylene (TCmX)	Restek	32000	200

Parameter	Supplier	Catalog No.	Concentration of Standard (ug/ml)
1-Bromo-2-nitrobenzene (internal standard)	Restek	32279	1000

7.2.2.1. Standards Preparation

7.2.2.1.1. Calibration Mix (Organochlorine Pesticide Mix)

The 5 point calibration standards are prepared as detailed in the following table using volumetric glassware and hexane as the diluent:

Initial Calibration Standards Prep (Organochlorine Pesticide Mix)					
Stock Std	**2.5 ppb**	**50 ppb**	**100 ppb**	**250 ppb**	**500 ppb**
Organochlorine Mix AB#3 *(2000 ug/ml)* *Restek (Cat No..32415)in Hexane:Toluene*	Volume brought up to 200 ml hexane: *0.25 ul*	Volume brought up to 200 ml hexane: *5.0 ul*	Volume brought up to 500 ml hexane: *25 ul*	Volume brought up to 200 ml hexane: *25 ul*	Volume brought up to 200 ml hexane: *50 ul*
Initial Calibration Standards Prep (TCMX/DCB Surrogate Mix)					
Stock Std	**25 ppb***	**50 ppb**	**100 ppb**	**150 ppb**	**200 ppb**
Tetrachloro-m-xylene/ Decachlorobiphenyl Surrogates Mix (200 ug/ml) *Restek (Cat, No, 32000) in Acetone*	Volume brought up to 200 ml hexane: *6.25 ul*	Volume brought up to 200 ml hexane: *50 ul*	Volume brought up to 500 ml hexane: *250 ul*	Volume brought up to 200 ml hexane: *150 ul*	Volume brought up to 200 ml hexane: *200 ul*
Initial Calibration Standards Prep (Mirex)					
Stock Std	**2.5 ppb**	**50 ppb**	**100 ppb**	**250 ppb**	**500 ppb**
Mirex *(1000 ug/ml)* *Accustandard (Cat. No. P-066S-10x) in Methanol*	20x dilution of 50 ppb	10x dilution of 500 ppb	5x dilution of 500 ppb	2x dilution of 500 ppb	Volume brought up to 100 ml hexane: *50 ul*

Note: 20 ul of 5 ug/ml Internal Standard solution is added to all calibration standards prior to analysis.

7.2.2.2. **Pesticide Surrogate Spike Mix (10 ug/ml**) : The Pesticide 10ug/ml surrogate spiking solution is prepared by diluting 10 ml of 200 ug/ml of the Pesticide Surrogate Mix (Restek-32000) in to 200 ml of Acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.3. **Pesticide Surrogate Spike Mix (2 ug/ml) for the reduced volume extraction option:** prepare a 2 ug/ml spiking solution by diluting 10 ml of the 10 ug/ml stock standard described in

7.2.2.2 to a final volume of 50ml in acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.4. **Pesticide Internal Standard Spike Mix (5 ug/ml):** The Pesticide 5 ug/ml internal standard spike mix is prepared by dilution 1 ml of 1000 ug/ml of the 1-Bromo-2-Nitrobenzene standard (Restek 32279) in to 200 ml of Hexane. 20 ul of this solution is added to all standards, QC samples and field sample extracts prior to analysis.

7.2.2.5. **Pesticide Spiking Standard (20 ug/ml):** The Pesticide Spiking Mix containing the single component pesticides is prepared at a final concentration of 20 ug/ml by diluting 500 ul of the Organochlorine Pesticide Mix AB#3 (Restek 32415 to a 50 ml final volume with acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.6. **Pesticide Spiking Standard (4 ug/ml) for the reduced volume extraction option**: prepare a 4 ug/ml spiking solution by diluting 10 ml of the 20ug/ml standard prepared in Section 7.2.2.4 above to a final volume of 50ml in acetone. For spiking instructions refer to the applicable prep SOP.

7.2.2.7. **System Performance Solution (Breakdown Check)** 3,3-DDT and Endrin at 0.25 ug/ml): The breakdown check is prepared by taking 250 ul of 500 ug/ml DDT/Endrin Mix (Supelco Catalog No 48282) and bringing it up to a volume of 500 ml with hexane

7.2.2.8. **Technical Chlordane Calibration Solution** (1.0 ug/ml solution w/ surrogates TCMX & DCB at 0.10 ug/ml): 100 ul of 1000 ug/ml Technical Chlordane Standard (Restek Catalog No 32021) and 50 ul of 200 ug/ml Surrogate Mix (Restek Catalog No. 32000). Dilute to 100 ml in Hexane. ***NOTE:*** ICAL consists of single midpoint calibration standard for multiple responders Technical Chlordane and Toxaphene.

7.2.2.9. **Toxaphene Calibration Solution** (1.0 ug/ml solution w/surrogates TCMX & DCB at 0.1 ug/ml) : 20 ul of 5000 ug/ml Toxaphene stock (Restek Catalog No 32071) and 50 ul of 200 ug/ml Surrogate Mix (Restek Catalog No. 32000). Dilute to 100 ml in Hexane. ***NOTE:*** ICAL consists of single midpoint calibration standard for multiple responders Technical Chlordane and Toxaphene.

7.2.2.10. **Initial Calibration Verification (ICV)** Preparation: follow the instructions above for midpoint standards substituting the second source standard (catalog no. suffix = sec) for the primary standards.

8. Sample Collection, Preservation, Shipment and Storage

8.1. Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

8.2. Samples from chlorinated water sources must be treated with sodium thiosulfate (0.008% solution) at the time of collection to remove chlorine. NOTE: containers pre-preserved with sodium thiosulfate must be requested in bottle orders for samples from chlorinated water sources.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 250 ml, 40 ml or 60 ml	250 ml (3510C prep)	Cool 4 ± 2ºC	7 days to extraction; Analyze within 40 days of extraction	SW846
Soils	Glass, 2 or 4 oz	100 g	Cool 4 ± 2ºC	14 days to extraction; Analyze within 40 days of extraction	SW846

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS)[1]	1 in 20 or fewer samples	Statistical Limits [4]
Matrix Spike (MS)[2]	1 in 20 or fewer samples	Statistical Limits [4]
MS Duplicate (MSD)[2]	1 in 20 or fewer samples	Statistical Limits [4]
Surrogates	every sample[3]	Statistical Limits [4]
Internal Standard [1]	Every sample[3]	Response within -50% to +100% of most recent cal standard

LCS Duplicate (LCSD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.

[2] The sample selection for MS/MSD are randomly selected, unless specifically requested by a client….predetermined by the extraction lab.

[3] Analytical and QC samples (MB, LCS, MS/MSD)

[4] Statistical control limits are updated annually and are updated into LIMS.

9.1.1. Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.

9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The recoveries of the LCS must fall within lab generated acceptance criteria (refer to the current TALS Method Limit Group database). If the LCS recovery results are outside of these limits, the extract is reanalyzed. If LCS recoveries are still outside of QC limits after extract reanalysis but recoveries for the Matrix Spike/ Matrix spike Duplicate (MS/MSD) are within QC limits, the data is reported and a Non Conformance Memo (NCM) is written.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to the current TALS Method Limit Group database). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated. If the LCS recoveries meet criteria the data is reported and a Non-Conformance Memo (NCM) is written.

9.1.4. Surrogate Standards: All samples, blanks and QC samples are spiked with a 2 component surrogate standard mix containing TCMX & DCB (see Section 7.2). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to the current TALS Method Limit Group database*)*. Minimum requirements for surrogate evaluation:

- Both surrogates must have reportable results that meet the acceptance criteria;
- Reported surrogates must be from a column with a passing CCV;
- At least one surrogate must pass on any column from which target analytes are identified and reported.

If both TCMX and DCB recovery are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as “estimated concentration”.

9.1.5. Internal Standard: The internal standard (1-bromo-2-nitrobenzene) must elute within 30 seconds of and have an area response of 50 to 100% as compared to the most recent preceding calibration standard.

9.1.6. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification: The LLOQ is the lowest concentration at which the laboratory has

demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

9.2. Instrument QC

9.2.1. GC System Performance Check

9.2.1.1. **Endrin/4,4'-DDT Breakdown:** Prior to performing any standards or sample analysis, a daily check is made on the chromatographic performance of the system. This performance check is made by injecting a standard of Endrin and DDT, each at a 250-ppb level (see Section 7.2), and calculating the percentage breakdown for each compound.

9.2.1.2. Ideally, only two peaks will be seen (one for Endrin and one for DDT). As a rule, this is not the case. It is normal to observe up to six peaks. Three peaks are attributable to Endrin and its degradation products: Endrin Aldehyde (EA) and Endrin Ketone (EK). Three peaks are attributable to DDT and its degradation products: DDE and DDD. Calculate the percentage breakdown as follows:

Endrin:

$$\frac{\text{(Areas of EA + EK)}}{\text{(Areas of EA + EK + Endrin)}} \times 100 = \%\ \text{breakdown Endrin}$$

DDT:

$$\frac{\text{(Areas of DDE + DDD)}}{\text{(Areas of DDE + DDD + DDT)}} \times 100 = \%\ \text{breakdown DDT}$$

9.2.1.3. If the percentage breakdown for either Endrin or DDT is greater than 15%, the system CANNOT be used for pesticide analysis. If the Endrin/DDT performance check fails, injection port/column maintenance must be performed. Usually, changing the glass wool/liner will cure most breakdown problems in the injection port.

Depending upon the nature of the samples, the entire injection port will occasionally need to be cleaned. This cleaning is best done with 1:1 Acetone: Hexane. Another routine maintenance operation to improve column performance is the removal of the first 3 cm of the column. (Note: the septa should be changed each time the injection port is opened).

9.2.1.4. After injection port/column maintenance has been performed, and the columns have been given time to equilibrate (baseline back down to normal) the Endrin/DDT must be re-injected and the system performance re-evaluated.

9.2.2. Initial Calibration Range and Initial Calibration Verification (ICV)

9.2.2.1. ***Initial Calibration Range*:** Single component pesticides are calibrated using a five-point calibration range. Multi-component pesticides are calibrated using a single point calibration at the anticipated midpoint of the calibration range. Standards are prepared following the instructions in Section 7.2.

9.2.2.2. Single response Pesticide Calibration: All single component pesticides and two surrogates are calibrated with a minimum of 5 concentrations. Single component pesticides are analyzed at 10, (5 ppb for the 125 ml initial volume method) 5, 100, 250 and 500 ppb. Surrogate standards are analyzed at 25, 50, 100, 150 and 200 ppb. See Section 7.2 for details on standard prep.

9.2.2.3. Multi-response Pesticide Calibration: Chlordane (technical) and Toxaphene initial calibration is accomplished by analysis of a single point at 1000 ppb (see Section 7.2 for details on standard prep).

9.2.2.4. ***Initial Calibration Verification (ICV):*** An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2 and must be from a source separate from the standards used in the Initial Calibration Range.

9.2.3. Continuing Calibration Verification (CCV): For single component pesticides, a mid-point Continuing Calibration Verification (CCV) must be analyzed every 12-hours or 20 samples (whichever is more frequent). 'Samples' here are defined as field samples and batch QC (MB, LCS, MS, MSD) and do not include CCVs, PEMs, instrument blanks, etc… For multi-response pesticides a CCV must be analyzed within 12 hours of any multi-response pesticide detects.

9.2.3.1 Analysis of Replicate CCVs: Occasionally dual, sequential CCVs may (for a variety of reasons) be included in an analytical

sequence. When such replicate CCVs are injected both must be evaluated as detailed in the table below (reference Eurofins Document CA-Q-W-008, 'Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV)').

Dual CCV Evaluation Decision Tree		
Inejection	**QC Acceptance**	**Action**
CCV1 **CCV2**	**Pass** **Pass**	Continue analytical sequence. Data acceptable based on calibration.
CCV1 **CCV2**	**Pass** **Fail**	Analyses before CCV1 may be accepted. Re-analyze all samples that were analyzed after failed CCV2.
CCV1 **CCV2**	**Fail** **Pass**	Re-analyzed all samples that were analyzed before CCV1 and after the previous compliant CCV. Analyses after CCV2 may continue.
CCV1 **CCV2**	**Fail** **Fail**	Re-analyze all samples since the last acceptable CCV. Perform maintenance and/or recalibration prior to re-analysis.

9.2.3.2 In the event that replicate CCVs are included in a sample injection sequence, the acceptance of the associated sample analyses must be properly evaluated and the evaluation process must be documented. Under no circumstances is it allowable to accept sample data based on the evaluation of only 1 of the CCV replicates.

9.2.3.3 Clear documentation of the evaluation of the CCV must be included in the analytical sequence run log (i.e., 'Pass' or 'Fail' in the comment section for each CCV). Additionally, clear documentation of corrective action taken in the event of CCV failures must be included in the run log (see table above for actions that need documentation).

Note: It is acceptable to use the CCV solution as a primer at the beginning of an analytical sequence however the injection should be documented as a 'Primer' in the sequence rather than as a CCV.

9.2.4. Calibration Acceptance Summary

9.2.4.1. Retention Time Windows: Retention time windows must be established to compensate for minor shifts in absolute retention

times as a result of sample loading and normal chromatographic variability (for more detail on the following procedures refer to Eurofins Corporate Technical Services Policy No. CA-T-P-005, “Policy for Determining RT Windows for GC/ECD Tests” and Eurofins Corporate Technical Services Work Instruction No. CA-T-WI-010, “Further Guidance on the RT Window Policy No. CA-T-P-005”).

9.2.4.1.1. Initial determination of RT windows.

9.2.4.1.1.1. The center of the retention time (RT) window shall be updated based on the middle level in the initial calibration of the first CCV in the daily sequence, whichever is most recent.

9.2.4.1.1.2. Use a default RT window of 0.01 minutes. At the end of the batch evaluate all CCVs and LCSs in the batch. If necessary, widen the window such that all analytes fall within the RT window. Reprocess the batch using the new RT windows.

9.2.4.1.1.3. Multiply the maximum deviation by 1.5. This is the retention time window, unless the result is less than 0.01 minutes, in which case the window is set at 0.01 minutes. For example, if the maximum RT deviation for a specific analyte is 0.008 minutes, then the RT window is set at ±0.012 minutes.

NOTE: For the multi-component analytes, for example Aroclors, Toxaphene and Technical Chlordane, the maximum deviation must be evaluated for each of the 3 to 6 major peaks used for sample calculations.

9.2.4.1.1.4. Retention time windows for analytes of interest must not overlap for GC analysis.

9.2.4.1.2. Ongoing evaluation of retention time windows

9.2.4.1.2.1. Evaluate the retention time windows on an ongoing basis. The center of the RT window is updated on the first CCV of the day. All analytes for all subsequent CCVs, LCS and matrix spikes must fall within the retention time window (except as discussed below).

9.2.4.1.2.2. Matrix spike analytes may fall outside of the retention time window if there is a large non-

target peak coeluting with the analyte in the matrix spike.

9.2.4.1.2.3. If any analytes fall outside of the retention time window in CCVs, LCS or matrix spikes (except as discussed above for matrix spikes) then the RT windows for those analytes shall be widened to the minimum degree required for the analyte to fall within the RT window. All samples in the batch shall be reprocessed with the new RT window, and the wider RT window shall remain in place for subsequent batches.

9.2.4.1.2.4. Retention time windows should be reliably narrower than ±0.03 minutes. If RT windows wider that this are necessary, the instrument should be evaluated and maintenance performed as needed. Subsequent to maintenance, RT windows shall be narrowed to the extent that is consistent with the data obtained.

9.2.4.2. **Initial Calibration Range:** Internal standard calibration is employed for this method. A response factor is calculated for each analyte at each calibration concentration.

Response factor = $((A_x)\ (C_{is}))/((A_{is})\ (C_x))$

Where:
A_x = area of the compound
C_x = Concentration of the compound
A_{is} = area of the internal standard
C_{is} = Concentration of the internal standard

9.2.4.2.1. Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

Where:
RF = Response Factor

9.2.4.2.2. If the % RSD across the 5 point range is <20% for any given compound the calibration can be assumed to be linear and the average response factor can be used to calculate concentrations of target compounds in samples.

9.2.4.2.3. If the % RSD is >20% for any given compound, a first order linear regression may be applied to the data to calculate the calibration curve and determine sample

concentration. If this method is employed, the r^1 (Correlation Coefficient) value must be $\geq$ 0.990 for the calibration to be acceptable. Calibration is checked every 12 hours or after every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single component pesticide standards.

9.2.4.2.4. Chlordane and Toxaphene Calibration: Chlordane and Toxaphene are multiple response pesticides and are calibrated with a minimum of 5 points as required (i.e, within 12 hours of either analyte being detected in a sample). Three to eight peaks are used for calculation of response factors and the same criteria detailed above is applied to determine acceptability of calibration.

9.2.4.2.5. Resolution: All single component analyte peaks must exhibit at least 80% chromatographic resolution. The analyst performs a visual check of the mid-level initial calibration standard and all subsequent calibration checks (ICV/CCV). If the resolution requirement is not met instrument maintenance should be performed followed by re-calibration. Percent resolution can be calculated when necessary as follows:

$$\% \text{ Resolution} = V/H \times 100$$

Where:

V= Depth of the valley between two peaks. The depth of the valley is measured along a vertical line from the level of the apex of the shorter peak to the floor of the valley between the two peaks

H = Height of the shorter of the adjacent peaks

9.2.4.3. **Relative Standard Error:** The percent relative standard error (% RSE) from the calibration curve is used to evaluate the initial calibration. This provides a measure of how much error is associated with using the calibration curve for quantitation. The average Relative Standard Error (RSD for average response factors) of the calibration points from the curve used must be ≤ 20%.

Some data systems will not measure the %RSE from a linear or quadratic fit. For the linear case, the correlation coefficient may be used as an alternative to the %RSE, and must be greater than or equal to 0.990. For the quadratic case the Coefficient of Determination may be used, and must be greater or equal to 0.990.

Note: The Relative Standard Error (RSE) is superior to the Correlation Coefficient (*r*) and Coefficient of Determination (r_2) for testing the fit of a set of calibration points to a line. The lower points on a curve have little effect on *r.* As a result, a curve may have a very good correlation coefficient (>0.990) while also having > 100% error at the low point

9.2.4.4. Initial Calibration Verification (ICV): An ICV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.2.4. The calculated concentration of the ICV must be within ±20%D of the expected concentration. Should the %D exceed 20% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 20% after a single ICV reinjection, a new Initial Calibration Range must be analyzed. All single component analyte peaks must exhibit 80% resolution (see Section 9.2.4.2.5)

9.2.4.5. Continuing Calibration Verification (CCV): A CCV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.3.. The calculated concentration of the CCV must be within ±20%D of the expected concentration. Should the %D exceed 20% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 20% after a single CCV reinjection, a new Initial Calibration Range must be analyzed. All single component analyte peaks must exhibit 80% resolution (see Section 9.2.4.2.5).

Step	***Standards***	***Type***	***Control Limit***	***Frequency***
Method # 8081B				
GC System Performance Check	*Endrin/DDT, 250 ppb*	*Performance check*	*15% breakdown*	*At beginning of 12 hour clock and after system maintenance*
*Initial Calibration**	2.5, 50, 100, 250 and 500 ppb for single response, 1000 ppb for multi response	*Average response factor or 1st order linear regression*	*For average RF: <20%RSD all analytes. For linear regression: r ≥0.990 or <20%RSE*	*As required when ICV or CCV do not meet requirements*
ICV	*100 ppb*	*Average*	*± 20%D*	*Once after each initial calibration*
CCV	*100 ppb*	*Average*	*± 20%D*	*Every 12 hrs or 20 samples, whichever is more frequent*

10. Procedure

10.1. Gas Chromatograph Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

10.1.2. General Operating Conditions

10.1.2.1. Injection System: A split/splitless injection port with electronic pressure control (EPC) is used. Thirty seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port.

10.1.2.2. The EPC is used in the ramp pressure mode. The ramp pressure program is as follows:

Initial Pressure	InitialTime	Rate	Final Pressure	Hold
12 psi	2.5 min	7 psi/min	4 psi	1.50 min
		5 psi/min	9 psi	1.40 min
		9 psi/min	13 psi	2.00 min

10.1.2.3. For pesticide analysis the normal operating conditions of the injection port are as follows:

Injection port Temperature:	250^{o}C
Column flow:	12.3 ml/minute
Split vent flow:	5 ml/minute
EPC:	Pressure Ramp
Detector temperature	330C

10.1.2.4. In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. The plug of glass wool is located in the liner between the double goose neck.

10.1.2.5. This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.

10.1.2.6. The glass wool will be changed when changing the liner. The changing of the glass wool/liner is based upon the breakdown of an Endrin/DDT standard. This is covered in further detail in section 10.2.1.

10.1.2.7. Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.

10.1.2.8. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

10.1.2.9. The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

10.1.2.10. Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.

10.1.2.11. Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.

10.1.2.11.1. A standard oven program for pesticide analysis is employed for all columns as follows:

Initial Temp	**Hold Time1**	**Rate1**	**Temp1**
160ºC	0.62 min	30º/min	244ºC
Hold Time2	**Rate 2**	**Final Temp**	**Final Time**
2.5min	21º/min	315ºC	3.0min

10.1.2.12. Detectors: Detectors operate at 330^{O}C and need to be supplied with 60 ml/min total flow. They are essentially maintenance free on a day-to-day basis. They are routinely baked out at 330^{O}C to remove persistent contaminants. On occasion the detectors may be baked out at a higher temperature to remove contaminants with an extremely high boiling point (CAUTION: Do not exceed the maximum detector temperature of 380^{O}C).

10.1.2.12.1. If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.

10.1.2.13. Chemstation: HP Chemstation software is used for automation of runs and data acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.

10.1.2.14. Eurofins Chrom data processing software is used for the processing of the chromatography data files. Calibrations, verification standards and samples are processed and reviewed using this database. Chrom is integral to Eurofins LIMS (TALS) which is used to generate all reports..

10.2. Analytical Sequence

10.2.1. The analytical sequence for performance checks, initial calibration, calibration verifications and sample analysis is described in the following sections.

10.2.2. Before calibration standards are analyzed the GC Performance Check Standard (see Section 9.2.2.1) must first be analyzed and evaluated to check the performance of the injection port and column with regard to catalytic active sites. The breakdown for both Endrin and DDT in the Performance Check Standard must be less than 15%. If the performance check fails this criteria system maintenance must be performed and the check successfully reanalyzed before proceeding with calibration.

10.2.3. A five point initial calibration (ICAL) is analyzed and evaluated for each of the 21 single response pesticides plus surrogates as described in Section 9.2.4.2. When needed a 5 point ICAL is also analyzed for Mirex.

10.2.4. Calibration for Technical Chlordane and Toxaphene: Chlordane and Toxaphene are multiple response pesticides containing at least 3-8 primary peaks each. A single point calibration check standard at a concentration of 1000ug/l is analyzed for Chlordane and Toxaphene. A full 5 point calibration range is analyzed for these compounds should they be detected in client samples..

10.2.5. A second source initial calibration verification (ICV) is analyzed and evaluated for each of the 21 single response pesticides as described in Section 9.2.4.3. When needed an ICV is also analyzed for Mirex. **NOTE**: The 12 hour time clock for Pesticides commences with the injection of the first Pesticide Calibration Standard or Verification.

10.2.6. GC Performance Check Standard is analyzed and the breakdown of Endrin/DDT is measured again before samples are analyzed and at the beginning of each subsequent 12 hour shift. If the breakdown check fails, then injection port/column maintenance is required

10.2.7. Client samples and QC samples may be analyzed after the analysis of the performance check. Sample analysis may proceed for up to 20 samples or 12 hours prior to analysis of another calibration verification (whichever is more frequent).

10.2.8. A Continuing Calibration Verification (CCV) for the 21 single response pesticides plus surrogates must be analyzed and evaluated every 12 hours or 20 samples (whichever is more frequent) as described in Section 9.2.4.4.. This is accomplished by running the midpoint calibration standard as a CCV (Pest Mix 100 ppb check standard and Mirex 100 ppb check standard; see Section 7.2.2.1). The calculated concentration for each compound in the CCV must be +/- 20 % of the expected concentration. Any samples analyzed after a failing CCV must be reanalyzed under a passing CCV. If, after performing instrument maintenance, the reanalysis of a CCV fails criteria, a new initial calibration range must be analyzed (see Section 9.2.4.2). Any data reported against a failing CCV must have a Non-Conformance Memo detailing the issue.

10.2.9. Analytical Sequence: The automation of GC runs is accomplished via the "SEQUENCE" macro of the Chemstation. The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle. It is common practice to run the check standards, evaluate the instrument status, and then complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro.

Example Analytical Sequence	
1. Hexane 2. Instrument Blank	12. Mirex 1 (10 ppb) (if needed) 13. Mirex 2 (20 ppb) (if needed)

Example Analytical Sequence	
3. Endrin/DDT Breakdown 4. Pesticide Mix 1 (2.5 ppb) 5. Pesticide Mix 2 (50 ppb) 6. Pesticide Mix 3 (100 ppb) 7. Pesticide Mix 4 (250 ppb) 8. Pesticide Mix 5 (500 ppb) 9. Chlordane (1000 ppb) 10. Toxaphene (1000 ppb) 11. Pesticide ICV (100 ppb)	14. Mirex 3 (100 ppb) (if needed) 15. Mirex 4 (250 ppb) (if needed) 16. Mirex 5 (500 ppb) (if needed) 17. Mirex ICV (100 ppb) (if needed) 18. Instrument Blank 19. Endrin/DDT Breakdown 20. Pest Mix 3 (100 ppb)…CCV 21. Mirex 3 (100 ppb) …CCV (if needed) 22. 20 or fewer samples or 12 hours 23. Instrument Blank 24. Endrin/DDT Breakdown 25. Pest Mix 3 (100 ppb)…CCV 26. Mirex 3 (100 ppb) …CCV (if needed) 27.20 or fewer samples or 12 hours

10.3. Dual Column Approach

10.3.1. The laboratory designates the rear column as the primary column and the front column as the secondary column. If the difference between the dual columns results in ≤40% RPD report the higher concentration.

10.3.2. The values are calculated from the chromatographic peaks that fall within the daily retention time windows. Retention times are updated using the retention times found in opening CCV for the most recent 12 hour period.

10.3.3. If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P*. **Exception:** NJDEP DKQP protocols require reporting the higher concentration in all instances.

10.3.4. If the surrogates on one column are >40% RPD compared to the other column, this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.

10.3.5. If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the compliant column. If the falls outside of acceptance criteria on the low side, reanalysis shall be performed.

10.3.6. If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

10.3.6.1. An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.

10.3.7. In some cases where the sample chromatography is complex and has largely varying peaks concentrations, the chromatographic separation may not be sufficient on the 0.53mm ID columns. In this case a confirmatory analysis on an instrument with 0.32 ID columns may be required. The supplemental data produced using analysis on the 0.32mm ID 'microbore' column may minimize overlapping and baseline interference difficulties, and better resolves potential positive identifications. Use of this alternative chromatographic technique shall be noted in the job summary and report case narrative.

10.3.8. In summary, the flow chart in Attachment 1 presents a recommended rational approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

10.4. Extract Cleanups

10.4.1. Cleanup methods are dictated by the original sample matrix and the parameters being determined.

10.4.2. Cleanup of all water samples, if needed, is performed using Florisil (Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision) and TBA sulfite (Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision). Blanks must also undergo cleanup following the same procedures as samples.

10.4.3. Cleanup of all soil samples is conducted using Florisil (Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision) and, if needed, TBA sulfite (Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision). Blanks must also undergo cleanup following the same procedures as samples.

10.4.4. Check Florisil prior to use to assure quantitative recovery of target analytes. (see Section 4.3.2 above and Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B,

10.5. Data Processing and Documentation

10.5.1. Before the analysis sequence is initiated the GC Maintenance logbook must be filled out. It should contain the following information: date, injector temp, oven temp, detector temp, injector flow, signal A, signal B, analysts initials, and notes for any necessary repairs.

10.5.2. After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor, method, job number, QA number, extraction date, lab prep batch, Chrom batch signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (O.K., dilution, non-inject, etc.).

10.5.3. The reporting limit is based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and %moisture (No unqualified analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).

10.5.4. Any compound concentration that exceeds the concentration of the calibration range must be diluted and re-analyzed.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculation of Sample Amounts (Internal Standard Procedure)

11.3.1.1 Aqueous Samples

$$\text{Concentration } (\mu g/L) = \frac{(As)(Cis)(D)}{(Ais)(RF)(Vs)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

11.3.1.2 Solid Samples

$$\text{Concentration } (\mu g/kg) = \frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws)\ (Vi)\ (1000)}$$

Where:

As	=	Area of the target analyte peak in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the internal standard peak
RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

11.4. Relative Response Factors

$$RRF = \frac{A_x}{A_{is}} \times \frac{C_{is}}{C_x}$$

Where:

A_x = Area of target analyte peak
Ais = Area of internal standard peak
Cis = Concentration of internal standard
Cx = Concentration of compound in standard

11.5. Percent Relative Standard Deviation (% RSD):

$$\% \text{ RSD} = \frac{\text{Standard Deviation of RRFs}}{\text{Mean RRF}}$$

11.6. Relative Standard Error (RSE)

$$\%\,RSE = 100\% \times \sqrt{\frac{\sum_{i=1}^{N}\left[\frac{C_i - PC_i}{C_i}\right]^2}{(N-P)}}$$

Where:

N = Number of points in the curve
P = Number of parameters in the curve (= 1 for average response factor, 2 for linear, 3 for quadratic)
CI = True concentration for level i
PCi = Predicted concentration for level i

11.7. Percent Difference (% D):

$$\% \text{ D} = \frac{RRF_c - \overline{RRF_i}}{\overline{RRF_i}} \times 100$$

Where: RRFc = RRF from continuing calibration

$\overline{RRF_i}$ = Mean RRF from current initial calibration

11.8. Percent Recovery (% R): Surrogates and Spikes

$$\text{Recovery (\%)} = \frac{\text{Concentration (or amount) found}}{\text{Concentration (or amount) added}} \times 100$$

11.9. Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

$$DW = \frac{Gd}{Gw} \times 100$$

Where:

DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted.

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Lower Limit of Quantitation (LLOQ) (aka Reporting Limit) Verification:

The LLOQ is the lowest concentration at which the laboratory has demonstrated target analytes can be reliably measured and reported with a certain degree of confidence, which must be ≥ the lowest point in the calibration curve. The lab verifies the LLOQ annually to demonstrate the capability of quantiation at lower analyte concentrations. The verification is performed by the extraction and analysis of an MDL spike at a concentration of 0.5-2 times the established LLOQ. Recovery of target analytes in the LLOQ verification should be within established in-house limits or within other such project-specific acceptance limits to demonstrate acceptable method performance at the LLOQ. Until the laboratory has sufficient data to determine acceptance limits, the LCS criteria ± 20% (i.e., lower limit minus 20% and upper limit plus 20%) will be used for the LLOQ acceptance criteria. The annual LLOQ verification is completed and documented with the required annual MDL evaluation.

12.3. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.4. Training Requirements

Refer to Eurofins SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

13.0. Pollution Control

13.1. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

13.2. The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage.

14.0. Waste Management

14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for “Waste Management and Pollution Prevention.”

14.2. The following waste streams are generated as a result of this analysis:

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.

- Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

  Teris Profile Number: 50016652
  Onyx Profile WIP Number: 282493

- Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

  Teris Profile Number: 50016624
  Onyx Profile WIP Number: 545240

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

  Teris Profile Number (incineration): 50016710
  Onyx Profile Number: (stabilization) 402535

15.0. References / Cross-References

15.1. United States Environmental Protection Agency, "Method SW8000C: Determinative Chromatographic Separations,", Test Methods for Evaluating Solid Wastes, SW846, Revision 3, March 2003.

15.2. United States Environmental Protection Agency, "Method 8081B, Organochlorine Pesticide by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 2, February 2007.

15.3. Eurofins Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. Eurofins Edison SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C,* most current revision.

15.5. Eurofins Edison SOP No. ED-ORP-018*: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B,* most current revision.

15.6. Eurofins Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546*

15.7. Eurofins Edison SOP No. ED-ORP-019*: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A,* most current revision

15.8. Eurofins Edison SOP No. ED-ORP-051: *Extraction of Semivolatile Organic Compounds in Water by Microextraction using SW846 Method 3511*

15.9. Eurofins Edison SOP No. ED-ORP-020*: Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision.

15.10. Eurofins Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision.

15.11. Eurofins Edison SOP No. ED-GEN-022, *Training,* most current revision.

15.12. Eurofins Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*), most current revision.

15.13. Eurofins Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), most current revision.

15.14. Eurofins Document CA-Q-W-008, 'Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV), most current version.

15.15. Eurofins Corporate Quality SOP No. CA-Q-P-004, "Reporting Results for Methods that Require Second-Column Confirmation", most current revision.

15.16. Eurofins Corporate Work Instruction No. CA-T-WI-008, "Minimum Requirements for Pesticide Analysis by SW846 Method 8081B", most current revision.

15.17. Eurofins Corporate Policy CA-T-P-004, "Policy for Determining RT Windows for GC/ECD Tests", most current revision.

15.18. Eurofins Corporate Technical Services Work Instruction No. CA-T-WI-010, "Further Guidance on the RT Window Policy No. CA-T-P-005", most current revision.

16.0. Method Modifications:

None

17.0. Attachments

Attachment 1: Dual Column Reporting Flowchart

18.0. Revision History

Revision 8, dated 06/12/2024

- Updated to include SW846 3511 as aqueous prep method.

Revision 7, dated 10/17/2022:

- Updated to Eurofins branding throughout document.
- Revised Section 8 to include requirement to treat samples from chlorinated sources with sodium thiosulfate
- Section 9.2.4.3: Included details of evaluation of initial calibration for Relative Standard Error (RSE).
- Section 11.6: added formula for calculation of RSE.
- Section 12.2 added: details the annual LLOQ verification requirement.

Revision 6, dated 03/25/2021:

- Added Section 10.5.3 which details how the reporting limit is established.
- Added Section 10.5.4 which discusses requirement to dilute analytes into range of calibration.

Revision 5, dated 10/12/2020:

- Edited throughout to reflect Eurofins branding and policy.
- Updated Lab Quality Manual section references as needed.

Revision 4, dated 09/13/2016:

- Section 9.2.4.1: completely rewrote this section to reflect requirements of Eurofins Corporate Technical Services Policy No. CA-T-P-005, “Policy for Determining RT Windows for GC/ECD Tests” and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010
- Section 15 (References): added references to TestAmerica Corporate Technical Services Policy No. CA-T-P-005, “Policy for Determining RT Windows for GC/ECD Tests” and TestAmerica Corporate Technical Services Work Instruction No. CA-T-WI-010, “Further Guidance on the RT Window Policy No. CA-T-P-005”.
- Attachment 1: RT Windows for Single Analytes/Surrogates: DELETED and renamed subsequent attachments.

Revision 3, dated 01/05/2016:

- Section 2.2: expanded to include summary of internal standard calibration.
- Section 7.2.2: added 1-Bromo-2-nitrobenzene (internal standard) to the list of standards (Restek 32279). Components and concentration added to table.
- Added Section 7.2.2.4 which describes the preparation of the internal standard solution. All subsequent sections renumbered accordingly.
- Section 7.2.2.1.1: added footnote to ICAL standard prep table detailing requirement to spike each standard with internal standard solution.
- Section 7.2.2.4: describe internal standard spiking protocol (20 ul of 5.0 ug/ml solution into all standards, QC extracts and field sample extracts.
- Section 9.1: added internal standard to ‘Sample QC’ table.
- Section 9.1.4: added following minimum requirements for surrogate evaluation and reporting: a) Both surrogates must have reportable results that meet the acceptance criteria; b)reported surrogates must be from a column with a passing CCV; c)At least one surrogate must pass on any column from which target analytes are identified and reported. (per corp “Minimum Requirements” document).
- Section 9.1.5: added this section which describes acceptance criteria for internal standards (retention time and response).
- Section 9.2.3: Added the following sentence: “‘Samples’ here are defined as field samples and batch QC (MB, LCS, MS, MSD) and do not include CCVs, PEMs, instrument blanks, etc… “. Deleted the phrase ‘and at the end of every sequence’. (both changes reflect ‘Minimum Requirements for Pesticide Analysis’ document).
- Added Section 9.2.4.1.3: “Updating absolute retention times: Update retention times with the retention times found in the opening CCV for that 12 hour period.”
- Section 9.2.4.2: replaced ‘external standard’ with ‘internal standard’. Added formula for calculation of response factors by internal standard method. Deleted text regarding calibration acceptance criteria from this section as it is duplicated in subsequent sections.
- Section 9.2.4.2.2: added phrase ‘for any given compound.’
- Added section 9.2.4.2.5: describes resolution check.

- Sections 9.2.4.3 and 9.2.4.4: added statement concerning 80% resolution requirement.
- Section 9.2.4.4: changed 'ICV' to 'CCV' in last sentence.
- Section 10.1.2.13: Revised first sentence to read: ": HP Chemstation software is used for automation of runs and data acquisition."
- Section 10.2.2: corrected reference to Section 9.2.2.1 (was incorrectly listed as 7.2.2.1).
- Section 10.2.8: revised to clarify that CCV bracketing is not required and that only samples analyzed after a failing CCV must be reanalyzed (i.e., samples analyzed immediately prior to a failing CCV need not be reanalyzed).
- Section 10.3.2: added the following regarding updating retention times: "Retention times are updated using the retention times found in opening CCV for the most recent 12 hour period."
- Section 11: added formulas for calculation of sample concentrations as well as %RSD, %D, % Recovery, RRF and dry weight correction.
- Section 15: removed reference to SOP No. ED-ORP-016*: Automated Soxhlet Extraction of Solid Samples – Pesticides/PCBs, SW846 Method 3541* as it is no longer is use.
- Section 15: added reference to TestAmerica Corporate Quality SOP No. CA-Q-P-004, "Reporting Results for Methods that Require Second-Column Confirmation"
- Section 15: added reference to TestAmerica Corporate Work Instruction No. CA-T-WI-008, "Minimum Requirements for Pesticide Analysis by SW846 Method 8081B".
- Section 15: added reference to TestAmerica Corporate Policy Memorandum No. CA-Q-QM-006, "Technical Guidelines for Analysis of Complex GC/ECD Chromatograms".

- Revision 2, dated 13 May 2015:
  - Section 1.1: updated RLs to reflect current lab practice.
  - Section 2.1 and throughout: revised initial aqueous/TCLP sample volumes to reflect current lab practice
  - Section 2.1.1: removed note describing option for Reduced Volume Extraction (RVE) as this is now standard lab practice.
  - Section 2.1.2 and throughout document: removed reference to prep by SW3541 as this method is no longer in use at Edison lab.
  - Section 4.3.2 and throughout: updated source of standards used for Florisil check.
  - Section 6.1.2.3.1: updated name of analytical GC columns currently in use.
  - Section 6.1.2.4.1: updated make-up gas to P-5 & Nitrogen.
  - Section 7.1.1: updated carrier gas to Hydrogen and make-up gas to P-5&Nitrogen.
  - Section 7.2.2 and throughout document: updated source and catalog numbers for analytical standards (Restek is now primary source of standards).
  - Section 7.2.2.1: updated standards prep instructions. Removed outdated references to standards and spiking mixes. Removed notes pertaining to RVE since this procedure is now standard and incorporated into instructions.
  - Section 7.2.2.10: added instructions for preparation of ICVs.
  - Section 8.1: adjusted water sample container from 1000ml to 250ml.
  - Section 9.2.2: removed references and instructions pertaining to the Pesticide Resolution Check (a CLP requirement we no longer perform).
  - Section 9.2.3.1: Added extensive discussion regarding the analysis of replicated CCVs.

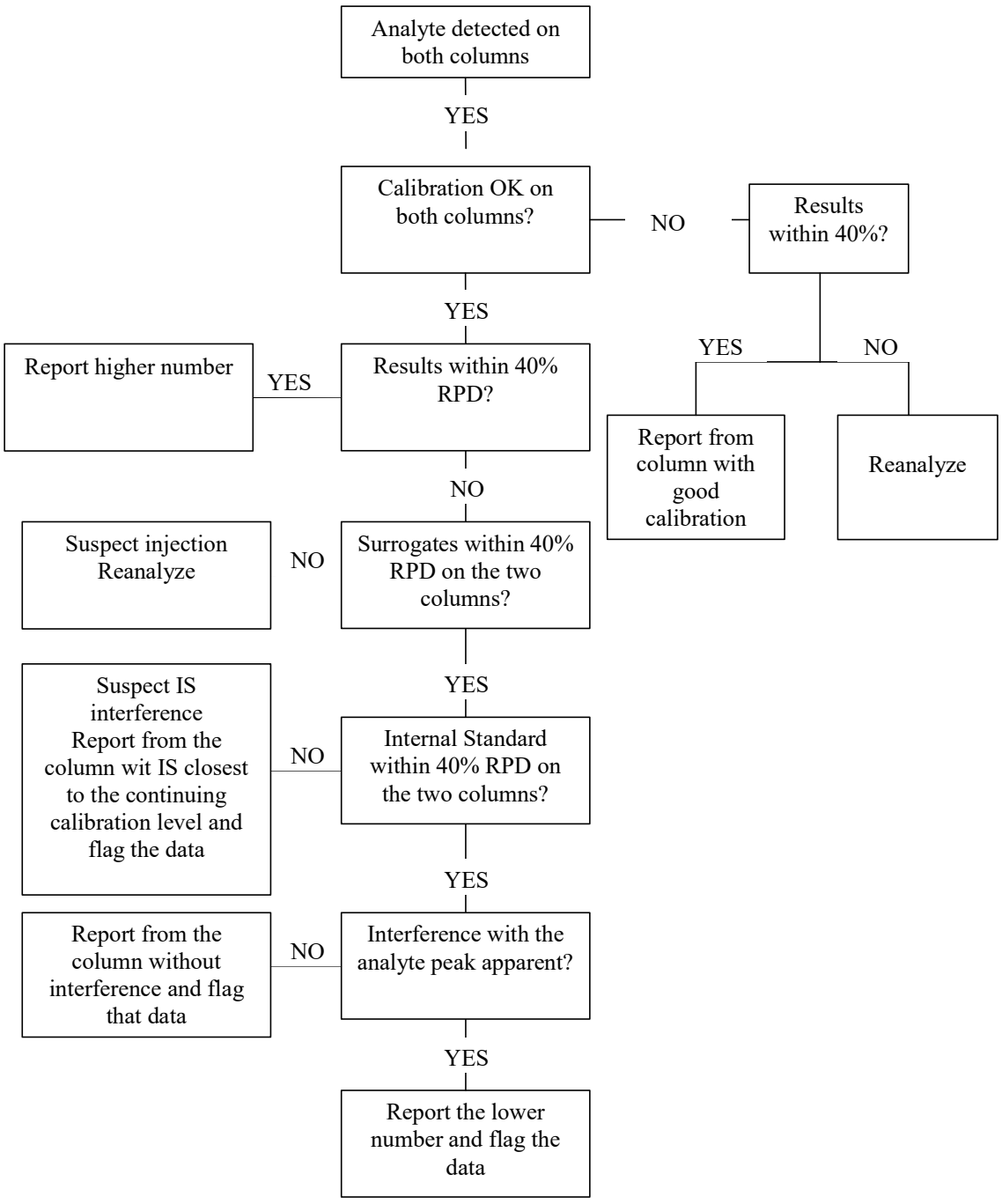
- Section 9.2.4.4: updated table to include new concentration for low standard (2.5 ppb).
- Section 10.1.2.14 and throughout document: replaced references to Target with TestAmerica Chrom.
- Section 10.2: made extensive revisions to discussion of the analytical sequence (removing duplicate entries and adjusting to reflect current procedures).
- Section 10.3: added note concerning NJ DKQP requirements for reporting highest concentration of dual columns.
- Section 15 (References): updated to include "Technical Guidance on the Use and Evaluation of Replicate Continuing Calibration Verification (CCV), most current version."
- Added Sections 7.2.2.11 and 7.2.2.12 reflecting the addition and preparation of Internal standard

- Revision 1, dated 09 October 2012
  - Throughout: Revised LQM section number references to reflect the most current LQM revision.
  - Section 2.1.1: added description of reduced initial volume (125ml)/final volume (1ml) option.
  - Section 2.1.2: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546.*
  - Section 7.2.2.1.1 added preparation of 5ppb standard for the reduced volume method.
  - Section 9.2.2.2 Added 5ppb standard for the reduced volume method
  - Section 9.2.4.4 Added 5 ppb standard to the initial calibration for the reduced volume method
  - Section 10.2.6 Added 5 ppb to the Analytical sequence for the low level method Section 15.0: Removed reference to TestAmerica Edison SOP EDS-GEN-019, *Organic Calculations,* most current revision.
  - Section 15.0: Added reference to TestAmerica Edison SOP ED-ORP-044, *Procedure for the Microwave Extraction of Solids, SW846 3546*

- Revision 0, dated 02/17/2011: New

ATTACHMENT 1

Dual Column Reporting Flowchart
(Note: NJDEP DKQP requires reporting the higher concentration in all instances)



Title: Analysis of Total and Amenable Cyanide in Water, Drinking water, Wastewater and Soil- Automated by Method EPA SW846 9012B; 335.4 and Standard Method 4500 CN^-C, G

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Approvals (Signature/Date):

Jasmine Parillo 11/6/23	Dan Helfrich 11/6/23
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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 Methods EPA 335.4, SW846 9012B, Lachat 10-204-00-1-A and SM 4500 CN^-C, G are described in this SOP. The SOP is applicable to water, drinking water, wastewater, soil and waste samples requiring cyanide determination. The method detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine values for both total cyanide, free cyanide and cyanide amenable to chlorination.

1.1.2 The automated method has a detector that is sensitive to approximately to 0.005 mg CN-/L. The laboratory's reporting limit are as follows:

Analyte	Water Reporting Limit (mg/L)	Soil Reporting Limit (mg/Kg) Micro Distillation
Cyanide	**0.01**	**0.24**

1.1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Cyanide is extracted by means of reflux distillation (MICRO DIST apparatus). Sample is distilled in a strongly acidic solution which breaks down cyanide complexes and converts the cyanide to hydrocyanic acid (HCN). HCN is absorbed into a sodium hydroxide absorber solution and the cyanide ion in the absorbing solution is determined by automated UV colorimetry at 570nm.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Interferences can be reduced or eliminated by using the distillation procedure.

4.2 The presence of sulfide in the sample matrix adversely effects the determination of total cyanide. Samples that contain hydrogen sulfide, metals sulfides or other compounds that may produce hydrogen sulfide during distillation should be treated.

4.2.1 Place a drop of the sample on lead acetate test paper (which has been pre-moistened with pH 4 acetate buffer solution) to detect the presence of sulfides. If sulfides are present (test strip turns black), the sample volume

required for the cyanide determination should be increased by 25 milliliters (ml).

4.2.2 The total volume of sample should be treated with powdered cadmium carbonate or lead carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide.

4.2.3 Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper

4.2.4 Filter the solution through a dry filter paper into a dry beaker, and from the filtrate measure the sample to be used for analysis.

NOTE: Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss of cyanide on the precipitated material.

4.3 Oxidizing agents such as chlorine decompose most cyanides.

4.3.1 To determine if oxidizing agents are present test a drop of sample with potassium iodine-starch paper. If a blue color appears that indicates the need for treatment. Add ascorbic acid, a few crystals at a time, to the sample until a drop of sample produces no blue color on the indicator paper. Add an additional 0.6g of ascorbic acid for each liter of sample volume.

4.4 Nitrates and nitrites may give high biased results. During distillation they form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose to generate HCN. They are interferences when present above 10 mg/L with other specific organic compounds.

4.4.1 Nitrate and nitrite interferences are eliminated by treatment with sulfamic acid just before distillation.

4.5 Thiocyanate is reported to be an interference when present at very high levels. Levels of 10 mg/L were not found to interfere.

4.6 Fatty acids, detergents, surfactants may cause foaming during the distillation process, making the endpoint difficult to detect. If this occurs, samples can be acidified with acetic acid (1.6M) to pH 6.0-7.0.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates. Potassium Cyanide will give off Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3-Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Barbituric Acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as potential health hazard; do not ingest.
Hydrochloric Acid	Corrosive Poison	5 ppm – Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Potassium Hydroxide	Poison Corrosive Reactive	2 mg/m3 – Ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting, diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness, swelling. Greater exposures cause severe burns with possible blindness.
Potassium Cyanide	Poison Corrosive	5 Mg/M3 TWA as CN	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Sodium Cyanide	Poison Corrosive	5 mg/m3 TWA as CN (skin)	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Silver Nitrate	Poison Corrosive Oxidizer	0.01mg/m3 (TWA) for silver metal dust and fume as Ag	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting, and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat, and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns and eye damage.
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1. Instrumentation

- MICRO DIST distillation apparatus with sample distillation tube press (Hach).

- Lachat 8000 and 8500 series: Flow injection analysis equipment-designed to deliver and react sample and reagents in the required order and ratios. Lachat 8000 series

  - Multichannel proportioning pump
  - Reaction manifold
  - Colorimetric detector
  - Data system
  - Heating Unit

6.2. Supplies

- Lachat Micro Distillation Tubing, p/n A17117 (collector tubes, caps, membranes, and sample tubes) or equivalent, disposable
- Various Class A volumetric flask and pipettors
- pH-indicator strips (pH 0-14)
- Potassium Iodine-starch paper
- Lead-Acetate paper (pre-moistened with pH 4 acetate buffer solution)
- Analytical balance
- Vacuum pump
- Specimen cups
- Transfer pipettes
- Graduated cylinder (class A)
- Micro-buret

7. Reagents and Standards

7.1. Reagents

7.1.1. Deionized water-18 megohm reagent grade Type II water.

7.1.2. Acetone – store at room temperature; for stability information refer to manufacturer's instructions.

7.1.3. Rhodanine indicator - p-dimethyl-aminobenzalrhodanine: Place 20 mg of p-dimethyl-aminobenzalrhodanine in a 100ml volumetric flask and dilute to the

mark with acetone. Store at room temperature. Stable for six months.

7.1.4. Acetate Buffer - Dissolve 82.0 gram of $NaC_2H_3O_2$ x $3H_2O$ in 100ml DI water. Add sufficient glacial acetic acid and adjust pH to 4.5 (approx. 10 drops). This solution is stable for 6 months, store at 4ºC.

7.1.5. Cadmium Carbonate (powder) ACS reagent –store at room temperature; discard after 5 years from date receipt or earlier if necessary.

7.1.6. Reagents for cyanides amenable to chlorination.

7.1.6.1. Calcium hypochlorite solution, (0.35M), $Ca(OCl)_2$. Dissolve 5 g of calcium hypochlorite in 100 ml of deionized water. Prepare the solution fresh daily, store at room temperature. Solution is stable for 1 month, Note: store in an amber bottle if not used immediately.

7.1.6.2. 0.25 N NaOH. Dissolve 10.0 g NaOH into 1 L of deionized water. Dilute to mark with deionized water and invert to mix. Solution is stable for 6 months, store at room temperature.

7.1.6.3. Sodium Arsenite, NaAsO2. Dissolve 2g of Sodium Arsenite in 100ml of DI water. Prepare solution every month; store at room temperature.

7.1.7. Reagents for cyanide distillation.

7.1.7.1. 0.25 M NaOH. See section 7.1.6.2

7.1.7.2. Sulfamic Acid Solution: Dissolve 9.6 g. H_2NSO_3H into 100ml of deionized water. Solution is stable for 6 months, store at room temperature.

7.1.7.3. Micro Distillation Releasing Agent (7.11 M sulfuric acid/ 0.79M magnesium chloride): In a hood, place a 500ml beaker on a top-loading balance and tare. Then add 110.8g DI water. Then add and dissolve completely 32.2g magnesium chloride hexahydrate ($MgCl_2$ x $6H_2O$) in this water. Slowly add 139g concentrated sulfuric acid a little at a time, swirling and allowing to cool. Solution is stable for 6 months, store at room temperature.

7.1.7.4. Bismuth nitrate Solution: Dissolve 3 g of $Bi(NO)_3$•$5H_2O$ in 10 mls DEIONIZED WATER , add 25 mls glacial acetic acid while stirring and dilute to a 100 ml final volume.

7.1.8. Reagents for automated colorimetric determination.

7.1.8.1. Pyridine-Barbituric acid Reagent: In a fume hood, place 15.0 g barbituric acid in a 1 L beaker and add 100 ml DEIONIZED WATER, rinsing down the sides of the beaker to wet the barbituric. Add 75 ml pyridine (C_5H_5N) while stirring and mix until

barbituric acid dissolves. Add 15 ml conc. HCl (12 M HCl) and mix. Transfer to a 1 L volumetric flask, dilute to mark and invert to mix. Prepare weekly, store solution in amber bottle and at room temperature.

7.1.8.2. Chloramine-T: Dissolve 2.0g chloramine-T hydrate in 500 ml deionized water. Prepare fresh daily. Store at room temperature.

7.1.8.3. Phosphate buffer: In a 1 L volumetric flask, dissolve 97.0 g potassium phosphate, monobasic, anhydrous, (KH_2PO_4) in approximately 800 ml of DEIONIZED WATER. Dilute to mark and invert to mix. Solution is stable for one month. Store at room temperature.

7.1.8.4. 0.25 M NaOH (carrier) See section 7.1.6.2.

7.2. Standards

7.2.1. Standard 0.0192N Silver nitrate solution (1 ml = 1 mg CN): Prepared by weighing 3.2647 g of dried $AgNO_3$, dissolved in deionized water and dilute to 1000ml. Solution is stable for 6 months or when QC check is outside the acceptable limits, whichever comes first, store at 4ºC.

7.2.2. Primary (Simple) Cyanide stock solution, 1000 mg/L: This standard is used for the preparation of calibration standards (Sec 10.2). Purchased commercially from Accustandard (catalog number WC-CN-10X-5). Follow manufacturer's instructions for storage and stability. Standardize the solution against 0.0192N silver nitrate solution quarterly.

If prepared in house, prepare as follows: Primary Cyanide stock solution, 1000 mg/L: Dissolve 2.51 gm of KCN and 2.0 gm of KOH in 900 ml of deionized water. Standardize the solution against 0.0192N silver nitrate solution weekly and dilute to the appropriate concentration so that 1ml=1mg CN. Solution is stable for 6 months, store at 4ºC. Standardize the solution as follows::

- Take 5.0 ml of the primary stock cyanide solution (Sec. 7.2.2) and dilute it to 50ml using the 0.25 M NaOH. Add 10-12 drops of the rhodanine indicator (Sec. 7.1.3) to produce a bright yellow color.
- Using the 10 ml micro buret, titrate the stock cyanide solution from a bright yellow color to a salmon hue endpoint. Record the volume of silver nitrate solution used for the titration in the Standardization logbook.
- Titrate a blank using 50mls of 0.25N NaOH, 10-12 drops of rhodanine indicator. Record the volume of silver nitrate solution used for the titration in the Standardization logbook.
- Calculate CN concentration as follows:

Based on the following: $Ag^{+} + 2\ CN \rightarrow [Ag(CN)_2]^{-}$

$$CN^{-}(mg/L) = \frac{A-B}{C} \times N \times \frac{2\ mol\ CN}{1\ eq\ AgNO_3} \times \frac{26.02g\ CN}{1\ mol\ CN} \times \frac{1000mg}{1\ g}$$

Where:
A= ml $AgNO_3$ for titration of sample
B=ml $AgNO_3$ for titration of blank
C= ml or g of sample
N= normality of $AgNO_3$

- Record the preparation in the reagent TALS. Record final concentration in the Standardization logbook.

- **Note***:/standardizedconcentration* .

7.2.3. Secondary (Simple) Cyanide Stock Solution, 1000mg/L: The secondary cyanide stock solution must come from a source different from the primary cyanide stock solution (ERA, cat# 997 or Accustandard, cat# WC-CN-10X-5). Follow manufacturer's instructions for storage and stability. Standardize the solution against 0.0192N silver nitrate solution quarterly. This standard is used for the preparation of ICV and CCV standards (Sec 9.2.1 and 9.2.2).

If prepared in house, prepare as follows: Dissolve 2.51 gm of KCN and 2.0 gm of KOH in 900 ml of deionized water. Note: the secondary cyanide stock solution must be prepared from a different source than the primary cyanide stock solution. Standardize the solution with 0.0192N silver nitrate solution weekly and dilute to the appropriate concentration so that 1ml=1mg CN. The solution is stable for 6 months, store at 4ºC. Follow the standardization procedure in 7.2.2.

Note: */standardizedconcentration* .

7.2.4. Primary Working Solution, 6 mg/L: Take 0.6 mL of the Primary Cyanide Stock Solution (Sec. 7.2.2) and dilute to a final volume of 100ml with 0.25N NaOH. Prepare fresh daily.

7.2.5. Secondary Working Solution, 6 mg/L: Take 0.6 mL of the Secondary Cyanide Stock Solution (Sec. 7.2.3) and dilute to a final volume of 100ml with 0.25N NaOH. Prepare fresh daily.

7.2.5.1. Secondary Working Solution for Easy-Dist: 10mg/L: Take 1.0 mL of the Secondary Cyanide Stock Solution (Sec. 7.2.3) and dilute to a final volume of 100ml with 0.25N NaOH. Prepare fresh daily.

7.2.6. Complex Cyanide stock solution (Potassium Ferrocyanide Trihydrate in 0.5 NAOH), 1000 mg/L: Purchased commercially from ERA (catalog number 998). Follow manufacturer's instructions for storage and stability. This

standard is used for the preparation of QC samples which are distilled prior to analysis (LCS and MS/MSD).

7.2.7. Complex Cyanide Working solution, 6 mg/L: Take 0.6 mL of the Complex Cyanide stock solution (Sec 7.2.6) and dilute to a final volume of 100 ml with 0.25N NaOH. Prepare fresh daily.

7.2.7.1. Complex Cyanide Working solution for Easy-Dist, 10 mg/L: Take 1.0 mL of the Complex Cyanide stock solution (Sec 7.2.6) and dilute to a final volume of 100 ml with 0.25N NaOH. Prepare fresh daily.

7.2.8. Soil Standard Reference Materia (LCSSRM): Purchased commercially from ERA or Phenova. Follow manufacturer's instructions for storage and stability. This standard is used for the preparation of QC samples which are distilled prior to analysis.

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	Glass or plastic	200 ml	NaOH, pH > 12; Cool 4 ± 2°C	14 Days	Method 335.4; SW846 Method 9012B
Soils	Glass	5.0 grams	Cool 4 ± 2°C	14 Days	Method 335.4; SW846 Method 9012B

[1] Inclusive of digestion and analysis.

9. Quality Control

9.1. Sample QC: Waters and soils, are separated into different QC batches.

9.1.1. Method Blank (MB): 0.25N NaOH is used for the blank and is carried through the entire sample preparation and analytical process. Add 6ml of 0.25 N NaOH to a disposable distillation sample tube. Results must be less than the reporting limit, or the batch must be redistilled and reanalyzed. One method blank must be analyzed with each set up of samples or every 20 samples, whichever is more frequent.

9.1.2. Laboratory Control Sample (LCS) 0.10 mg/L: A LCS (aqueous batches) or LCSSRM (soil batches) must be analyzed with each distillation batch or every

20 samples, whichever is more frequent. Prepare the solution fresh daily by adding 0.1 mL of the 6 mg/L Complex CN working solution (Sec. 7.2.7) and 5mL of 0.25 NaOH. The results must be within the control limits; if not, the batch must be redistilled and reanalyzed. Control limits: for 9012B ±15% of the true value; for 335.4 ±10% of the true value; and for SM4500CN ±10% of the true value.

- *Soil - Laboratory Control Sample Standard Reference Material (LCSSRM),* weigh out 0.25g of the standard reference material, add 5.0 ml DI water to the disposable distillation tube. The results must be within vendor specified QC limits or the batch must be redistilled and reanalyzed.

9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD) One MS/MSD pair must be analyzed for every 10 samples.

- Aqueous samples (drinking water and wastewater): **MICRO DIST, 0.2 mg/L:** two portions of the same sample (matrix spike and matrix spike duplicate), each 6 ml, are added to separate disposable distillation tubes then add 0.2 mL of the 6 mg/L Complex CN primary working solution (Sec 7.2.7) into each sample. The results must be within 90-110 %..

- Soil samples: **MICRO DIST, 4.8 mg/Kg:** add 0.25g of the sample to two separate disposable distillation tubes (matrix spike and matrix spike duplicate) and add 5.0 ml of deionized water. Spike these samples with 0.2 mL of the 6 mg/L Complex CN primary working solution (Sec. 7.2.7). The results must be within laboratory generated limits.

- Amenable to chlorination: MS/MSD – For aqueous samples, measure 6 ml of sample for both the MS and MSD and add to a 40ml glass vial. Add 0.2 ml of the 6mg/L Complex CN primary working solution (Sec 7.2.7) into each sample; the spiking concentration is 0.20mg/L. For soils samples, weigh out 0.25g of sample and add 5.0ml of water then follow spiking procedure for aqueous samples. The spike must be added before the chlorination step begins. The form of cyanide used for spiking is completely amenable to decomposition with chlorine and therefore, the recovery of the spikes is expected to be 0%. If cyanide remains following the chlorination step, it is evident that the chlorination was not complete.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV), 0.20 ppm: The ICV must be analyzed immediately after an acceptable calibration and before any samples are analyzed. The ICV will be prepared from the secondary Simple CN source (Sec 7.2.3). See Sec 10.2.3.3 for the ICV preparation instructions by Micro Dist. The determined concentration must be within the ICV control limits; if not the analysis should be terminated until the source of the problem is identified and corrected. ICV control limits for 9012B is ±15% of the true

value; for 335.4 ±10% of the true value and for SM4500CN ±15% of the true value.

9.2.2. Continuing Calibration Verification (CCV), 0.20 ppm: A CCV is analyzed after every 10 samples and at the end of the sample run. The CCV will be prepared from the same source as the ICAL. CCV control limits for 9012B is ±15% of the true value; for 335.4 ±10% of the true value and for SM4500CN ±10% of the true value. If the result exceeds the limit, the analysis should be terminated until the source of the problem is identified and corrected and all samples following the last acceptable CCV must be reanalyzed.

9.2.3. Initial Calibration Blank (ICB): An ICB is analyzed immediately after the ICV to verify calibration and acceptable instrument performance. The results must be less than the reporting limit. Use 0.25N NaOH for the ICB.

9.2.4. Continuing Calibration Blank (CCB): A CCB must be analyzed after every 10 samples and at the end of the sample run. The results must be less than the reporting limit. If the CCB result exceeds the reporting limit, all samples following the last acceptable CCB must be reanalyzed. Use 0.25N NaOH for the CCB.

9.2.5. Minimum Reporting Level (MRL), 0.01 mg/L: The MRL is required for 335.4 Drinking water samples only. The MRL must be analyzed immediately after the ICV/ICB pair and before any samples are analyzed. The MRL is prepared in the same manner as the low level standard. Add 5ml of 0.25N NaOH solution to the sample tube, then add 0.01ml of 6ppm Primary Standard (Sec. 7.2.4). Follow procedure for sample prep in Sec. 10.1.3. The recovery for the MRL should be within 50-150% of the true value. If not, re-analyze the entire batch (see exception note below). If the MRL is biased high, non-detect (ND) samples can be reported with flags and add NCM in report.

Note: For Method 335.4, if MRL fails and samples are not drinking water samples, place an 'x' in front of the MRL when data is uploaded into TALS; the MRL will not be reported with the non-drinking water samples.

10. Procedure

10.1. Sample Preparation

10.1.1. Each sample matrix must be spot tested for the presence of interfering ions.

10.1.1.1. Check that pH of each sample is >12 using the pH indicator strip.

10.1.1.2. Check for the presence of chlorine using KI-Starch paper. See section 4.3.1.

10.1.1.3. Check for the presence of sulfide using lead acetate paper (pre-moistened with pH4 acetate buffer solution). See section 4.2.1. – 4.2.4. Method 9012B: If positive add bismuth nitrate to the sample. At the same time add bismuth nitrate to all standards before distillation, see Section 10.2.3.

10.1.2. Procedure for the pretreatment of cyanides amenable to chlorination. **Note**: If total cyanide is detected, the laboratory would need to analyze both the cyanide amenable to chlorination sample and to re-perform (repeat) the total cyanide sample that was originally analyzed through the entire procedure.

10.1.2.1. Divide the sample into two equal aliquots of 6 ml sample and chlorinate one for the determination of cyanide amenable to chlorination. For aqueous samples, measure the 6 ml of sample and add into a 40 ml glass vial. For soils samples, weigh out 0.25g of sample and add 5ml of water. Add calcium hypochlorite solution (Section 7.1.6.1) drop-wise while agitating the mixture using a magnetic stir plate and maintaining the pH at between 11 and 12 with sodium hydroxide solution (Section 7.1.6.2). Cover the vials with aluminum foil to protect the samples from the light.

Note: A MS/MSD must be performed for samples requiring cyanide amenable to chlorination as a negative quality control; see Sec 9.1.3.

CAUTION: Toxic cyanogen chloride is the initial reaction product during an alkaline chlorination. This reaction should take place in a fume hood.

10.1.2.2. Excess residual chlorine must be maintained in the solution for 1 hour while agitating. Monitor pH with pH test paper and chlorine with KI-starch paper. A distinct blue color on the paper indicates sufficient chlorine in solution. If necessary add more calcium hypochlorite and/or NaOH.

10.1.2.3. Following one hour, remove any residual chlorine by dropwise addition of NaAsO2 solution (Sec. 7.1.6.3). Test with KI starch paper to ensure that there is no residual chlorine present as a reducing agent..

10.1.2.4. Test for total cyanide in both the chlorinated and unchlorinated aliquot of the sample. Cyanide amenable to chlorination is determined by the difference between the total cyanide result of the un-chlorinated aliquot and the chlorinated aliquot.

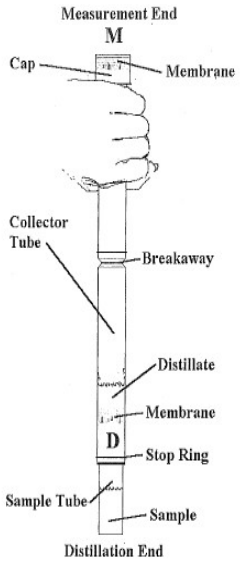
10.1.3. <u>MICRO DIST Distillation Procedure</u>

10.1.3.1. Set the controller to 120 deg C. Allow the block to warm up which takes approximately 40 minutes.

10.1.3.2. With the 'M' end or 'measuring end' up, place the collector tubes for the samples into the tube rack. Add 2 mL of 0.25N NaOH solution and add a membrane and cap. Label the top of the tube with the sample ID. Up to 21 samples tubes can fit on one block.

10.1.3.3. Pipet 6.0ml of aqueous sample into each sample tube or weigh out 0.25 to 0.30 grams of sample plus 5.0 ml of DI water for soil samples. Add 0.25ml of the sulfamic acid solution and 0.75ml of the releasing agent to all tubes. Label the tube with the sample ID.

***NOTE**: Homogenize soil samples properly prior to taking an aliquot for analysis.



10.1.3.4. Immediately push the 'D' end or 'distillation or discarded' end of the collector tube over the open end of each sample tube to start the seal.

10.1.3.5. Place the assembly into the press, putting the sample tube through the hole in the white base. Before pressing, the user should hold the collector tube firmly at the breakaway point to ensure the tube does not shift during the pressing procedure.

10.1.3.6. Press down on the press handle until the stop ring on the sample tube hits the 'D' end of the collector tube.

10.1.3.7. Put on heat resistant gloves and push the sample tube and 'D' end of each tube into the preheated block so that the collector tube stop ring touches the block.

10.1.3.8. Set timer for 30 minutes. Do not pull the tubes out before the 30 minutes to check them. This will cause the sample to suck-back.

10.1.3.9. After the 30 minutes have passed, put on heat resistant gloves. Remove the first tube from the block and immediately pull off its sample tube using a downward, twisting motion. Important: Remove the sample tube within 4 seconds of removing it from the block or suck-back of the sample will occur, which may cause low recoveries. Dispose of the sample tube and remaining hot solution by dropping it into the sink or waste bucket.

Note: Do not pull out all the samples at once. Each sample needs to be separated immediately upon removal from the block.

10.1.3.10. Place the tubes in the collector tube rack with the 'M' end up. Parafilm the 'D' end of the tube and allow the tubes to cool for at least 10 minutes.

10.1.3.11. For each collector tube, hold the tube horizontally and rinse the walls with the distillate in order to homogenize it. Slowly roll the distillate around the tube to gather any droplets clinging to the walls. Then slowly return the collector tube to an upright position with the 'D' end up.

10.1.3.12. With the 'D' end up, break the collector tube in half by pulling the 'D' end hard towards the user to break it and twisting and tearing off the 'D' end. Rinse the 'D' end with a small volume of DI water and pour into the 'M' end. The 'D' end can be discarded.

10.1.3.13. Do not invert the sample. With the 'M' end down, place the tube in the collector tube rack. Dilute to the mark with deionized water. Seal the open end with parafilm if the sample is not going to be analyzed immediately.

10.1.3.14. The solution is now ready for semi-automated colorimetry.

10.1.4. <u>MICRO DIST Distillation Procedure using reuse, preclean Microdistillation tube</u>

10.1.4.1. With the D end up, place the collector tubes in the tube rack.

10.1.4.2. In the sample tube, pipet 6.0ml of aqueous sample into each sample tube or weigh out 0.25 to 0.30 grams of sample plus 5.0 ml of DI water for soil samples. Add 0.25ml of the sulfamic acid solution and 0.75ml of the releasing agent to all tubes. Label the tube with the sample ID.

10.1.4.3. Place the sample tube into the press, putting the sample tube through the whole in the white base. Place the D end on top of the sample tube and press until the D end meets the ring around the sample tube.

10.1.4.4. Add 2 mL of 0.25N NaOH solution to the D end which is connected to the with sample tube.

10.1.4.5. Attach a silicone tubing about 1.5" long to the tube labeled M end (labeled with sample ID). Then connect the tube labeled M end to the tube labeled D end (which is connected to sample tube).

10.1.4.6. Put on heat resistant gloves and push the sample tube and 'D' end of each tube into the preheated block so that the collector tube stop ring touches the block.

10.1.4.7. Set timer for 30 minutes. Do not pull the tubes out before the 30 minutes to check them. This will cause the sample to suck-back.

10.1.4.8. After the 30 minutes have passed, put on heat resistant gloves. Remove the first tube from the block and immediately flip the tube upside down and gently tap the tube on the counter a couple times. Remove the D end connected to sample tube. Rinse the D end/sample tube with few drops of DI water and add to the M end tube. The D end/sample tube can now be placed in a bucket for cleaning, refer to SOP ED-GEN-013, Glassware cleaning, for the full procedure.

10.1.4.9. Place the tubes in the collector tube rack with the 'M' end up.

10.1.4.10. Dilute to the mark with deionized water. The solution is now ready for semi-automated colorimetry. Seal the open end with parafilm, if the sample is not going to be analyzed immediately.

10.2. Calibration

10.2.1. All standards (Calibration Standards, calibration blank, ICV/CCV) must be distilled in the same manner as samples. A calibration must be analyzed each day before sample analysis.

10.2.2. Preparation of Standards for samples without sulfide:

10.2.2.1. Use the primary working solution (Sec. 7.2.4) to prepare the calibration standards below.

10.2.2.2. Prepare calibration standards and CCV for Micro Distillation batch as follows; the final volume in collector tube is 6.0 ml.

Volume (mL) of 6 mg/L CN primary working solution	Volume (mL) of 0.25N NaOH added to each Distillation Sample Tube	Concentration CN-(mg/L)
0.40	5	0.40
0.20	5	0.20
0.10	5	0.10
0.05	5	0.05
0.025	5	0.025
0.010	5	0.010
0	5	0.0
CCV	5	0.20

Note: [1] Add a calibration standard of 0.005 CN mg/l as directed on special projects.
[2] Prepare CCV with each distillation batch.
[3] Due to limited volume of the distillate, it may be necessary to prepare and distill two or three CCVs in the batch.

10.2.2.3. Prepare ICV using a secondary Simple CN source (Sec 7.2.3) as follows:

Volume (mL) of 6 mg/L CN secondary working solution (ICV)	Volume (mL) of 0.25N NaOH added to each Distillation Sample Tube	Concentration CN-(mg/L)
0.20	5	0.20

10.2.2.4. Correlation coefficient of 0.995 or greater must be obtained.

10.2.2.5. After running the calibration, recalculate each of the standard's concentration. Review Table 1 of the instrument report specifically the *%Residual* and *Det. Conc. (mg N/L)* column for the resulting concentration. The % Residual of the calibration standards should be within 50% for the first calibration standard, 0.01 mg/L (1 to 2 times the RL), 20% for 0.025 mg/L and 0.050 mg/L standards (3 to 5 times the RL) and 10% the rest of the standards (greater than 5 times the RL). If this criteria is not met, re-analyze the calibration standards (in some cases, new calibration standards may need to be prepared).

10.2.3. Method 9012B: Preparation of Standards for samples with sulfide:

10.2.3.1. From the 6 mg/L primary working standard prepare five standards as above to be distilled in the same manner as the samples using the method of standard additions. Note: Bismuth Nitrate must also be added to the standards. Standards distilled in this way will give a linear curve at lower concentrations but as concentration increased the recovery decreases.

10.2.3.2. Correlation coefficient of 0.995 or greater must be obtained.

10.3. Sample Analysis

10.3.1. System startup:

10.3.1.1. Follow manifold scheme for this method.

10.3.1.2. Turn on heating unit at 60° C for 15 minutes before analysis.

10.3.1.3. Put all reagent lines into deionized water and turn pump on. Check for leaks and slow flow rate.

10.3.1.4. Switch over reagent lines to proper reagent. Let reagent run through manifold for 15 minutes before analysis to allow baseline to stabilize.

10.3.2. Set up tray table with all appropriate QC checks. CCV/CCB every 10 samples and at the end of the run. The following analytical sequence must be used:

Instrument Calibration
ICV (QCS)
ICB
MRL
CCV
CCB
MB
LCS or LCSSRM for soils
8 Samples with Analytical Spikes
CCV (IPC)
CCB
10 samples
CCV (IPC)
CCB
Repeat until run is complete
CCV (IPC)
CCB

10.3.3. Place standards and samples into autosampler cups.

10.3.4. Run tray.

10.3.5. Dilute and reanalyze samples that exceed the calibration curve using the distilled calibration blank or CCB.

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{\text{(spiked sample) - (unspiked sample)}}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Final results calculation in aqueous samples:

Concentration = mg/L

Concentration (mg/L) from instrument is imported to TALS
Samples with dilution factor are calculated in TALS for final results

11.4. Final results calculation in solid samples:

$$\text{Concentration mg/kg} = \frac{C \times V \times D}{W}$$

Where:
C = sample concentration in extract (mg/L)
V = Volume of extract (mL)
D = Dilution Factor
W = Weight/Volume of sample aliquot extracted (grams)

NOTE: All dry weight corrections are made in TALS.

11.5. Final results calculation amenable to chlorination:

mg CN^- amenable to chlorination/L = A - B

where:
A: mg CN^-/L: found in unchlorinated portion of sample
B: mg CN^-/L: found in chlorinated portion of sample

11.6. Data Reduction

11.6.1. Cyanide soil and waters are batched separately in TALS Analyst Desktop. Update Cyanide prep batches as follows:.

11.6.1.1. For soils, enter in 0.25-0.30g under the 'initial amount' column and 6ml under the 'final amount' column for each sample including the MB and LCSSRM.

11.6.1.2. For waters, enter 6ml under the columns 'initial amount' and 'final amount' for each sample including the MB and LCS. Under the columns 'chlorinechk' and 'sulfidechk' record the results from the test strip paper. 'N' designates the interference was not present. Record 'pH>12' under the column 'DistillpHchk.'

11.6.1.3. Since the calibration standards are distilled, they will be recorded in the prep batch. Under the column 'Lab ID,' type in "IC' for 7 rows. Enter 6ml under the columns 'initial amount' and 'final amount' for each standard. Enter how many milliliters of the 6ppm primary working solution were used for each calibration standard following the chart in Sec. 10.2.2.1.

11.6.1.4. For both soil and water batches, record reagent information including reagent ID and expiration date on the batch information page. Record the actual time the samples were distilling under 'batch comments.' This should be designated as distillation start and end time.

11.6.2. Instrument data is imported to TALS by following these steps:

11.6.2.1. After the run has finished, click the data icon at the top of the screen. Choose the correct file. Go to 'File" and choose "Export Data' from the drop down menu. A screen will pop up, click 'ok' and then another screen will pop up, click 'ok.'

11.6.3. From the desktop, open the 'LIMSdata' icon on the Lachat 8000 and the 'Exports' icon on the Lachat 8500. Select the correct file, and then right click. Go to 'Send to-> LimsImport ->' Choose the correct method. The data has now been imported to TALS.

11.6.4. All reagent information is recorded in the "Batch Information" page. Use the 'Worksheet tab' if additional pages are necessary.

11.6.5. Record special notes and observations in the "worksheet" tab (i.e. sample appearance and notes on why samples were rejected or diluted).

11.6.6. All raw data is attached as a pdf file. The raw data includes the instrument report and calibration curve.

11.6.7. Complete the Data Review Checker (DRC) in TALS: Prior to data submission the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch (TALS Analyst Desktop II module).

11.6.7.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.6.7.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.6.7.3. Acknowledge by filling in responses to all unacknowledged findings. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.6.7.4. Fill in appropriate comments in the response box, then hit 'OK.' 'The column labeled 'unacknowledged findings' should show '0' for all questions once completed; then batch is ready for 2nd level review.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal

restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Pyridine/Hydroxide Solution: Hazardous Waste Liquid, n.o.s. This material is collected at satellite accumulation in 30 gallon Poly containers. When the container is full, the container is transferred to the waste room and held in secondary containment until a waste vendor is called for a pick up.

  Teris Profile Number: 50016717
  Onyx Profile WIP Number: 584650

15.0. References / Cross-References

15.1. Methods for the Determination of Inorganic Substances in Environmental Samples, EMSL-Cincinnati, EPA/600/R-93/100, August 1993, Method - 335.4 Determination of Total Cyanide by Semi-Automated Colorimetry

15.2. Standard Methods for the Examination of Water and Wastewater, American Public Health Association, Washington, DC, Method 4500 CN C, G-2016.

15.3. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW846 Method 9012B.

15.4. QuikChem Method 10-204-00-1-A, Determination of Cyanide in Waters.

15.5. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.6. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.7. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.8. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

15.9. TestAmerica Edison SOP No. ED-GEN-013, *Glassware cleaning*, current revision.

16.0. Method Modifications:

Item	Method no.	Modification
1	335.4	Sec 4.3.1: This SOP referenced method 335.4 (not SW846) when treating samples suspected to contain chlorine and nitrates/nitrites.

2	QuikChem Method 10-204-001-41-A	Sec 7.1.8.3: Preparation procedure for the Phosphate buffer follows Quikchem method 10-204-001-41-1 (not Method 335.4).
3	SM 4500 CN^- C	Sec 10.1.3.3: Sulfamic acid solution is added to all samples during distillation. Addition of Sulfamic acid is a procedure described in Standard methods 18th ed, Method 4500 CN^- C. Methods SW846 9012B and EPA 335.4 require the addition of Sulfamic acid for samples which are known to contain NO3 and/or NO2.
5	335.4; SW9012B	Sec 7.1.7.2-7.1.7.3; 10.1.4.2; 10.1.4.3- Modified procedure for reagent preparation and amount of reagent added in sample while keeping the reagent sample volume ratio and concentration the same.
6	335.4; SW9012B	*335.4 & 9012B: It is recommended that at least two standards (a high and a low) be distilled and compared to similar values on the curve to ensure that the distillation technique is reliable.* Sec 9.1.2: For Micro Distillation, one LCS (0.10 mg/L) will be prepared for each distillation batch; however calibration standards, ICV and CCV will be distilled in the same manner as the samples.
7	335.4; SW9012B	*Method 335.4; SW9012B: Distill standards for samples with Sulfide.* This SOP follows the procedure described in the Hach Micro Dist User Manual, Method Number 10-204-00-1-X when distilling samples by Micro Dist- *It is imperative that the standards be distilled with the samples.*
8	Micro Dist (Method No. 10-204-00-1-X)	Add between 0.50-1.0g of soil/sludge to 5 ml DI water. This SOP will use 0.25g of soil sample to 5 ml DI water. Improved accuracy with the soil LCS when using 0.25g rather than 0.50g sample weight.

17.0. Attachments

N/A

18.0. Revision History

- Revision 18, dated 08 November 2023
  - Sec. 10.1.3.12 & 10.1.4.8: Revised solution to use when rinsing the CN sample tube after distillation from 0.25N NaOH to DI water.
  - Sec. 10.2.2.5: Added criteria to use when evaluating the calibration curve.

- Revision 17, dated 23 February 2023
  - Sec. 9.2.5: Clarified the applicable matrix, procedure, and corrective action for the QC – MRL.

- Revision 16, dated 22 September 2022
  - Sec. 7.1.6.3 and 10.1.2.3: Replaced ascorbic acid with sodium arsenite to remove residual chlorine.
  - Sec. 9.2.5: Fixed MRL acceptance range to 50-150%.
  - Sec. 10.3.2: Revised instrument tray set up to reflect actual laboratory practices.
  - Sec 15: Revised Standard Method reference to Rev 2016.

- Revision 15, dated 06 January 2021
  - Sec 7.1.6.1: Clarified the stability information for the reagent $Ca(OCl)_2$.

- Revision 14, dated 16 December 2020
  - Sec 3: Revised the Definitions locations as per the most current LQM.
  - Deleted all reference to Easy-Dist (midi-dist) and wipes distillation, not applicable in this SOP.
  - Sec. 7.1.6.1: Revised Calcium Hypochlorite storage requirements and preparation frequency to daily.
  - Sec. 9.1.2: Clarified that only soil LCS should be designated LCSSRM in TALS sample worksheet.
  - 9.1.3: Clarified the MS/MSD procedure for the Cyanide amenable to chlorination.
  - Sec 9.2.5: Added MRL to the list of instrument QC.
  - Sec. 10.1.2: Added requirements that sample which is detected for total cyanide must be re-analyzed and prepared with the cyanide amenable to chlorination sample.
  - Sec 10.1.2.1 & 10.1.2.2: Revised to clarify the procedures for CN amenable to chlorination (i.e. taking identical samples for total and amenable samples; 'excess' added in Sec 10.1.2.2).
  - Sec 10.1.4: Added procedure for setting up distillation using the reuse, preclean distillation tubes.
  - Sec. 11.6.7: Replaced EDS-WI-008, Wetchem Data Review Checklist with TALS DRC; procedures added for DRC.
  - Sec 15.9: Deleted WI EDS-WI-008, not applicable; subsequent sections adjusted.
  - Sec 15.10: Added reference – SOP ED-GEN-013.

- Revision 13.1, dated 23 October 2017
  - Updated SOP header with Eurofins emblem.

- Revision 13, dated 23 October 2017
  - Sec. 9.1.2: Corrected preparation instructions for LCS from 0.20 mg/L to 0.10 mg/L. Updated control limits for Method 9012B to +/-15%.
  - Sec. 9.2.1: Updated the ICV control limits for Method 9012B.
  - Sec. 9.2.2: Updated the CCV control limits for Method 9012B; removed instructions to prepare a CCV with each Micro-Distillation batch.
  - Sec. 10.3.2: Updated the example analytical sequence for Micro Dist and Easy Dist.
  - Sec 16, Item 6 of Method Modification – Deleted text- CCV (0.20 mg/L) will be distilled with each distillation batch.

- Revision 12, dated 26 September 2017
  - Sec. 1.1.2: Updated the Soil RL to 0.24 mg/Kg to reflect the initial soil sample weight change from 0.5g to 0.25g.
  - Sec. 6.2: Added Lachat Micro Distillation tubes and pH test strips to list of supplies.
  - Sec. 7.1.7.2: Revised preparation instructions for sulfamic acid solution; revised to use a double strength concentration sulfamic acid solution, however the amount added in sample is reduced in half.
  - Sec. 7.1.7.3: Replaced 2.5M MgCl2 and 1:1 H2SO4 solution with Microdistillation releasing agent (7.11 M sulfuric acid/ 0.79M magnesium

chloride) to be used instead of the separate H2SO4 and MgCl2 solution; equivalent reagent concentration is added in sample.
  - Sec. 7.2.3: Added ERA cat# 997 as an alternative source for a Simple CN secondary standard.
  - Sec 7.2.4 & 7.2.5: Added preparation instructions for the Micro Dist Primary/Secondary Working standards.
  - Sec 7.2.6: Deleted LCSH
  - Sec 7.2.7: Updated preparation instructions for the Complex CN Working Solution (Micro Dist).
  - Sec 7.2.8: Added soil standard reference material.
  - Sec 9.1.1: Updated preparation instructions for MB from deionized water to 0.25 N NaOH to reflect actual laboratory practices.
  - Sec 9.1.2: Updated preparation instructions for LCS needed for Micro-Dist; removed LCSH prep for Micro Distillation. Revised the LCS sample weight to use from 0.5g to 0.25g. Added Easy Dist prep instructions for LCSSRM.
  - Sec 9.1.3: Updated the matrix spike preparation instructions for Micro Dist for soil and water.
  - Sec 9.2.1: Updated the ICV preparation instructions for Micro-Dist;
  - Sec 9.2.2: Revised the standard source used for preparing CCV; source will be the same source as the calibration standards and will be distilled with each Micro Dist distillation batch.
  - Sec 10.1.1.1: Added instructions to check pH of each sample.
  - Sec 10.1.4.2: Revised the volume to add in collector tube to 2.0ml of 0.25N NaOH.
  - Sec 10.1.4.3: Revised the reagents and amount of reagents added into the sample/distillation tube; Revised the initial weight used for solid samples from 0.5g to 0.25-0.30g. Added note to homogenize soil sample prior to taking aliquot.
  - Sec 10.1.4.12: Added instructions to rinse the D-end and add rinsate to the M-end.
  - Sec 10.2.2: Added instructions to distill all standards used for the analysis of CN by Micro Distillation.
  - Sec 10.2.3.2 & 10.2.3.3: Added preparation instructions for the calibration standards and check standards (ICV,CCV) by Micro Distillation. Added note that CCV must be distilled with each distillation batch.
  - Sec 10.3.2: Clarified analytical sequence for Micro Dist and Easy Dist.
  - Sec 10.3.5: Updated instructions for sample dilutions.
  - Sec 11.7.1: Added instructions for completing Batch information page in TALS.
  - Sec 16: Method modification items 5-8 added.

- Revision 11, dated 11 August 2014
  - Sec. 7.2.2 & 7.2.3: Clarified procedure as to when to apply the standardized value of the CN stock solution.
  - Sec. 9.1.2: LLCS and HLCS will be analyzed for both methods SW9012 and EPA335.4 Deleted text: *'Distillation of two LCSs (LLCS and HLCS) is not a method requirement for Method 335.4. Use HLCS as the QC sample for Method 335.4.*

- Revision 10, dated 12 March 2014
  - Deleted Method 9012A reference throughout the SOP; replaced SW method reference with 9012B.

- Sec 15.3 Added reference to Standard Methods Editorial revision 2011; subsequent sections adjusted accordingly.

- Revision 9, dated 12 November 2012
  - Sec 1.1.2: Revised RL for solids from 0.12 mg/kg to 0.10 mg/kg; sample final volume is 5 ml.
  - Sec 9.1.2 & 9.1.3: Deleted text: *After micro-distillation, volume is brought up to 6 ml with deionized water.* The final volume of the distillate is not adjusted.
  - Deleted Sec 10.1.4.13, final distillate will not to be diluted to 6 ml; subsequent sections adjusted accordingly.
  - Sec 10.1.4.13 (formerly Sec 10.1.4.14): deleted text: *Shake the tube with a gentle whipping motion to mix in diluent water.* Added text: *With the 'M' end down, place the tube in the collector tube rack.*

- Revision 8, dated 25 October 2012
  - Deleted Sec 7.2.4 (5 mg/L primary working solution), standard not applicable; subsequent sections adjusted accordingly.
  - Sec 7.2.2, 7.2.3 & 7.2.6: Clarified to include which QC samples these standards are applicable to.
  - Sec 7.2.6 & 7.2.7: Added complex cyanide standard, 1000 mg/L stock standard and 10 mg/L working solution to be used for distilled QC samples (LCS and MS/MSD).
  - Sec 9.1.2: Added note: *Distillaton of two LCSs (LLCS and HLCS) is not a method requirement for Method 335.4. Use HLCS as the QC sample for Method 335.4.*
  - Sec 9.1.2: Revised the LLCS concentration to 0.10 mg/L; revised to reflect the correct spiking amount (ml) and source standard (complex CN working solution) in the preparation procedure.
  - Sec 9.1.3: Revised the spiking solution from simple to complex CN.

- Revision 7, dated 13 September 2012
  - Sec 1: Revised to include the RL for microdistillation in soils.
  - Sec. 2.0: Revised to include 'MICRO DIST' as one of the ways to distill samples.
  - Sec. 6.0: Updated list of equipment (added MICRO DIST and Lachat 8000 and 8500 series).
  - Sec. 9.1.1: Changed the frequency for the Method Blank analysis from 1 per 10 samples to 1 per 20 samples.
  - Sec. 9.1.2: Changed the frequency for the LLCS/HLCS analysis from 1 per 10 samples to 1 per 20 samples. Also, included the micro distillation preparation procedure for LLCS/HLCS.
  - Sec. 9.1.3: Clarified the requirement of one MS/MSD per 10 samples. Also, included the micro distillation preparation procedure for MS/MSD.
  - Sec. 10.1.4: Added procedure for micro distillation of samples.

- Revision 6, dated 22 March 2012
  - Sec. 1.1.1: added method 4500 CN G to the list of methods.
  - Sec 1.1.3, 12.1 & 12.2: Revised QAM references to reflect the most recent QAM revision.
  - Sec. 7.2.2/7.2.3: Added information on commercially purchased CN stock standard.

- Sec 9.1.3: Added MS/MSD requirement for cyanide amenable to chlorination under sample QC.
- Sec. 10.1.2.1: Added prep for soil samples requiring cyanide amenable to chlorination.
- Sec 11.6: Added calculation formula for CN amenable to chlorination; subsequent sections adjusted accordingly.
- Sec 15.9: Added applicable reference.

- Revision 5, dated 22 November 2010
  - Sec. 1.1.1: Added wipes to list of applicable matrices.
  - Sec. 1.1.2: Added reporting limit for wipes.
  - Sec. 6.2: Added specimen cups, transfer pipettes, graduated cylinder and micro-burets to the list of supplies.
  - Sec. 7.1.4: Changed the amount of acetic acid added from “approximately 100 ml” to “approximately 10 drops” to reflect actual laboratory practices.
  - Sec. 7.1.6.2: Clarified reagent prep
  - Sec. 8.0: Added wipes to section.
  - Sec. 9.1: Added sample QC requirements for wipe batches.
  - Sec. 9.1.1: Included the prep of the method blank for wipe batches
  - Sec. 9.1.2: Revised the prep of the LLCS and HLCS to reflect actual laboratory practices. Also, included prep of the LCS/LCSD for wipe batches.
  - Sec. 9.1.3: Revised MS/MSD prep for aqueous and soil.
  - Sec. 9.2.2: Deleted text “If the CCV is not within the control limits, a second analysis should be performed.”
  - Sec 9.2.3 & 9.2.4: Clarified the type of solution utilized for the ICB and CCB.
  - Sec. 10.1.3.1: Revised step of the distillation procedure to reflect actual laboratory practices.
  - Sec. 10.2.2.1: Revised calibration standards prep Table.
  - Sec. 10.3.2: Added comment on how to set up the analytical sequence for wipe samples.
  - Sec. 11.5: Included calculation for wipe samples.
  - Sec. 11.6: Added steps to import data to TALS.
  - Section 16: Added method revision for addition of wipe matrix to SW846 9012A/9012B.

- Revision 4, dated 28 September 2009
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Incorporated SOP ED-WET-007 (Analysis of Total Cyanide in Drinking water-Automated, Method 335.4) into this SOP; retired SOP ED-WET-007 at the effective date of this SOP.
  - Sec. 7.2.2: Added information for the standardization of the Cyanide Stock. Replace frequency of the standardization from monthly to weekly to comply with Standard methods.
  - Sec 7.2.3 – 7.2.6: Added standards and preparation instructions.
  - Sec 9.1.3: Clarified % recovery limits for water and solid samples.
  - Sec 9.2.1 & 9.2.2: Clarified corrective actions to be taken if QCS/ICV and IPC/CCV results are outside the acceptable limits.
  - Sec 10.3.2: Revised sample analytical sequence to comply with method 335.4.
  - Sec. 11.5: Revised new data reduction procedures in accordance with new TALS.

- Sec. 15: Replace reference 335.2 and 335.3 with the EPA 335.4 reference; added applicable reference.
- Sec 16: Added Method modification to specify actual source of reagent information.

- Revision 3b, dated 13 July 2009
  - Sec 2.1: .For each batch of ten samples or less, a matrix spike and matrix spike duplicate will be distilled and analyzed. (Replaced 20 samples to 10 samples per batch).
  - Sec 9.5.1.3 & 9.5.2.2.: Revised Correlation coefficient from 0.997 to 0.995 as per corporate SOP on Calibration curves.
  - Sec 10.3: Up to 10 samples can be put in a batch. .Revised number of samples in a batch from 20 to 10 samples.

- Revision 3a, dated 13 March 2009
  - Sec 1.1: Delete Methods 335.1/.3; Add Method Lachat 10-204-00-1-A (Methods deleted in response to MUR).
  - Sec 3.6: Delete text: If this occurs, a Dow Corning 544 can be added to prevent the foam from collecting in the condenser. Replace the above procedure with: *If this occurs, samples can be acidified with acetic acid (1.6M) to pH 6.0-7.0.*
  - Sec 5.2: Add: Solution is stable for 6 months or when QC check is outside the acceptable limits, whichever comes first, store at 4ºC.
  - Sec 5.4: Acetate Buffer-Dissolve 82.0g of NaC2H3O2 x 3 H2O in 100ml DI water. Add glacial acetic acid drop by drop until the pH is 4.5. This solution is stable for 6 months, store at 4ºC. (Changed amount of acetic acid added to reflect actual laboratory practices).
  - Sec 5.6: Remove: Dow Corning 544 antifoam agent. Lab does not use this reagent , samples will be acidified with acetic acid.
  - Sec. 5.7.1, 5.8.2, 5.8.3, 5.8.4: Add: Solution is stable for 6 months, store at room temperature.
  - Sec 5.7.2: 0.25M NaOH. Add: Dissolve 10.0g NaOH into 1L deionized water. Solution is stable for 6 months, store at room temperature.
  - Sec. 5.9.1 & 5.9.2: Add: Store at room temperature.
  - Sec. 5.9.3: Phosphate buffer: In a 1L volumetric flask, dissolve 97g of potassium phosphate, monobasic, anhydrous (KH2PO4) in approximately 800ml of deionized water. Dilute to the mark. Solution is stable for one month. Store at room temperature. (Changed reagent preparation to comply with the Lachat method).
  - Sec 6.3: Add: Laboratory Control Sample (LCS): Prepare a 5ppm and 10ppm CN standard by diluting the 1000ppm Primary CN stock. Prepare fresh daily.
    - LLCS: Take 0.25ml of the 5ppm CN standard and bring up to a 50ml final volume with 0.25N NaOH. The true value is 0.025ppm.
    - HLCS: Take 1.0ml of the 10ppm CN standard and bring up to a 50ml final volume with 0.25N NaOH. The true value is 0.2ppm

  - Sec 9.10: Delete text: “Free Cyanide is estimated using the weak acid dissociable procedure following the colorimetric procedure without distillation. For reference refer to SM 4500-CN-A.” Revise the above text to: “*Free Cyanide will be treated following the procedure for the Cyanide Amenable to chlorination and therefore must be distilled.” See Section 9.2*

- Sec. 10.7: MS and MSD Recovery: Delete: The recovery must be within laboratory generated limits. Revise the above text to: *The recovery must be within 90-110% (*Revise MS/MSD recovery to comply with method 335.4).
- Sec. 15.1: References: Replace 335.2 & 335.3 reference with EPA 335.4 reference

Title: Method SW846 3060A, The Alkaline Digestion of Soil and Wipe Samples for the Analysis of Hexavalent Chromium

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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 SW846 Method 3060A is an alkaline digestion procedure for extracting Hexavalent Chromium [Cr(VI)] from soluble, adsorbed, and precipitated forms of chromium compounds in soils, sludges, sediments, and similar waste materials. Materials applicable to this method include soil, sediment, debris, brick, concrete, sludge, and precipitate. TestAmerica Edison has modified the method to include digestion of surface wipe samples. To quantify total Cr(VI) in a solid matrix, three criteria must be satisfied: 1) the extracting solution must solubilize all forms of Cr(VI), 2) the conditions of the extraction must not induce reduction of native Cr(VI) to Cr(III), and 3) the method must not cause oxidation of native Cr(III) contained in the sample to Cr(VI). Method 3060A meets these criteria for a wide spectrum of solid matrices.

1.1.2 Method 3060A digestates can be analyzed for hexavalent chromium using Method 7196 (colorimetrically by UV-VIS spectrophotometry). Reference the applicable analytical method SOP for details on reporting limits.

1.1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

2.1. This method uses an alkaline digestion to solubilize both water-insoluble (with some exceptions) and water soluble Cr(VI) compounds in solid waste samples. The pH of the digestate must be carefully adjusted during the digestion procedure. The sample is digested using 0.28M Na_2CO_3/0.5M NaOH solution and heating at 90-95ºC for 60 minutes to dissolve the Cr(VI) and stabilize it against reduction to Cr(III).

2.2. Under the alkaline conditions of the extraction, minimal reduction of Cr (VI) or oxidation of native Cr(III) occurs. The addition of Mg^{+2} in a phosphate buffer to the alkaline solution has been shown to suppress oxidation, if observed. The accuracy of the extraction procedure is assessed using spike recovery data for soluble and insoluble forms of Cr(VI) (e.g. K_2Cr2O_7 and $PbCrO_4$), coupled with measurement of ancillary soil properties, indicative of the potential for the soil to maintain a Cr(VI) spike during digestion, such as oxidation reduction potential (ORP), pH, organic matter content, ferrous iron, and sulfides. Recovery of an insoluble Cr(VI) spike can be used to assess the first two criteria, and method-induced oxidation is usually not observed except in soils high in Mn and amended with soluble Cr(III) salts or freshly precipitated $Cr(OH)_3$.

3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 When analyzing a sample digestate for total Cr (VI), the reducing/oxidizing tendency of each sample matrix can be determined at the client's request. Establishing the tendency of Cr(VI) to exist or not exist in the unspiked sample(s), assists in the interpretation of QC data for matrix spike recoveries outside conventionally accepted criteria for total metals.

4.1.1 This can be accomplished by characterization of each sample for additional analytical parameters such as pH, ferrous iron, sulfides, and Oxidation Reduction Potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD).

4.2 Certain substances, not typically found in the alkaline digests of soils, may interfere in the analytical methods for Cr(VI) following alkaline extraction if the concentrations of these interfering substances are high and the Cr(VI) concentration is low. Refer to the SOP for Method 7196A for the specific agents that may interfere with Cr(VI) quantification.

4.3 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

4.4 A sample containing a high buffering capacity may need to be digested using additional digestion solution.

4.5 It is very important to monitor the temperature of the digestion solution during the digestion to maintain a temperature of 90-95^{o}C during the entire time period.

4.6 Do not use Ghost wipe, Whatman, mixed cellulose ester (MCE) or glass fiber filters for sampling wipes because they convert Cr(VI) to Cr(III). Use either PVC filters or binderless quartz fiber filters.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials Section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.

6.0 Equipment and Supplies

6.1. Equipment

- Digestion vessel: water bath equipped with shaker (Precision, Model 50)
- pH meter: Orion benchtop or equivalent
- Vacuum filtration apparatus
- Analytical balance capable of accurate weighings to 0.01 grams

6.2. Supplies

- Class A volumetric flasks with stoppers
- Filter paper 0.45 and 0.1 um size.
- Nalgene 115-ml filter disposable unit with 0.45um filter
- Binderless quartz fiber filter, 37 or 47 mm diameter, 0.45 mm thick (for wipe analysis)
- Digestion container: 125ml Nalgene small-mouth polypropylene bottles
- Certified thermometer (NIST traceable)-capable of reading 100ºC
- Finnpipet (1-5 ml) with tips
- Graduated cylinder (class A)
- Specimen cups
- Transfer pipettes
- Wood spatulas

7. Reagents and Standards

7.1. Reagents

7.1.1. The following Reagent grade chemicals are used for all solutions required in this test. For stability information, refer to manufacturer's instructions or 2 years from date opened if no instructions are available. All reagents are stored at room temperature:

7.1.1.1. Concentrated Nitric acid
7.1.1.2. Anhydrous Sodium Carbonate
7.1.1.3. Sodium Hydroxide
7.1.1.4. Potassium Dichromate
7.1.1.5. Magnesium Chloride-$MgCl_2$ (anhydrous)
7.1.1.6. Potassium Phosphate, Dibasic
7.1.1.7. Potassium Phosphate, Monobasic

7.1.2. Phosphate Buffer- 0.5M K_2HPO_4/0.5M KH_2PO_4 buffer at pH 7: Dissolve 87.09g K_2HPO_4 and 68.04g KH_2PO_4 into 700ml reagent water. Bring up to a final volume of 1 liter. Store at room temperature. Stable for 6 months.

7.1.3. Digestion Solution: Weigh 20g of NaOH and 30g of Na_2CO_3 and dissolve in a 1 liter volumetric flask using Type I water. Dilute to the mark and then transfer the solution to a tightly capped polyethylene bottle. Store at room temperature. Prepare fresh monthly. **Note**: Check the pH of the digestion solution to be certain it has a pH of greater than 11.5 before using it. If the pH drops below 11.5, a new solution must be prepared.

7.1.4. 1:1 HNO3: Add 500ml DI to a 1 Liter volumetric flask and then slowly add 500ml of concentrated HNO3. Solution is stable for 6 months. Store at room temperature.

7.2. Standards

7.2.1. Potassium Dichromate Primary Standard Solution (1000 mg/L Cr+6):

Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3093-01 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. Store at room temperature. Solution is stable for 6 months.

7.2.2. Potassium Dichromate Secondary Standard Solution (1000 mg/L Cr+6): Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3090-04 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. **Note**: Prepare the Secondary standard solution from a different source than what was used to prepare the primary standard solution. Stable for 6 months, store at room temperature.

7.2.3. Hexavalent Chromium Primary Spiking Solution (100 mg/L Cr+6) Prepare by adding 10 ml of 1000 mg/L Cr+6 primary standard (Section 7.2.1) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.4. Hexavalent Chromium Secondary Spiking Solution (100 mg/L Cr+6) Prepare by adding 10 ml of 1000 mg/L Cr+6 Secondary standard (Section 7.2.2) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.5. Lead Chromate-AR Grade; for storage and stability information, refer to manufacturer's instructions

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container[1]	Min. Sample Size	Preservation	Holding Time[3]	Reference
Soils	Glass or plastic	2.50g[2]	Cool 4 ±2ºC	30 Days	SW 846 Method 3060A
Wipes[4]	Glass or plastic	1 wipe	Cool 4 ±2ºC	30 Days	SW 846 Method 3060A

[1] Containers should not contain stainless steel
[2]Additional volume may be required if the sample is chosen for QC or if a re-prep is required.
[3]The alkaline digestate must be analyzed within 168 hours after extraction from the soil or wipe.
[4]In chrome plating environments, wipe samples taken on a PVC filter or an uncoated binderless quartz fiber filter, should be placed in a vial containing 5 mL of an aqueous solution containing 10% Na_2CO_3 with 2% $NaHCO_3$ immediately after sampling to eliminate the interference from the acid used in the chrome plating process. An alternate medium

which does not require extraction in the field is a binderless quartz fiber filter coated with 1% NaOH.

9. Quality Control

9.1. Sample QC - The following quality control samples are prepared daily with each batch of 20 samples or less, whichever is more frequent. Soil and wipe samples must be divided into separate QC batches.

9.1.1. Method Blank:

- *Soil Samples:* One method blank must be digested and analyzed per soil batch. A method blank consists of 50 ml digestion solution and is carried through the entire analytical procedure. The concentration of the method blank must be less than the reporting limit or the entire batch must be re-digested and re-analyzed.

- *Wipe Samples:* One method blank must be digested and analyzed per wipe batch. The method blank will consist of a blank wipe and 50 ml digestion solution and will be carried through the entire analytical procedure. The concentration of the method blank must be less than the reporting limit or the entire batch must be re-digested and re-analyzed.

9.1.2. Laboratory Control Sample (Soils and wipes): Laboratory control samples from Secondary sources, shall be digested and analyzed per batch as follows:

9.1.2.1 Soil LCS:

- **LCS Soluble (LCSSRM):** Obtained from an independent source (ERA). Prepare the LCS soluble solution by diluting the concentrated LCS solution as indicated in the manufacturer's instructions. If necessary, prepare a different dilution so that the concentration after analysis falls within the calibration curve. Add 5ml of the diluted LCS soluble solution into 50ml digestion solution. The results must be within vendor specified QC limits or the entire batch must be re-digested and re-analyzed. Document the preparation of the LCS soluble solution in the TALS reagent module.

- **LCS Insoluble (LCSI):** Prepared by adding 0.011g of PbCrO4 into 50ml digestion solution. The results must be within 80-120% of the true value or the entire batch must be re-digested and re-analyzed.

- **LCS matrix matched (LCSSRM):** Obtained from an independent source (ERA). Add 2.5g of the soil LCS into 50ml digestion solution. The results must be within vendor specified QC limits or the entire batch must be re-digested and re-analyzed. This LCSSRM is included in DKQP guidelines however it is not reported to clients.

9.1.2.2 Wipe LCS/LCSD:

For wipe sample batches, a Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate must be prepared using the LCS soluble solution. Add a blank wipe to the digestion bottles for each of the LCS and LCSD and follow the prep for LCS Soluble detailed in Section 9.1.2.1 above.

9.1.3. **Predigestion Matrix Spikes (Soils only):** Both the soluble and insoluble pre-digestion matrix spikes must be analyzed per batch of ≤ 20 soil samples.

9.1.3.1. **Soluble Matrix Spike (MSS):** spike the sample with 1.0 ml of the 100 ppm Cr (VI) primary spiking solution prepared in Section 7.2.3 (equivalent to 40mg/kg Cr (VI)).

9.1.3.2. **Insoluble Matrix Spike (MSI):** to a separate sample aliquot add 0.011g of $PbCrO_4$. It is used to evaluate the dissolution during the digestion process.

9.1.3.3. Both matrix spikes are then carried through the digestion process. The acceptance range for the matrix spike recoveries is 75-125%. If the matrix spike recoveries fall outside these recovery limits, the entire batch must be rehomogenized, redigested, and reanalyzed.

9.1.3.4. If either matrix spike recovery is outside of the acceptable range of 75-125% but, the LCS is within the criteria specified in Section 9.1.2, additional laboratory characterization of each sample in the batch for ORP and pH can be performed at the client's request to determine if the sample exhibits reducing conditions. Further characterization for total sulfides, total organic carbon, chemical oxygen demand, or biological oxygen demand may be required by the client.

9.1.4. **Duplicate (DU) (Soils only):** One duplicate laboratory sample must be analyzed per soil batch. The sample used for the predigestion spike should be used for this purpose. Duplicate samples must have a Relative Percent Difference (RPD) of ≤20%, if both the original and the duplicate are ≥ four times the laboratory reporting limit. A control limit of ± 2.0mg/kg (laboratory reporting limit) is used when either the original or the duplicate sample is < four times the laboratory reporting limit.

9.1.5. **Post Digestion Spike (PDS) (Soils only):** Following the analysis (colorimetric determination), a post-digestion spike must be analyzed per soil batch. The spike concentration must be equivalent to 40mg/kg or twice the sample concentration, whichever is greater. The post digestion spike must be performed on a field sample, not on a field blank or preparation blank. It would be helpful to perform this analysis on the sample used for the matrix spike. Recovery limits for the post-digestion spike are 85-115%. If the post digestion spike fails to meet the recovery limits, a new aliquot of the sample

must be re-spiked and re-analyzed as described in SOP ED-WET-011 (The Analysis of Digestates for Hexavalent Chromium by SW846 7196A).

9.2. Instrument QC

See applicable analytical SOP, ED-WET-011 (The Analysis of Digestates for Hexavalent Chromium by SW846 7196A).

10. Procedure

10.1. Sample Preparation

10.1.1. Homogenize soil samples following TestAmerica Edison SOP ED-GEN-007 (Subsampling).

10.1.2. Weigh 2.5g ± 0.10g of sample into a clean labeled 125ml nalgene bottle. Record weight up to two decimal places. For wipe samples, add 1 sample wipe into a clean labeled 125 ml nalgene bottle.

10.1.3. Add 50 +/- 1.0ml of digestion solution to each sample. Then add 400mg of MgCl2 and 0.5ml of 1.0M Phosphate Buffer.

10.1.4. Matrix spike preparation (soils only): Spike the samples designated MS soluble and MS insoluble as detailed in Section 9.1.3. Add 50 +/- 1.0ml of digestion solution to each sample followed by 400mg of MgCl2 and 0.5ml of 1.0M Phosphate Buffer.

10.1.5. LCS preparation: For soil samples, prepare as detailed in Section 9.1.2.1. For wipe samples, prepare as detailed in Section 9.1.2.2. Add 400mg of MgCl2 and 0.5ml of 1.0M Phosphate Buffer.

10.1.6. Place all samples in hot water bath at a temperature of 90-95ºC and begin shaking at 175rpm for 5 minutes. Then release pressure by venting the caps. After the caps are resealed tightly, begin heating and shaking for 60 minutes. Start timing after the water bath has returned to 90-95ºC and maintain this temperature throughout the 60 minute digestion.

10.1.7. Measure and record the digestion start time and temperature, time and temperature after 30 minutes, and the digestion end time and temperature. Use a calibrated thermometer to measure the temperature of one of the samples in the water bath.

10.1.8. Following the heated digestion, gradually cool the solution to room temperature and quantitatively transfer the contents of the bottle to the vacuum filtration apparatus with Type I water rinses. Filter the digestate through a 0.45um filter (commercially purchased filter). Transfer the rinsates and filtrate to a clean labeled 100ml plastic cup. (**Note**: Use a polyethylene bottle if the colorimetric analysis is not following immediately).

10.1.9. Determine the concentration of hexavalent chromium in the sample extract using Method 7196A USEPA SW846 3rd Edition, (TestAmerica Edison SOP ED-WET-011, The Analysis of Digestates for Hexavalent Chromium by SW846 7196A).

10.2. Calibration:

Refer to the applicable analytical SOPs: TestAmerica Edison SOP ED-WET-011, The Analysis of Digestates for Hexavalent Chromium by SW846 7196A.

10.3. Sample Analysis

Refer to the applicable analytical SOPs: TestAmerica Edison SOP ED-WET-011, The Analysis of Digestates for Hexavalent Chromium by SW846 7196A.

11.0. Calculations / Data Reduction

11.1. Calculations:

Refer to the applicable analytical SOP (ED-WET-011) for calculations.

11.2. Data Reduction:

11.2.1. All sample preparation information is recorded directly in the logbook and in TALS at the time of sample preparation.

11.2.2. Record reagent information in the prep batch information (this can be viewed in the “batch information” page).

11.2.3. Record special comments and observations in the ‘worksheet’ tab.

11.2.4. The logbook pages for the prep are attached to the analytical batch as pdf files.

11.2.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch (TALS Analyst Desktop II module).

11.2.5.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.2.5.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on ‘Run checklist.’

11.2.5.3. Acknowledge by filling in responses to all unacknowledged findings. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose ‘Acknowledge Item.’

11.2.5.4. Fill in appropriate comments in the response box, then hit 'OK.' 'The column labeled 'unacknowledged findings' should show '0' for all questions once completed; then batch is ready for 2nd level review.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These

boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium.

Teris Profile Number (incineration): 50016710
Onyx Profile Number: (stabilization) 402535

- Sample waste-After analysis, the samples and standards are collected in a polyethylene container labeled 'Cr (VI) waste.' Once the container is full, it is brought to the waste room for disposal.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW-846, Method 3060A.

15.2. Occupational Health and Safety Administration (OSHA) Method W4001, Hexavalent Chromium, Methods Development Team, OSHA Salt Lake Technical Center, Salt Lake City, UT, April 2001.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. Test America Edison SOP ED-WET-011, The Analysis of Digestates for Hexavalent Chromium by SW846 7196A, most current revision.

16.0. Method Modifications:

The method has been modified to include procedures specific to the digestion of wipe samples.

17.0. Attachments

N/A

18.0. Revision History

- **Revision 13.1, dated 08 January 2021**
  - Updated SOP header with Eurofins emblem; no procedural change.
  - Sec 3: Revised the Definitions location as per the most current LQM.

- **Revision 13, dated 14 February 2019**

  - Sec. 9.1.2: Added matrix matched LCS applicable to DKQP projects.
  - Sec. 11.2.5: Revised the use of data review paper checklist to TALS DRC review, instructions added.
  - Deleted reference WI# EDS-WI-008 in Sec 15.7, not applicable.
  - Updated approval signatures to current.

- **Revision 12, dated 20 November 2014**

  - Removed references to SW846 7199 throughout the SOP; laboratory no longer perform this method.
  - Sec. 9.1.2: Revised the LCSS limits from +/-15% to vendor's certified limits to reflect actual laboratory practices.
  - Sec. 9.1.3.3: Removed notation that redigestion/reanalysis of batch is only for NJ sites or with PM/client's advice; Re-digestion and reanalysis is performed for all failed QC batch and is a method requirement.
  - Sec. 10.1.7: Revised procedures for checking sample temperature during sample digestion; temperature of the sample will be checked at start, middle and end of digestion.

- **Revision 11, dated 11 December 2012**

  - Sec 1 and 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec 9.1.2 (LCSI) and 9.1.3.2 (MSI): Deleted 0.012g to reflect actual laboratory practices.

- **Revision 10, dated 18 November 2010**

  - Section 1.1: included statement regarding the modification of the method for preparation of wipe samples.
  - Section 1.1.2: removed soil RL details and included statement referring reader to the applicable method SOP for RL details.
  - Section 4.1: Oxidizing/reducing parameters will be analyzed if requested by the client.
  - Section 4.6: Added interferences for wipe samples.
  - Section 6.2: Removed bottle top dispenser from list of supplies. Added graduated cylinder, specimen cups, transfer pipettes, and binderless quartz fiber filters to the list of supplies.
  - Section 7.1: Reformatted Reagents Section. Added Potassium Phosphate, Dibasic and Potassium Phosphate, Monobasic to list of reagents.
  - Section 8.0: Added wipes to the sample collection, preservation, shipment, and storage Section.

- Section 9.1: Modified Sample QC Section to specify wipe criteria. Added Sections 9.1.2.1 and 9.1.2.2 to differentiate the LCS procedures for soils and wipes.
- Section 9.1.3.4: Section was revised to specify the additional laboratory characterization only when required by the client.
- Sections 9.1.3, 9.1.4 and 9.1.5: added language to clarify that these QC types apply only to soils (and not to wipes).
- Section 10.1.2: added procedures for wipe sample preparation.
- Section 10.1.5 and 10.1.6: added to detail the QC sample (LCS and matrix spike) prep procedures for soils and wipes.
- Section 11.0: Replaced all calculations with a reference to the applicable analytical method SOP. Add
- Section 15.0: Added reference for OSHA Method W4001, Hexavalent Chromium (wipe sample methodology).
- Section 16.0: added method modification language for addition of wipe samples.

- **Revision 9, dated 03 February 2010**
  - Section 9.1.3.1: Changed MS to MSS to be consistent with TALS naming convention.
  - Section 10.1.3: Added Section (previously part of Section 10.1.4) to clarify that spike should be added directly to the sample aliquot prior to the addition of the digestion solution, magnesium chloride, and phosphate buffer. Subsequent Sections adjusted.
  - Section 10.1.4: Section added to include LCS soluble and LCS insoluble. Subsequent Sections adjusted.

- **Revision 8, dated 25 August 2009**
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1.2: Added reporting limit.
  - Section 7.1.8: Added prep information.
  - Removed midpoint calibration checks standard (CCS) and Calibration blank in the QC Section, both QC samples are not applicable to this SOP.
  - Deleted pH adjustment procedure, previously named Section 9.6; this procedure will be included in the analytical SOP.
  - Section 15: Added applicable references.

- **Revision 7b, dated 30 April 2009**
  - Section 5.6: Added stability and storage information.
  - Section 5.7: Added stability information
  - Section 5.8: Added stability information

- Section 10.8: Added information about the pH adjusted PS to conform to method requirements.
- Section 15: Added applicable references.

- **Revision 7a, dated 10 February 2009**

  - Section 4.4: Add filter size to Nalgene 115-ml disposable filter.
  - Section 4.10: Add Bottle top dispenser use for adding the digestion solution.
  - Section 4.11: Add Wood spatulas; Use wooden spatulas instead of the stainless steel spatulas.
  - Section 6.1: Delete pre-made standard purchased from Inorganic Ventures. Cr6+ Primary Standard will be prepared in the lab.
  - Section 6.2: Specified source of the 1000 ppm Cr6+
  - Section 6.3 and 6.6: Delete 10mg/l Cr6+ standards; Working standard 10mg/L Cr+6 is not utilize for soil analysis.
  - Section 10.3: expanded LCS information to include preparation information for both LCSs.
  - Section 10.4.2: Specified the exact amount of PbCrO4 added to the insoluble matrix spike.
  - Section 6.5: Added storage information.
  - Section 6.4: Added text: source must be different from the primary standard.
  - Section 9.3: Expanded Section to include the spiking of LCS and MS.
  - Section 9.4: Expanded Section to include recording specific information for the water bath.

- **Revision 7, dated March 2007**

  - Section 10.3: Revised to specify the types of LCS used including their source and acceptance limits.
  - Section 10.6: Deleted the requirement that ‘CCS is taken through the entire digestion process to reflect actual laboratory practices.

- **Revision 6, dated 30 October 2006**

  - Section 9.6.3: Corrected referenced SOP number from EDS-WET-010 to EDS-WET-011.
  - Section 10.5:Revised first sentence to read “One duplicate laboratory sample per batch *must* be analyzed.” (‘Must’ previously read ‘should’).
  - Section 10.4.3: The text in italics was added to the following sentence in Section 10.4.3:
    “NOTE: If the spiked sample concentration is greater than 4x the predigestion spike concentration, no redigestion/reanalysis is required unless the samples are from a NJ site in which case the lab project manager should be contacted for further guidance.”
  - Section 10.8: Added the following text:
    “If the Post Digestion Spike fails to meet recovery limits, a new aliquot of the sample must be re-spiked and re-analyzed.

Title: The Analysis of Digestates for Hexavalent Chromium by EPA SW846 7196A

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Approvals (Signature/Date):

Jasmine Parillo Department Manager	9/23/2024 Date	Dan Helfrich Health and Safety Manager	9/20/2024 Date
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1.0 Scope and Application

1.1 Analytes, Matrix(s), and Reporting Limits

1.1.1 This SOP is applicable to the determination of hexavalent chromium digestates of solid samples using SW846 Method 7196A. Solid samples must be first digested according to Method 3060A USEPA SW846 3rd Edition, TestAmerica Edison SOP ED-WET-010 (The Alkaline Digestion of Soil Samples for the analysis of hexavalent chromium).

1.1.2 The laboratory's reporting limit for hexavalent chromium in soil is 2.0 mg/kg.

1.1.3 On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Solid samples are digested utilizing SOP ED-WET-010, alkaline digestion of soil samples via Method 3060A. Following the sample preparation and digestion procedure, dissolved hexavalent chromium is determined spectrophotometrically at 540 nm by reaction with diphenylcarbazide in acid solution.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1 Molybdenum and Mercury react to form color with diphenylcarbazide, however the intensity of color is much lower than those for chromium at the specified pH. Concentrations up to 200 mg/L can be accepted.

4.2 Vanadium interferes strongly, but can be tolerated up to 10 times the concentration of Cr (VI) present.

4.3 Iron may cause a yellow color but should not affect the colorimetric measurement at 540 nm.

4.4 Reducing matter may reduce hexavalent Cr to trivalent Cr in varying amounts. No preventative measure is available at this time; however, interference is checked by post digestion spike samples.

4.4.1 The reducing/oxidizing tendency of each matrix may be determined by characterization of samples for additional analytical parameters, such as pH, ferrous iron, sulfides, and Oxidation Reduction Potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon

(TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD).

4.5 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.

5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at an elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

6.0 Equipment and Supplies

6.1. Instrument

- Spectrophotometer for use at 540 nm, providing a light path of 1cm or longer
- pH meter, Orion benchtop or equivalent

6.2. Supplies

- Nalgene 115-ml disposable filter unit with 0.45um filter
- Membrane filters, 0.45um and 0.1 um
- Specimen cups (plastic)
- Eppendorf or Finnpipettes, varying volumes
- Class A volumetric flasks
- Class A graduated cylinder
- Vacuum pump
- Magnetic stirrer (Teflon coated)
- Transfer pipettes

7. Reagents and Standards

7.1. Reagents

7.1.1. Sulfuric acid (10% v/v): Measure 100 ml of concentrated reagent grade H_2SO_4 into a 1000 ml volumetric flask (1/2 filled w/D.I. water) and dilute to mark with deionized water. Stable for six months, store at room temperature.

7.1.2. Indicator solution (DPC): Place 5.0 g 1,5-Diphenylcarbazide (AR Grade) in an amber 1000 ml volumetric flask and dilute to mark with acetone. Prepare monthly or when solution becomes cloudy. Store at room temperature.

7.1.3. Acetone: reagent grade; for stability and storage information refer to manufacturer's instructions.

7.1.4. 1:1 HNO3: Add 500ml DI to a 1 Liter volumetric flask and then slowly add 500ml of concentrated reagent grade HNO3. Solution is stable for 6 months. Store at room temperature.

7.1.5. 1N NaOH: Place 4.0g reagent grade NaOH in a 100ml volumetric flask and dilute to the mark with deionized water. Stable for six months, store at room temperature.

7.2 Standards

7.2.1 Potassium Dichromate Primary Standard Solution (1000 mg/L Cr+6): Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3093-01 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. Stable for 6 months, store at room temperature.

7.2.2 Potassium Dichromate Secondary Standard Solution (1000 mg/L Cr+6): Weigh 0.2829 g of Potassium Dichromate ACS grade, J.T. Baker, cat# 3090-04 (dried for 1 hour at 105ºC) and dissolve in deionized water in a 100 ml flask. Dilute to the mark. **Note**: Prepare the secondary standard from a source or lot different from the primary standard solution. Stable for 6 months, store at room temperature.

7.2.3 Hexavalent Chromium Primary Spiking Solution (100 mg/L Cr+6): Prepare by adding 10 ml of 1000 mg/L Cr+6 primary standard (Sec 7.2.1) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.4 Hexavalent Chromium Secondary Spiking Solution (100 mg/L Cr+6) Prepare by adding 10 ml of 1000 mg/L Cr+6 secondary standard (Sec 7.2.2) to a 100 ml volumetric flask and dilute to volume with deionized water. Stable for 6 months, store at room temperature.

7.2.5 Lead Chromate: AR Grade; for stability and storage information refer to manufacturer's instructions.

8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container [1]	Min. Sample Size[2]	Preservation	Holding Time [3]	Reference
Soils	Glass, plastic	2.5 grams	Cool 4 ± 2°C	30 Days - *from sampling to extraction*	SW846 Method 3060A
				168 Hours – *from extraction to determinative analysis*	SW846 Method 7196A

[1] Containers should not contain stainless steel
[2] Additional volume may be required if the sample is chosen for QA or if a re-prep is required.
[3]The alkaline digestate must be analyzed within 168 hours after extraction from the soil.

9. Quality Control

9.1. **Sample QC** - The following quality control samples are prepared with each batch of 20 samples or less.

9.1.1. Method Blank: One method blank must be digested and analyzed per batch. A method blank consists of 50 ml digestion solution and is carried through the entire digestion and analytical procedure. The concentration of the method blank must be less than the reporting limit or the entire batch must be re-digested and re-analyzed.

9.1.2. Laboratory Control Sample: Two secondary source (LCS) shall be digested and analyzed per batch.

- **LCS soluble (LCSSRM)** is obtained from an independent source (Phenova). Prepare the LCS soluble solution by diluting the concentrated LCS solution as indicated in the manufacturer's instructions. If necessary, prepare a different dilution so that the concentration after analysis falls within the calibration curve. Add 5ml of the diluted LCS soluble solution into 50ml digestion solution. The results must be within vendor specified QC limits or the entire batch must be redigested and re-analyzed. Document the preparation of the LCS soluble solution in the Reagent module in TALS.

- **LCS insoluble (LCSI)** is prepared by adding 0.010g-0.020g of PbCrO4 into 50ml digestion solution. The results must be within 80-120% of the true value or the entire batch must be redigested and re-analyzed. (Typical amount of $PbCrO_4$ added to LCSI is 0.011g, this is equivalent to 708 mg Cr (VI)/Kg).

9.1.3. Predigestion Matrix Spikes: Both a soluble and insoluble pre-digestion matrix spikes must be analyzed per batch of ≤ 20 field samples.

9.1.3.1 Soluble matrix spike (MSS): spike the sample with 1.0 ml of the 100 ppm Cr (VI) primary spiking solution prepared in Sec 7.2.3 (equivalent to 40mg/kg Cr(VI)).

9.1.3.2 Insoluble matrix spike (MSI): to a separate sample aliquot add 0.010g- 0.020g of $PbCrO_4$. It is used to evaluate the dissolution during the digestion process. Typical amount of $PbCrO_4$ added to LCSI is 0.011g, this is equivalent to 708 mg Cr (VI)/Kg.

9.1.3.3 Both matrix spikes are then carried through the digestion process. The acceptance range for the matrix spike recoveries is 75-125%. If the matrix spike recoveries fall outside these recovery limits, the entire batch must be rehomogenized, redigested, and reanalyzed.

- Note: If redigesting/reanalyzing the batch, the Soluble matrix spike (MSS) is spiked at twice the sample concentration or 40mg/kg Cr(VI) whichever is greater.

9.1.3.4 If upon reanalysis, the matrix spike is not within the recovery limits of 75%-125%, but the LCS is within the criteria specified in Section 9.1.2, additional laboratory characterization of each sample in the batch for ORP (see Eurofins Edison SOP ED-WET-066, Redox by SM2580) and pH (see Eurofins Edison SOP ED-WET-061, pH Soil by SW846 9045C) may be required by the client to determine if the sample exhibits reducing conditions. Further characterization for total sulfides, Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), or Biochemical Oxygen Demand (BOD) may also be required by the client.

9.1.4. Duplicate (DU): One duplicate laboratory sample must be analyzed per batch. The sample used for the predigestion spike should be used for this purpose. Duplicate samples must have a Relative Percent Difference (RPD) of ≤20%, if both the original and the duplicate are ≥ four times the laboratory reporting limit. A control limit of ± 2.0mg/kg (laboratory reporting limit) is used when either the original or the duplicate sample is < four times the laboratory reporting limit.

9.1.5. Post Digestion Spike (PDS): Following the analysis (colorimetric determination), a post-digestion spike must be analyzed per batch. The spike concentration must be equivalent to 40mg/kg or twice the sample concentration, whichever is greater. To spike the extract with 40mg/Kg Cr

(VI), add 0.50 ml of 100 ppm Cr (VI) primary standard to a 50.0 ml alkaline extract, pour out 45.0 ml of the spiked sample and add color indicator and adjust pH (see Sec 10.3.4 & 10.3.5); bring the final volume to 50ml with deionized water. The post digestion spike must be performed on a field sample, not on a field blank or preparation blank. If possible perform the post digestion spike analysis on the sample used for the matrix spike. Recovery limits for the post-digestion spike are 85-115% of the true value. If the post digestion spike fails to meet the recovery limits, a post spike must be re-analyzed using the alkaline extract. Adjust the pH of the sample Duplicate (alkaline extract) back up to 8.0-8.5 using 1N NaOH; then respike and reanalyze following Sec. 10.3.4-10.3.5.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV): The ICV (0.50 mg/L Cr (VI)) is analyzed immediately after an acceptable initial calibration. The ICV standard must be from a source separate from the calibration standards (i.e., different manufacturer or separate lot) and its recovery must be within ±10% of the expected value. The ICV is prepared and analyzed similar to the calibration standards; prepare by adding 0.5 ml of 100 ppm Cr (VI) Secondary Spiking Solution (Sec. 7.2.4) into a specimen cup containing 50 ml of digestion fluid. If the measured concentration exceeds the ±10% limit, a second analysis should be performed. If the result still exceeded the ±10% limit, the analysis should be terminated until the source of the problem is identified and corrected.

9.2.2. Continuing Calibration Verification (CCV): A CCV is a mid-point calibration Cr (VI) standard (0.5ppm) from a different source than the calibration standards. Add 0.5 ml of 100 ppm Cr (VI) Secondary Spiking Solution (Sec. 7.2.4) into a specimen cup containing 50 ml of digestion fluid. The CCV is analyzed after every 10 samples (20 readings including backgrounds), and after reading the last sample. Acceptance criteria for the CCV are 90-110% of the true value. If the result is not within the acceptance limits, all samples following the last acceptable CCV must be re-analyzed.

9.2.3. Initial Calibration Blank and Calibration Check Blank (ICB,CCB): An initial calibration blank (ICB) must be analyzed immediately following the calibration curve and a continuing calibration blank (CCB) is analyzed after each CCV. The sample consists of 50 ml digestion solution and is carried through the entire analytical procedure. The concentration of the CCB must be below the reporting limit if not, all samples following the last acceptable CCB must be re-analyzed.

10. Procedure

10.1. Sample Preparation:

10.1.1. Just prior to analysis, SLOWLY adjust the pH of the sample extract to a pH of 7.0-8.0 with constant stirring using 1:1 nitric acid. Record pH and time. If a flocculent precipitate forms after the addition of 1:1 HNO3, filter the sample through a 0.45 um membrane filter, a larger size filter may be used to pre-filter the sample. Monitor the pH with a calibrated pH meter. If the pH drops below 7.0, discard the sample extract and re-digest the sample.

10.1.1.1. Remove and rinse the stir bar. Quantitatively transfer the sample extract to a 100 ml volumetric flask and dilute to the mark with deionized water.

10.1.1.2. If necessary, the solution can be diluted to eliminate the effects of color on the analytical determination. Use the smallest dilution necessary and record results.

10.2. Calibration

10.2.1. Prepare fresh calibration standards everyday or before each analysis.

10.2.2. Prepare a set of 6 calibration standards. Prepare 0.0, 0.05, 0.1, 0.5, 0.75, and 1.25 mg/l $K_2Cr_2O_7$ standards by aliquoting 0, 0.05, 0.1, 0.5, 0.75 and 1.25 mls of 100ppm Cr (VI) Primary Spiking Solution (Sec 7.2.3) respectively into the specimen cups containing 50mls of digestion fluid. At the same time prepare the mid-point Initial Calibration Verification and Initial Calibration Blank, see Sec 9.2.

10.2.3. Adjust pH to 7.5±0.5 with 1:1 HNO_3 and dilute to 100 ml with DI water. Record pH reading and analysis time in the logbook.

10.2.4. Develop the color for the standards as explained in Section 10.3 of this SOP.

10.2.5. Following the preparation of the calibration standards, “zero” the spectrophotometer with the 0 ppm standard. Analyze the standards.

10.2.6. The correlation coefficient must be 0.995 or better.

10.2.7. After calibration is entered into TALS, calculate %RE (%relative error) on two calibration standards, the standard at or near the mid-point of the calibration and the standard at the lowest level. Go to calibration events and click on details, review the column labeled ‘read back amount’ and ‘% Error’ for the low standard and midpoint standard.

Low standard (10ppb): The %RE (relative error) must be +/- 50%; if not, stop the analysis and recalibrate.

Mid-point normal (75ppb): The %RE (relative error) must be +/- 10%; if not, stop the analysis and recalibrate

10.2.8. Immediately following calibration of the spectrophotometer and before reading samples, verify the calibration of the spectrophotometer by analyzing an ICV and ICB.

10.3. Sample Analysis

10.3.1. Warm up spectrophotometer and set to 540nm.

10.3.2. Prepare two rows of plastic cups and label them accordingly (first row with sample no. and the 2nd row as background).

10.3.3. Pour out 45.0 ml of the sample extracts, which have already been pH adjusted to between 7.0 and 8.0, into the appropriately labeled cups. Do the same for the background cups using 10 ml of the sample extract.

10.3.4. Add 1.0 ml indicator solution and swirl. (Do not add reagent to background sample).

10.3.5. Slowly add 10% H_2SO_4 (Sec 7.1.1) dropwise while swirling and monitoring pH until pH is 2 ±0.5. Record reading in the appropriate logbook.

10.3.6. Transfer sample to 50 ml volumetric flask and bring to volume with deionized water. Empty contents into labeled specimen cup to mix.

10.3.7. Let stand 5-10 minutes for full color development.

10.3.8. Read calibration standards, QC samples, and samples in a 1 cm cell on a spectrophotometer at 540 nm. (Note: Calibration standards and samples must be read within the same timeframe following full color development).

10.3.9. Continue analyzing samples and quality control samples by first reading the sample followed by its background sample.

10.3.10. Dilute samples that exceed calibration curve using deioinized water and analyze following Sec. 10.3.3-10.3.7. Add dilution factor to TALS under "dilution" column. Note: The dilution should be made from the sample extract that has not had the indicator added.

10.3.11. After every twenty readings (10 samples) and after reading the last sample in the batch, verify the spectrophotometer calibration by reading a calibration check standard (CCV) and calibration check blank (CCB).

11.0. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

$$\text{MS \% Recovery} = \frac{(\text{spiked sample}) - (\text{unspiked sample})}{\text{spiked concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value} - \text{dup. sample value}|}{[(\text{orig. sample value} + \text{dup. sample value})/2]} \times 100$$

11.3. Concentration = Hexavalent Chromium in mg/Kg = $\frac{A \times B \times E * 100}{C \times D}$

Where:
A = Concentration from the calibration curve in mg/L
B = Final digested volume in Liters
C = Wet sample weight in kg
D = percent solids
E = Dilution (if necessary)

NOTE: All dry weight corrections are made in TALS at the time the final report is prepared.

11.4. Trivalent Chromium Calculation: When required, the calculation for trivalent chromium is completed using TALS Method '7196a_CR3.' Samples must be logged in under this method as well as the methods for Total chromium (metals) and Hexavalent chromium. Upon completion of the total and hexavalent chromium analyses the analyst may process the trivalent chromium results as follows:

- In order for TALS to perform an automated trivalent chromium calculation enter '1' (for 'yes') in trivalent chromium batch information page in response to the prompt 'automatically perform calculation.' As long as both methods are at 2nd level review status in TALS the program will automatically pull in the metals results for chromium and the hexavalent chromium results to perform the automated calculation. TALS will display an 'ok' message on the worksheet tab if the Cr6 result is less than Total Cr result.
- In cases where Total Cr results are not linked to TALS method 7196a_CR3, trivalent chromium is calculated manually. Enter '0' (for 'no') in batch information page 'Perform calculation.' Results will be entered manually.

11.5. Data reduction:

11.5.1. All data is recorded directly in TALS and recorded in the logbook at the time the analysis is performed.

11.5.2. Attach the prep and analytical logbook pages and calibration curve to the batch as a pdf file.

11.5.3. On the worksheet tab, enter the sample absorbance under 'Uncorrected Abs' and background absorbance under 'Color Blank Abs.' Also, enter the pH between 7-8 as 'initial pH',' pH between 1.5-2.5 as 'Final pH' and the background pH under 'Notes.'

11.5.4. Record special notes and observations in the "worksheet" tab and record reagent information in the prep batch information page (see "view batch information" page of ADII).

11.5.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch (TALS Analyst Desktop II module).

11.5.5.1. Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.5.5.2. Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.5.3. Acknowledge by filling in responses to all unacknowledged findings. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.5.4. Fill in appropriate comments in the response box, then hit 'OK.' 'The column labeled 'unacknowledged findings' should show '0' for all questions once completed; then batch is ready for 2nd level review.

12.0. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to Eurofins Edison SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0. Pollution Control

It is Eurofins 's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability).

Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins Edison SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are produced when this method is carried out:

- Expired Standards – The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.
- Sample waste-After analysis, the samples and standards are collected in a polyethylene container labeled 'Cr6+ waste.' Once the container is full, it is brought to the waste room for disposal.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW-846, Method 7196A.

15.2. Test America Edison SOP ED-WET-010, The Alkaline Digestion of Soil Samples for the Analysis of Hexavalent Chromium, most current revision.

15.3. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.4. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.5. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.6. TestAmerica Edison SOP ED-WET-066, Redox, Analysis of Oxidation-Reduction Potential, most current revision.

15.7. TestAmerica Edison SOP ED-WET-061, Analysis of pH for soil and organic samples electrochemically, Method 9045C, most current revision.

15.8. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16. Method Modifications:

N/A

17. Attachments

N/A

18. Revision History

- **Revision 14, dated 23 September 2024**
  - Updated TestAmerica references to Eurofins throughout the document.
  - Sec 10.2.7: Added requirements and procedures to evaluate %RE in the calibration.

- **Revision 13, dated 14 June 2024**
  - Updated SOP header to Eurofins logo.
  - Sec. 11.5.6: Removed reference to WI EDS-WI-008, manual wetchemistry data review checklist; TALS DRC is used as outlined in Sec. 11.5.5.
- **Revision 12, dated 17 June 2020**
  - Updated SOP header with Eurofins emblem.
  - Sec. 9.1.2: Changed LCS source vendor from ERA to Phenova.
  - Sec. 11.5.5: Revised data review procedure to include TALS DRC procedures.
  - Sec 15.8: Deleted Wetchem Data Review Checklist Work Instruction # EDS-WI-008, not applicable.

- **Revision 11, dated 05 March 2018**
  - Sec 11.4: Added procedure for Trivalent chromium calculation using TALS method 7196a_CR3; subsequent section adjusted accordingly.

- **Revision 10, dated 23 February 2016**
  - Sec 9.1.2 and 9.1.3.2: Revised the allowable amount of PbCrO4 to add to the QC sample LCSI to reflect the range referenced in the Method. Also included the typical amount of $PbCrO_4$ that is added to the QC sample (LCSI and MSI).
  - Sec 9.1.3.3: Added spiking instructions for MSS when redigesting a batch; MSS should be spiked at 2x sample concentration or 40mg/kg, whichever is greater.

- **Revision 9, dated 25 September 2013**
  - Sec 1 & 12: Updated LQM section references to reflect the most current LQM revision.
  - Sec. 9.1.2: Changed nomenclature to LCSSRM and revised the acceptance limits from 85-115% to vendor specified QC limits.
  - Sec. 9.1.3.3: Deleted text: '*Note: If the spiked sample concentration is greater than 4X the predigestion spike concentration, no redigestion/reanalysis is required unless the samples are from a NJ site in which case the project manager should be contacted for further guidance.'*

Per method 3060a, all batches must be rehomogenized, redigested and reanalyzed if MSS or MSI fail regardless of location.

- Sec. 10.3.5: Changed pH range from 1.6-2.2 to pH 2.0 +/-0.5 as per the method.
- Sec. 11.4.3: Changed pH range from 1.6-2.2 to pH 1.5-2.5 as per the method.

- **Revision 8, dated 06 September 2011**
  - Sec 3: Revised the LQM reference for the list of definitions.
  - Sec 9.1.3.4: Clarified lab procedures for failing matrix spikes.
  - Sec. 9.2.2: Added procedure if the CCV is not within acceptance limits.
  - Sec. 9.2.3: Added procedure if CCB is not below reporting limit.
  - Sec. 11.4: Revised data reduction section to reflect actual laboratory practices.
  - Sec 15.9: Reference added.

- **Revision 7, dated 25 August 2009**
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Sec 1.1: added laboratory's reporting limit; change the range of analysis to 0.05-1.25mg/L to reflect actual laboratory practices.
  - Sec 9.1.5: Added preparation instruction for Post digestion spike.
  - Sec. 9.2: added ICV in the Instrument QC.
  - Sec 10.1: Added procedure for adjusting pH of sample extract; procedure formerly described in the Digestion SOP (ED-WET-010).
  - Sec 15: Added applicable references.

- **Revision 6a, dated 21 April 2009**
  - Sec.4.3: Added membrane filters, 0.45um and 0.1um.
  - Sec 4.4: Delete Erlenmeyer flasks and beakers
  - Sec 4.8:Add: Eppendorf or Finnipettes; varying volume
  - Sec 5.1, 5.2 & 5.4: Add stability and storage information.
  - Sec 5.5: Added reagent used for the pH adjusted PS: 1N NaOH: Place 4.0g NaOH in a 100 volumetric flask and dilute to the mark with deionized water. Solution is stable for 6 months. Store at room temperature.
  - Sec. 6.1: Potassium Dichromate Primary Standard Solution (1000mg/L Cr+6): Weight 0.2829g of dried (105ºC) potassium dichromate and dissolve in deionized water in a 100ml flask. Dilute to the mark. Store at room temperature. Solution is stable for 6 months.
  - Sec 6.1: Deleted pre-made standard purchased from Inorganic Ventures. Cr6+ Primary Standard will be prepared in the lab.
  - Sec 6.2: Specified source of the 1000 ppm Cr(VI).

- Sec 6.3 and 6.6: Deleted the 10mg/L Cr (VI) working standard for soil analysis.
- Sec 6.5: Add storage information.
- Sec 6.4: Add text: Source must be different from the primary standard.
- Sec 9.2.2: Replace 100 ppm stock standard with 100 ppm Cr6+ Primary spiking solution.
- Sec. 9.2.3: Add text: Record pH reading in the logbook.
- Sec. 9.3.8: Add text: Make the dilution from the sample that has not had the indicator added.
- Sec 9.3.6: Add text: Before reading samples, verify the calibration of the spectrophotometer by analyzing a CCV and CCB.
- Sec. 10.7: Added procedure for reanalyzing Post Spike sample to conform to method requirements: *If the post digestion spike fails to meet the recovery limits, a post spike must be reanalyzed using the alkaline extract. Adjust the pH of the sample Duplicate (alkaline extract) back up to pH 8.0-8.5 using 1N NaOH; then respike and reanalyze*.

- **Revision 6, dated March 2007**

  - Section 7.2: Section deleted (start color development for digested samples within one hour after adjusting pH between 7.5±0.5); section not applicable.
  - Section 9.3.7: The text in italics was inserted to clarify the time period for measuring sample absorbance.

    "Note: Calibration standards and samples must be read within the same timeframe following full color development."
  - Section 10.2: Deleted the requirement that 'CCS is taken through the entire digestion process' to reflect actual laboratory practices.
  - Section 10.4. Revised to specify the types of LCS used including their source and acceptance limits.

- **Revision 5, dated 30 October 2006**

  - Section 9.2.2: Revised the high point of the calibration curve from 1.0 ppm to 1.25 ppm. Standards preparation instructions revised accordingly.
  - Section 9.2.2: Revised to include instructions for preparation of a second source mid-point Calibration Check Standard.
  - Section 10: Inserted a new Section 10.1 explaining the QA batching system and inserted a new section 10.4 detailing the LCS requirements. All existing sections renumbered accordingly.
  - Section 10.6 (previously Section 10.5): The italicized text was added to the following sentence:

    "NOTE: If the spiked sample concentration is greater than 4x the predigestion spike concentration, no redigestion/reanalysis is required *unless the samples are*

from a NJ site in which case the lab project manager should be contacted for further guidance."

- Section 10.6 (previously Section 10.5): Added STL Edison SOP and method references for ORP and pH.
- Section 10.7 (previously Section 10.6): Added the following text: "If the post digestion spike fails to meet the recovery limits, a new aliquot of the sample must be re-spiked and re-analyzed."
- Section 10.8 (previously Section 10.7): Revised first sentence to read "One duplicate laboratory sample per batch *must* be analyzed." ('Must' previously read 'should').

Title: The Analysis of Waters for Hexavalent Chromium by EPA SW846 Method 7196A

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Approvals (Signature/Date):

Jasmine Parillo Department Manager	9/23/2024 Date	Dan Helfrich Health and Safety Manager	9/20/2024 Date
Carl Armbruster Quality Assurance Manager	9/23/2024 Date	Mark Acierno Laboratory Director	9/21/2024 Date

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1.0 Scope and Application

1.1. SW846 Method 7196A may be used to determine the hexavalent chromium content of groundwater samples or digestates of solid samples. This SOP is applicable for the determination of hexavalent chromium in groundwater samples. Groundwater samples associated with regulated drinking water samples (i.e. potable water wells) are not applicable to this Method.

1.2. The range of analysis by this method is 10-200 ug/l hexavalent chromium.

1.3. On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

2.0 Summary of Method

Hexavalent chromium is determined spectrophotometrically at 540nm by reaction with diphenylcarbazide in acid solution. For each batch of samples a method blank, a laboratory control sample, matrix duplicate, and a post verification spike are analyzed and compared to method limits and laboratory control limits.

3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

4.0 Interferences

4.1. Molybdenum and Mercury react to form color with diphenylcarbazide, however the intensity of color is much lower than those for chromium at the specified pH. Concentrations up to 200 mg/L can be accepted.

4.2. Vanadium interferes strongly, but can be tolerated up to 10 times the concentration of hexavalent chromium present.

4.3. Iron may cause a yellow color but should not affect the colorimetric measurement at 540 nm.

4.4. Reducing matter may reduce hexavalent Cr to trivalent Cr in varying amounts. No preventative measure is available at this time; however, interference is checked by a post verification spike samples.

4.4.1. The reducing/oxidizing tendency of each matrix may be determined by characterization of samples for additional analytical parameters, such as pH, ferrous iron, sulfides, and Oxidation Reduction Potential. Other indirect indicators of reducing/oxidizing tendency include Total Organic Carbon (TOC), Chemical Oxygen Demand (COD), and Biological Oxygen Demand (BOD).

4.5. For waste materials containing soluble trivalent chromium concentrations greater than four times the laboratory hexavalent chromium reporting limit, the hexavalent chromium results obtained using this method may be biased high due to method-induced oxidation.

5.0. Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. **Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table.** A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure

Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Dichromate	Oxidizer Corrosive Carcinogen	0.1 Mg/M3 TWA as CrO3	Extremely destructive to tissues of the mucous membranes and upper respiratory tract. May cause ulceration and perforation of the nasal septum. Symptoms of redness, pain, and severe burn can occur. Dusts and strong solutions may cause severe irritation. Contact can cause blurred vision, redness, pain and severe tissue burns. May cause corneal injury or blindness.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison Carcinogen	1 Mg/M3-TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

6.0. Equipment and Supplies

6.1. Instrumentation

- pH meter, Orion benchtop or equivalent
- Spectrophotometer for use at 540 nm with a light path of 1 cm (Hach DR2800 or equivalent).

6.2. Supplies

- Membrane filters, 0.45 um and 0.1 um (NALGENE disposable)
- Glasswares: volumetric pipets (class A) and volumetric Flasks (class A)
- Graduated Cylinder (class A)
- Transfer pipettes
- Calibrated pipettes: Eppendorf or equivalent
- Vacuum pump
- Specimen Cups (plastic)
- Magnetic stirrer (teflon-coated)

7.0. Reagents and Standards

7.1. Reagents

7.1.1. Sulfuric acid (10% v/v) - Measure 100 ml of concentrated reagent grade H_2SO_4 into a 1000 ml volumetric flask (1/2 filled w/D.I. water) and dilute to mark with deionized water. Reagent is stable for 6 months. Store at room temperature.

7.1.2. Indicator solution - Place 5.0 g 1, 5-Diphenylcarbazide (AR Grade) in an amber 1000ml volumetric flask and dilute to mark with acetone. Prepare monthly or when solution becomes cloudy. Store at room temperature.

7.1.3. Acetone (reagent grade) - Store at room temperature. For stability information, refer to manufacturer's instructions or 2 years from date opened if no instructions are available.

7.2. Standards

7.2.1. Primary Potassium Dichromate ($K_2Cr_2O_7$) Stock Standard (1000 mg/L Cr+6) - Weigh 0.2829 g of dried (105°C) analytical reagent grade (or better) potassium dichromate ($K_2Cr_2O_7$) crystals (J.T.Baker catalog nos. 3090, 3093 or equivalent). Dissolve in deionized water in a 100ml flask. Dilute to the mark. Standard solution is stable for 6 months. Store at room temperature.

7.2.2. Hexavalent Chromium Primary Spiking Solution (100 mg/L Cr+6) - Prepare by adding 10ml of the 1000 mg/L Primary Potassium Dichromate Standard (see Section 7.2.1) to a 100ml volumetric flask and dilute to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.2.3. Hexavalent Chromium Primary Spiking Solution (10 mg/L Cr+6) - Prepare by adding 1.0ml of 1000 mg/L Cr+6 Standard (see Section 7.2.1) to a 100ml volumetric flask and diluting to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.2.4. Secondary Potassium Dichromate ($K_2Cr_2O_7$) Stock Standard (1000 mg/L Cr+6) - Weigh 0.2829 g of dried (105°C) analytical reagent grade (or better) potassium dichromate ($K_2Cr_2O_7$) crystals (J.T.Baker catalog nos. 3090, 3093 or equivalent). *The source of this standard must be from a different lot or manufacturer than the Primary Potassium Dichromate Stock Standard detailed in Section 7.2*.1. Dissolve in deionized water in a 100ml flask. Dilute to the mark. Standard solution is stable for 6 months. Store at room temperature.

7.2.5. Hexavalent Chromium Secondary Spiking Solution (100 mg/L Cr+6) - Prepare by adding 10ml of 1000 mg/L Cr+6 Secondary Standard (see Section 7.2.4) to a 100ml volumetric flask and diluting to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

7.2.6. Hexavalent Chromium Secondary Spiking Solution (10 mg/L Cr+6) - Prepare by adding 1.0ml of 1000 mg/L Cr+6 Secondary Standard (see Section 7.2.4) to a 100ml volumetric flask and diluting to volume with deionized water. Standard solution is stable for 6 months. Store at room temperature.

8.0. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time [1]	Reference
Waters	HDPE[2]	250 mL	Cool 4 ±2°C	24 Hours	SW 7196A

[1] Samples must be analyzed within 24 hours from time of collection. Store samples at 4°C before analysis.
[2] High-density polypropylene container

9.0. Quality Control

9.1. **Sample QC** - - The following MB, LCS, and PVS are analyzed with each batch of 20 samples or each time samples are setup, whichever is more frequent. The DU is analyzed at a frequency of 1 per 10 samples or each time samples are set up, whichever is more frequent.

9.1.1. Method Blank (MB): Use deionized water for the method blank. Analyze using all reagents in the same volumes as used in the analysis of samples. The blank value should fall below the reporting limit if not, all samples associated with the method blank must be re-analyzed. Do not subtract the blank value from sample values.

9.1.2. Post Verification Spike (PS): The PVS concentration must be two times the concentration of Cr(VI) found in the sample or 30.0 ug/L (whichever is greater). If necessary, dilute the PS to fall within the range of the calibration curve. If the PS recovery is not within 85-115%, dilute the sample and re-analyze.

Note: The post spike is identified as “MS” in TALS QC.

9.1.3. Matrix Duplicate (DU): A duplicate is prepared and analyzed in exactly the same manner as the original sample. The relative percent difference (RPD) between the sample and duplicate should be less than or equal to 20%.

9.1.4. Laboratory Control Sample (LCS): The hexavalent chromium LCS is a certified QC standard purchased from Phenova (Catalog WP

Hexavalent Chromium). The LCS is used to monitor the accuracy of the entire analytical process. The LCS recovery must be within vendor specified QC limits or all samples associated with the LCS must be reanalyzed.

9.2. Instrument QC

9.2.1. Initial Calibration Verification (ICV): The ICV (75.0 ug/L Cr (VI)) is analyzed immediately after an acceptable initial calibration. The ICV standard must be from a source separate from the calibration standards (i.e., different manufacturer or separate lot) and its recovery must be within ±10% of the expected value. The ICV is prepared by adding 0.375ml of a 10 mg/L Cr6+ Hexavalent Chromium Secondary Spiking Solution (Section 7.2.6) into a 50 ml volumetric flask and diluting to 50ml with deionized water. If the measured concentration exceeds the ±10% limit, a second analysis should be performed. If the result still exceeded the ±10% limit, the analysis should be terminated until the source of the problem is identified and corrected.

9.2.2. Calibration Check Verification (CCV): A midpoint Calibration Check Verification spiked at 75.0 ug/L is prepared in the same way as the ICV (See Section 9.2.1). The CCV is analyzed after every 10 samples (20 readings including backgrounds) and after reading the last sample. The result must be within ± 10% of the true value. If not, all samples following the last acceptable CCV must be re-analyzed.

9.2.3. Initial and Continuing Calibration Blank (ICB/CCB): Initial Continuing Calibration Blank (ICB) must be analyzed after the ICV. Continuing Calibration Blank (CCB) must be analyzed after every 10 samples or after each Calibration Check Verification (CCV). Use deionized water for the ICB/CCB. The value of the ICB/CCB should be less than the RL. If not, all samples following the last acceptable ICB/CCB must be re-analyzed.

9.2.4. Minimum Reporting Level (MRL), 10ppb: The MRL must be analyzed daily before samples are analyzed. Prepare the MRL by adding 0.05ml of a 10 mg/L Hexavalent Chromium Primary Spiking Solution (Section 7.2.3) into a 50 ml volumetric flask and diluting to 50ml with deionized water. The results must fall within 50-150% of the true value.

10.0 Procedure

10.1. Calibration

10.1.1. Prepare fresh calibration standards everyday before analysis.

10.1.2. Initial Calibration Standards: (6 points). Place about 25 mL of deionized (DI) water in six 50 ml flasks. Prepare the six points as follows:

Cr(6+) Standard conc. (ug/L)	Volume (ml) of 10 mg/L Cr+6 (Section 7.2.3) to add	Final volume (ml) using Deionized water

0.0	0.0	50
10.0	0.05	50
50.0	0.25	50
75.0	0.375	50
100	0.50	50
200	1.0	50

10.1.3. Analyze the calibration standards in the manner detailed in Sections 10.2.1 thru 10.2.7. Zero the spectrometer with the 0 ug/L standard.

10.1.4. Perform linear regression on data resulting from analysis of the initial calibration. The correlation coefficient must be 0.995.

10.1.5. After calibration is entered into TALS, calculate %RE (%relative error) on two calibration standards, the standard at or near the mid-point of the calibration and the standard at the lowest level. Go to calibration events and click on details, review the column labeled 'read back amount' and '% Error' for the low standard and midpoint standard.

Low standard (10ppb): The %RE (relative error) must be +/- 50%; if not, stop the analysis and recalibrate.

Mid-point normal (75ppb): The %RE (relative error) must be +/- 10%; if not, stop the analysis and recalibrate

10.1.6. Analyze the Initial Calibration Verification (ICV) standard in the same manner as the initial calibration standards detailed above. If the ICV meets the criteria listed in Section 9.2.1, the analyst may continue with sample analysis (Section 10.2).

10.2. Sample Analysis

10.2.1. Warm up spectrophotometer and set to 540 nm.

10.2.2. Place 45.0 mL of sample into a 100 ml specimen cup. Also, place 45 mL of sample in a separate cup for background.

10.2.3. Add 1.0 ml indicator solution (see Section 7.1.2) and swirl. (Do not add indicator to background sample).

10.2.4. Slowly add 10% H_2SO_4 (See Section 7.1.1) dropwise to sample and background while measuring pH until pH is 1.5-2.5. Record reading.

10.2.5. Place sample in a 50 ml volumetric flask and bring to volume with deionized water. Pour back into original cup and swirl.

10.2.6. Let stand 5-10 minutes for full color development.

10.2.7. Read calibration standards, QC samples, and samples on spectrophotometer at 540 nm. Analyze the calibration check standard and calibration check blank immediately following the calibration standards.

10.2.8. Dilute samples that exceed calibration curve using deionized water. Recolorize and read again on spectrophotometer noting dilution factor.

10.2.9. Continue analyzing samples and quality control samples by first reading the sample followed by its background sample.

10.2.10. After every 10 samples and after reading the last sample in the batch, verify the calibration by reading Calibration Check Verification (CCV) and Continuing Calibration Blank (CCB).

11. Calculations / Data Reduction

11.1. Accuracy:

$$\text{ICV / CCV, LCS \% Recovery} = \frac{\text{observed concentration}}{\text{known concentration}} \times 100$$

11.2. Precision (RPD):

$$\text{Matrix Duplicate (MD)} = \frac{|\text{orig. sample value - dup. sample value}|}{[(\text{orig. sample value + dup. sample value})/2]} \times 100$$

11.3. Calculate the hexavalent chromium result for each aqueous sample as follows:

Hexavalent Chromium in ug/L = A x E

A = Concentration from the calibration curve in ug/L
E = Dilution (if necessary)

11.4. Trivalent Chromium Calculation: When required, the calculation for trivalent chromium is completed using the TALS method '7196a_CR3' which must be logged for the samples being processed along with methods for total chromium (metals) and hexavalent chromium. Upon completion of the total and hexavalent chromium analyses the analyst may process the trivalent chromium results as follows:

- To have TALs perform an automated trivalent chromium calculation: on the trivalent chromium batch information page enter '1' (for 'yes') in response to the

prompt to automatically perform the calculation. As long as both methods are at 2nd level review status in TALS the program will automatically pull in the metals results for chromium and the hexavalent chromium results to perform the automated calculation. TALS will display an 'ok' message on the worksheet tab if the Cr6 result is less than Total Cr result.

- If the analyst intends to manually enter trivalent chromium results an entry of '0' (for 'no') should be made the and the results entered manually.

11.5. Data Reduction

11.5.1. Record analytical data directly into TALS and in the 'Hexachrome Run Logbook.'

11.5.2. Record Reagent information on the "batch information" page.

11.5.3. Special comments or observations should be recorded on the "worksheet" tab. Record Sample absorbances and pH readings on the "worksheet" tab.

11.5.4. Attach the calibration curve plot and the logbook pages for the analysis to the analytical batch as pdf files.

11.5.5. Complete the Data Review Checker (DRC) in TALS: Prior to data submission the analyst must execute the DRC (Data Review Checker) program in the applicable analytical batch (TALS Analyst Desktop II module).

11.5.5.1 Open the analytical batch and click on the Edit tab above to enter the Edit Mode.

11.5.5.2 Press F8 command key on the computer keyboard or right click anywhere on the batch worksheet then click on 'Run checklist.'

11.5.5.3 Acknowledge by filling in responses to all unacknowledged findings. Highlight the checklist with findings, then highlight the associated list of findings; right click and choose 'Acknowledge Item.'

11.5.5.4 Fill in appropriate comments in the response box, then hit 'OK.' 'The column labeled 'unacknowledged findings' should show '0' for all questions once completed; then batch is ready for 2nd level review.

12. Method Performance

12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 19 (Test Methods and Method Validation) of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value

determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

12.2. Demonstration of Capabilities

For DOC procedure refer to Section 19 in the most current revision of Eurofins Edison's Quality Assurance Manual (ED-QA-LQM).

12.3. Training Requirements

Refer to Eurofins Edison SOP No. ED-GEN-022, Training, for the laboratory's training program.

13.0 Pollution Control

It is Eurofins 's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

14.0. Waste Management

14.1. Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to Eurofins Edison SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).

14.2. The following waste streams are generated as a result of this analysis:

- Water Retain Samples -These materials are originally received aqueous samples. These solutions are then transferred into the first drum of the neutralization system and neutralized to a pH of 6 – 9 with sodium bicarbonate (Siedler Chemical SC-0219-25). This solution is discharged into the municipal sewer system.

15.0. References / Cross-References

15.1. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed., U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW-846, Method 7196A.

15.2. TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.

15.3. TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.

15.4. TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.

15.5. TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

16.0. Method Modifications:

N/A

17.0. Attachments

N/A

18.0. Revision History

- Revision 12, 23 September 2024
  - Sec. 10.1.5: Added requirements and procedures to evaluate %RE in the calibration
- Revision 11, 10/01/2021
  - Sec. 9.2.4: Added criteria to analyze a Minimum Reporting Level (MRL) daily.
- Revision 10, 14 May 2020
  - Updated SOP header with Eurofins logo.
  - Sec 1.1: Clarified matrix applicability for this SOP.
  - Sec. 9.1: Revised the frequency of Matrix Duplicate from 1/20 samples to 1/10 samples.
- Revision 9, 12 February 2019
  - Sec. 9.1.4: Changed LCS source vendor from ERA to Phenova.
  - Sec. 11.5.5: Replaced Wetchemistry Data review checklist (EDS-WI-008) to TALS Data Review Checker (DRC); instructions added.
  - Deleted Sec 15.6, WI EDS-WI-008, not applicable; subsequent sections adjusted accordingly.
- Revision 8, 30 Nov 2016
  - Sec. 11.4: Added Cr3 calc procedure
- Revision 7, 26 November 2012
  - Section 9.1.2: Revised the minimum concentration for PS from 15 ug/L to 30 ug/L to comply with Method 7196A.
- Revision 6, 04 October 2012
  - Section 1&12: Updated LQM reference to reflect the most current LQM revision.
  - Sec 5: Updated to comply with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP)

- Sec. 9.1.1: Revised to include corrective action procedure when MB is greater than the RL.
- Sec. 9.1.4: Changed limits from 85-115% to vendor specified QC limits.
- Sec. 9.2.2 & 9.2.3: Revised to include corrective action procedure when CCV and CCB are outside the acceptance limits.

- Revision 5, 7 September 2010
  - Section 2.0: Removed text 'Dissolved;' this SOP is only applicable to aqueous samples for method SW7196A which are not filtered.
  - Sec 3: Revised to update LQM reference for the list of definitions.
  - Section 6.2: Added graduated cylinders (class A) and transfer pipettes to the list of supplies.
  - Removed Nitric acid on list of reagents, not applicable for the analysis of Cr6+ waters.
  - Section 9.1.1: Revised method blank criteria from <MDL to <RL.
  - Section 9.1.4: Replaced LCS vendor specified limits to 85-115%.
  - Section 9.2.2: Removed text: "immediately after analyzing the calibration standards." Renamed CCS to CCV.
  - Section 9.2.3: Revised CCB criteria from <MDL to <RL.
  - Section 10.1.4: Revised the correlation coefficient criteria from ≥0.997 to ≥0.995.
  - Section 13 & 14: Revised to comply with the current Method SOP format (CW-QS-002, Writing a Standard Operating Procedure (SOP)).
  - Section 11.4: Revised Data Reduction section in accordance with TALS.
  - Section 15: Added applicable references.

- Revision 4, January 22, 2008
  - Updated to new format as per TestAmerica's SOP format.
  - Added the shelf-life and storage requirements to reagents in section 7, Reagents and standards.
  - Added J.T.Baker (Catalog Nos. 3090, 3093 or equivalent) as source of primary and secondary standards. Deleted reference to Inorganic Ventures as a source of the primary standard.
  - Replaced the source standard used for the preparation of 10 mg/L Cr+6 (primary and secondary spiking solution) from 100 mg/L Cr+6 to 1000 mg/L Cr+6.
  - Added the control limits for Post Verification Spike (PVS).
  - Added ICV as Instrument QC.
  - Added the preparation procedure and control limits for the Calibration Check Standard (CCS).
  - Deleted Matrix spike (matrix spike low and matrix spike high) as sample QC requirement.
  - Section 10.3.3: Revised pH range from 1.6-2.2 to 1.5-2.5 to reflect method requirements.
  - Standardized all analytical units to ug/L and mg/L.
  - Added the following to the References section: TestAmerica Edison SOP ED-GEN-022 (Training) and TestAmerica Laboratory Quality Manual (ED-QA-LQM).

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Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL
Solid					
Volatile Organic Compounds by GC/MS	8260D	5035A_FW (low level prep)	1,1,1-Trichloroethane	71-55-6	0.00100
			1,1,2,2-Tetrachloroethane	79-34-5	0.00100
			1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.00100
			1,1,2-Trichloroethane	79-00-5	0.00100
			1,1-Dichloroethane	75-34-3	0.00100
			1,1-Dichloroethene	75-35-4	0.00100
			1,2,3-Trichlorobenzene	87-61-6	0.00100
			1,2,4-Trichlorobenzene	120-82-1	0.00100
			1,2,4-Trimethylbenzene	95-63-6	0.00100
			1,2-Dibromo-3-Chloropropane	96-12-8	0.00100
			1,2-Dichlorobenzene	95-50-1	0.00100
			1,2-Dichloroethane	107-06-2	0.00100
			1,2-Dichloropropane	78-87-5	0.00100
			1,3,5-Trimethylbenzene	108-67-8	0.00100
			1,3-Dichlorobenzene	541-73-1	0.00100
			1,4-Dichlorobenzene	106-46-7	0.00100
			2-Butanone (MEK)	78-93-3	0.00500
			2-Hexanone	591-78-6	0.00500
			4-Methyl-2-pentanone (MIBK)	108-10-1	0.00500
			Acetone	67-64-1	0.00600
			Benzene	71-43-2	0.00100
			Bromoform	75-25-2	0.00100
			Bromomethane	74-83-9	0.00200
			Carbon disulfide	75-15-0	0.00100
			Carbon tetrachloride	56-23-5	0.00100
			Chlorobenzene	108-90-7	0.00100
			Chlorobromomethane	74-97-5	0.00100
			Chlorodibromomethane	124-48-1	0.00100
			Chloroethane	75-00-3	0.00100
			Chloroform	67-66-3	0.00100
			Chloromethane	74-87-3	0.00100
			cis-1,2-Dichloroethene	156-59-2	0.00100
			cis-1,3-Dichloropropene	10061-01-5	0.00100
			Cyclohexane	110-82-7	0.00100
			Dichlorobromomethane	75-27-4	0.00100
			Dichlorodifluoromethane	75-71-8	0.00100
			Ethylbenzene	100-41-4	0.00100
			Ethylene Dibromide	106-93-4	0.00100
			Isopropylbenzene	98-82-8	0.00100
			Methyl acetate	79-20-9	0.00500

Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL
Solid					
			Methyl tert-butyl ether	1634-04-4	0.00100
			Methylcyclohexane	108-87-2	0.00100
			Methylene Chloride	75-09-2	0.00200
			m-Xylene & p-Xylene	179601-23-1	0.00100
			n-Butylbenzene	104-51-8	0.00100
			N-Propylbenzene	103-65-1	0.00100
			o-Xylene	95-47-6	0.00100
			sec-Butylbenzene	135-98-8	0.00100
			Styrene	100-42-5	0.00100
			tert-Butylbenzene	98-06-6	0.00100
			Tetrachloroethene	127-18-4	0.00100
			Toluene	108-88-3	0.00100
			trans-1,2-Dichloroethene	156-60-5	0.00100
			trans-1,3-Dichloropropene	10061-02-6	0.00100
			Trichloroethene	79-01-6	0.00100
			Trichlorofluoromethane	75-69-4	0.00100
			Vinyl chloride	75-01-4	0.00100
			Xylenes, Total	1330-20-7	0.00200
			1,2-Dichloroethane-d4 (Surr)	17060-07-0	
			4-Bromofluorobenzene	460-00-4	
			Dibromofluoromethane (Surr)	1868-53-7	
			Toluene-d8 (Surr)	2037-26-5	
Volatile Organic Compounds by GC/MS	8260D	5035A_M (medium level prep)	1,1,1-Trichloroethane	71-55-6	0.0500
			1,1,2,2-Tetrachloroethane	79-34-5	0.0500
			1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	0.0500
			1,1,2-Trichloroethane	79-00-5	0.0500
			1,1-Dichloroethane	75-34-3	0.0500
			1,1-Dichloroethene	75-35-4	0.0500
			1,2,3-Trichlorobenzene	87-61-6	0.0500
			1,2,4-Trichlorobenzene	120-82-1	0.0500
			1,2,4-Trimethylbenzene	95-63-6	0.0500
			1,2-Dibromo-3-Chloropropane	96-12-8	0.0500
			1,2-Dichlorobenzene	95-50-1	0.0500
			1,2-Dichloroethane	107-06-2	0.0500
			1,2-Dichloropropane	78-87-5	0.0500
			1,3,5-Trimethylbenzene	108-67-8	0.0500
			1,3-Dichlorobenzene	541-73-1	0.0500
			1,4-Dichlorobenzene	106-46-7	0.0500
			2-Butanone (MEK)	78-93-3	0.250
			2-Hexanone	591-78-6	0.250
			4-Methyl-2-pentanone (MIBK)	108-10-1	0.250

Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL
Solid					
			Acetone	67-64-1	0.250
			Benzene	71-43-2	0.0500
			Bromoform	75-25-2	0.0500
			Bromomethane	74-83-9	0.0500
			Carbon disulfide	75-15-0	0.0500
			Carbon tetrachloride	56-23-5	0.0500
			Chlorobenzene	108-90-7	0.0500
			Chlorobromomethane	74-97-5	0.0500
			Chlorodibromomethane	124-48-1	0.0500
			Chloroethane	75-00-3	0.0500
			Chloroform	67-66-3	0.0500
			Chloromethane	74-87-3	0.0500
			cis-1,2-Dichloroethene	156-59-2	0.0500
			cis-1,3-Dichloropropene	10061-01-5	0.0500
			Cyclohexane	110-82-7	0.0500
			Dichlorobromomethane	75-27-4	0.0500
			Dichlorodifluoromethane	75-71-8	0.0500
			Ethylbenzene	100-41-4	0.0500
			Ethylene Dibromide	106-93-4	0.0500
			Isopropylbenzene	98-82-8	0.0500
			Methyl acetate	79-20-9	0.250
			Methyl tert-butyl ether	1634-04-4	0.0500
			Methylcyclohexane	108-87-2	0.0500
			Methylene Chloride	75-09-2	0.0500
			m-Xylene & p-Xylene	179601-23-1	0.0500
			n-Butylbenzene	104-51-8	0.0500
			N-Propylbenzene	103-65-1	0.0500
			o-Xylene	95-47-6	0.0500
			sec-Butylbenzene	135-98-8	0.0500
			Styrene	100-42-5	0.0500
			tert-Butylbenzene	98-06-6	0.0500
			Tetrachloroethene	127-18-4	0.0500
			Toluene	108-88-3	0.0500
			trans-1,2-Dichloroethene	156-60-5	0.0500
			trans-1,3-Dichloropropene	10061-02-6	0.0500
			Trichloroethene	79-01-6	0.0500
			Trichlorofluoromethane	75-69-4	0.0500
			Vinyl chloride	75-01-4	0.0500
			Xylenes, Total	1330-20-7	0.100
			1,2-Dichloroethane-d4 (Surr)	17060-07-0	
			4-Bromofluorobenzene	460-00-4	
			Dibromofluoromethane (Surr)	1868-53-7	

Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL
Solid					
			Toluene-d8 (Surr)	2037-26-5	
Semivolatile Organic Compounds (GC/MS)	8270E	3546	1,1'-Biphenyl	92-52-4	0.330
			1,2,4,5-Tetrachlorobenzene	95-94-3	0.330
			1,4-Dioxane	123-91-1	0.0330
			2,2'-oxybis[1-chloropropane]	108-60-1	0.330
			2,3,4,6-Tetrachlorophenol	58-90-2	0.330
			2,4,5-Trichlorophenol	95-95-4	0.330
			2,4,6-Trichlorophenol	88-06-2	0.133
			2,4-Dichlorophenol	120-83-2	0.133
			2,4-Dimethylphenol	105-67-9	0.330
			2,4-Dinitrophenol	51-28-5	0.266
			2,4-Dinitrotoluene	121-14-2	0.0670
			2,6-Dinitrotoluene	606-20-2	0.0670
			2-Chloronaphthalene	91-58-7	0.330
			2-Chlorophenol	95-57-8	0.330
			2-Methylnaphthalene	91-57-6	0.330
			2-Methylphenol	95-48-7	0.330
			2-Nitroaniline	88-74-4	0.330
			2-Nitrophenol	88-75-5	0.330
			3 & 4 Methylphenol	15831-10-4	0.330
			3,3'-Dichlorobenzidine	91-94-1	0.133
			3-Nitroaniline	99-09-2	0.330
			4,6-Dinitro-2-methylphenol	534-52-1	0.266
			4-Bromophenyl phenyl ether	101-55-3	0.330
			4-Chloro-3-methylphenol	59-50-7	0.330
			4-Chloroaniline	106-47-8	0.330
			4-Chlorophenyl phenyl ether	7005-72-3	0.330
			4-Methylphenol	106-44-5	0.330
			4-Nitroaniline	100-01-6	0.330
			4-Nitrophenol	100-02-7	0.670
			Acenaphthene	83-32-9	0.330
			Acenaphthylene	208-96-8	0.330
			Acetophenone	98-86-2	0.330
			Anthracene	120-12-7	0.330
			Atrazine	1912-24-9	0.133
			Benzaldehyde	100-52-7	0.330
			Benzo[a]anthracene	56-55-3	0.0330
			Benzo[a]pyrene	50-32-8	0.0330
			Benzo[b]fluoranthene	205-99-2	0.0330
			Benzo[g,h,i]perylene	191-24-2	0.330
			Benzo[k]fluoranthene	207-08-9	0.0330

Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL
Solid					
			Bis(2-chloroethoxy)methane	111-91-1	0.330
			Bis(2-chloroethyl)ether	111-44-4	0.0330
			Bis(2-ethylhexyl) phthalate	117-81-7	0.330
			Butyl benzyl phthalate	85-68-7	0.330
			Caprolactam	105-60-2	0.330
			Carbazole	86-74-8	0.330
			Chrysene	218-01-9	0.330
			Dibenz(a,h)anthracene	53-70-3	0.0330
			Dibenzofuran	132-64-9	0.330
			Diethyl phthalate	84-66-2	0.330
			Dimethyl phthalate	131-11-3	0.330
			Di-n-butyl phthalate	84-74-2	0.330
			Di-n-octyl phthalate	117-84-0	0.330
			Fluoranthene	206-44-0	0.330
			Fluorene	86-73-7	0.330
			Hexachlorobenzene	118-74-1	0.0330
			Hexachlorobutadiene	87-68-3	0.0670
			Hexachlorocyclopentadiene	77-47-4	0.330
			Hexachloroethane	67-72-1	0.0330
			Indeno[1,2,3-cd]pyrene	193-39-5	0.0330
			Isophorone	78-59-1	0.133
			Naphthalene	91-20-3	0.330
			Nitrobenzene	98-95-3	0.0330
			N-Nitrosodi-n-propylamine	621-64-7	0.0330
			N-Nitrosodiphenylamine	86-30-6	0.330
			Pentachlorophenol	87-86-5	0.266
			Phenanthrene	85-01-8	0.330
			Phenol	108-95-2	0.330
			Pyrene	129-00-0	0.330
			2,4,6-Tribromophenol (Surr)	118-79-6	
			2-Fluorobiphenyl	321-60-8	
			2-Fluorophenol (Surr)	367-12-4	
			Nitrobenzene-d5 (Surr)	4165-60-0	
			Phenol-d5 (Surr)	4165-62-2	
			Terphenyl-d14 (Surr)	1718-51-0	

Organochlorine Pesticides (GC)	8081B	3546	4,4'-DDD	72-54-8	0.00670
			4,4'-DDE	72-55-9	0.00670
			4,4'-DDT	50-29-3	0.00670
			Aldrin	309-00-2	0.00670
			alpha-BHC	319-84-6	0.00200
			beta-BHC	319-85-7	0.00200

Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL
Solid					
			Chlordane (technical)	12789-03-6	0.0670
			cis-Chlordane	5103-71-9	0.00670
			delta-BHC	319-86-8	0.00200
			Dieldrin	60-57-1	0.00200
			Endosulfan I	959-98-8	0.00670
			Endosulfan II	33213-65-9	0.00670
			Endosulfan sulfate	1031-07-8	0.00670
			Endrin	72-20-8	0.00670
			Endrin aldehyde	7421-93-4	0.00670
			Endrin ketone	53494-70-5	0.00670
			gamma-BHC (Lindane)	58-89-9	0.00200
			Heptachlor	76-44-8	0.00670
			Heptachlor epoxide	1024-57-3	0.00670
			Methoxychlor	72-43-5	0.00670
			Toxaphene	8001-35-2	0.0670
			DCB Decachlorobiphenyl	2051-24-3	
			Tetrachloro-m-xylene	877-09-8	
Polychlorinated Biphenyls (PCBs) by Gas Chromatography	8082A	3546	Aroclor 1016	12674-11-2	0.0670
			Aroclor 1221	11104-28-2	0.0670
			Aroclor 1232	11141-16-5	0.0670
			Aroclor 1242	53469-21-9	0.0670
			Aroclor 1248	12672-29-6	0.0670
			Aroclor 1254	11097-69-1	0.0670
			Aroclor 1260	11096-82-5	0.0670
			Aroclor-1262	37324-23-5	0.0670
			Aroclor 1268	11100-14-4	0.0670
			Polychlorinated biphenyls, Total	1336-36-3	0.0670
			DCB Decachlorobiphenyl	2051-24-3	
			Tetrachloro-m-xylene	877-09-8	
Herbicides (GC)	8151A	8151A_SP	2,4,5-T	93-76-5	0.0333
			2,4-D	94-75-7	0.0333
			Silvex (2,4,5-TP)	93-72-1	0.0333
			2,4-Dichlorophenylacetic acid	19719-28-9	
Metals (ICP/MS)	6020B	3050B	Aluminum	7429-90-5	20.0
			Antimony	7440-36-0	1.00
			Arsenic	7440-38-2	1.00
			Barium	7440-39-3	2.00
			Beryllium	7440-41-7	0.400
			Cadmium	7440-43-9	1.00

Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL
Solid					
			Calcium	7440-70-2	100
			Chromium	7440-47-3	2.00
			Cobalt	7440-48-4	2.00
			Copper	7440-50-8	2.00
			Iron	7439-89-6	60.0
			Lead	7439-92-1	0.600
			Magnesium	7439-95-4	100
			Manganese	7439-96-5	4.00
			Nickel	7440-02-0	2.00
			Potassium	7440-09-7	100
			Selenium	7782-49-2	1.25
			Silver	7440-22-4	0.400
			Sodium	7440-23-5	100
			Thallium	7440-28-0	0.400
			Vanadium	7440-62-2	2.00
			Zinc	7440-66-6	8.00
Mercury (CVAA)	7471B	7471B_Prep	Mercury	7439-97-6	0.0170
Chromium, Hexavalent	7196A	3060A	Cr (VI)	18540-29-9	2.00
Chromium, Trivalent (Colorimetric)	7196A_CR3		Cr (III)	16065-83-1	2.00
Cyanide, Total andor Amenable	9012B	9012B_Prep	Cyanide, Total	57-12-5	0.240
Percent Moisture	Moisture		Percent Moisture	STL00177	1.00
			Percent Solids	STL00234	1.00
Ground Water					
Volatile Organic Compounds by GC/MS	8260D	5030C	1,1,1-Trichloroethane	71-55-6	1.00
			1,1,2,2-Tetrachloroethane	79-34-5	1.00
			1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	1.00
			1,1,2-Trichloroethane	79-00-5	1.00
			1,1-Dichloroethane	75-34-3	1.00
			1,1-Dichloroethene	75-35-4	1.00
			1,2,3-Trichlorobenzene	87-61-6	1.00
			1,2,4-Trichlorobenzene	120-82-1	1.00
			1,2,4-Trimethylbenzene	95-63-6	1.00
			1,2-Dibromo-3-Chloropropane	96-12-8	1.00
			1,2-Dichlorobenzene	95-50-1	1.00
			1,2-Dichloroethane	107-06-2	1.00
			1,2-Dichloropropane	78-87-5	1.00
			1,3,5-Trimethylbenzene	108-67-8	1.00

Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL
Solid					
			1,3-Dichlorobenzene	541-73-1	1.00
			1,4-Dichlorobenzene	106-46-7	1.00
			2-Butanone (MEK)	78-93-3	5.00
			2-Hexanone	591-78-6	5.00
			4-Methyl-2-pentanone (MIBK)	108-10-1	5.00
			Acetone	67-64-1	5.00
			Benzene	71-43-2	1.00
			Bromoform	75-25-2	1.00
			Bromomethane	74-83-9	1.00
			Carbon disulfide	75-15-0	1.00
			Carbon tetrachloride	56-23-5	1.00
			Chlorobenzene	108-90-7	1.00
			Chlorobromomethane	74-97-5	1.00
			Chlorodibromomethane	124-48-1	1.00
			Chloroethane	75-00-3	1.00
			Chloroform	67-66-3	1.00
			Chloromethane	74-87-3	1.00
			cis-1,2-Dichloroethene	156-59-2	1.00
			cis-1,3-Dichloropropene	10061-01-5	1.00
			Cyclohexane	110-82-7	1.00
			Dichlorobromomethane	75-27-4	1.00
			Dichlorodifluoromethane	75-71-8	1.00
			Ethylbenzene	100-41-4	1.00
			Ethylene Dibromide	106-93-4	1.00
			Isopropylbenzene	98-82-8	1.00
			Methyl acetate	79-20-9	5.00
			Methyl tert-butyl ether	1634-04-4	1.00
			Methylcyclohexane	108-87-2	1.00
			Methylene Chloride	75-09-2	1.00
			m-Xylene & p-Xylene	179601-23-1	1.00
			n-Butylbenzene	104-51-8	1.00
			N-Propylbenzene	103-65-1	1.00
			o-Xylene	95-47-6	1.00
			sec-Butylbenzene	135-98-8	1.00
			Styrene	100-42-5	1.00
			tert-Butylbenzene	98-06-6	1.00
			Tetrachloroethene	127-18-4	1.00
			Toluene	108-88-3	1.00
			trans-1,2-Dichloroethene	156-60-5	1.00
			trans-1,3-Dichloropropene	10061-02-6	1.00
			Trichloroethene	79-01-6	1.00
			Trichlorofluoromethane	75-69-4	1.00

Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL
Solid					
			Vinyl chloride	75-01-4	1.00
			Xylenes, Total	1330-20-7	2.00
			1,2-Dichloroethane-d4 (Surr)	17060-07-0	
			4-Bromofluorobenzene	460-00-4	
			Dibromofluoromethane (Surr)	1868-53-7	
			Toluene-d8 (Surr)	2037-26-5	
Semivolatile Organic Compounds (GC/MS)	8270E	3510C_LVI	1,1'-Biphenyl	92-52-4	10.0
			1,2,4,5-Tetrachlorobenzene	95-94-3	10.0
			2,2'-oxybis[1-chloropropane]	108-60-1	10.0
			2,3,4,6-Tetrachlorophenol	58-90-2	10.0
			2,4,5-Trichlorophenol	95-95-4	10.0
			2,4,6-Trichlorophenol	88-06-2	10.0
			2,4-Dichlorophenol	120-83-2	10.0
			2,4-Dimethylphenol	105-67-9	10.0
			2,4-Dinitrophenol	51-28-5	40.0
			2,4-Dinitrotoluene	121-14-2	10.0
			2,6-Dinitrotoluene	606-20-2	2.00
			2-Chloronaphthalene	91-58-7	10.0
			2-Chlorophenol	95-57-8	10.0
			2-Methylnaphthalene	91-57-6	10.0
			2-Methylphenol	95-48-7	10.0
			2-Nitroaniline	88-74-4	10.0
			2-Nitrophenol	88-75-5	10.0
			3 & 4 Methylphenol	15831-10-4	10.0
			3,3'-Dichlorobenzidine	91-94-1	10.0
			3-Nitroaniline	99-09-2	10.0
			4,6-Dinitro-2-methylphenol	534-52-1	20.0
			4-Bromophenyl phenyl ether	101-55-3	10.0
			4-Chloro-3-methylphenol	59-50-7	10.0
			4-Chloroaniline	106-47-8	10.0
			4-Chlorophenyl phenyl ether	7005-72-3	10.0
			4-Methylphenol	106-44-5	10.0
			4-Nitroaniline	100-01-6	10.0
			4-Nitrophenol	100-02-7	20.0
			Acenaphthene	83-32-9	10.0
			Acenaphthylene	208-96-8	10.0
			Acetophenone	98-86-2	10.0
			Anthracene	120-12-7	10.0
			Atrazine	1912-24-9	2.00
			Benzaldehyde	100-52-7	10.0
			Benzo[a]anthracene	56-55-3	1.00

Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL
Solid					
			Benzo[a]pyrene	50-32-8	1.00
			Benzo[b]fluoranthene	205-99-2	2.00
			Benzo[g,h,i]perylene	191-24-2	10.0
			Benzo[k]fluoranthene	207-08-9	1.00
			Bis(2-chloroethoxy)methane	111-91-1	10.0
			Bis(2-chloroethyl)ether	111-44-4	1.00
			Bis(2-ethylhexyl) phthalate	117-81-7	2.00
			Butyl benzyl phthalate	85-68-7	10.0
			Caprolactam	105-60-2	10.0
			Carbazole	86-74-8	10.0
			Chrysene	218-01-9	2.00
			Dibenz(a,h)anthracene	53-70-3	1.00
			Dibenzofuran	132-64-9	10.0
			Diethyl phthalate	84-66-2	10.0
			Dimethyl phthalate	131-11-3	10.0
			Di-n-butyl phthalate	84-74-2	10.0
			Di-n-octyl phthalate	117-84-0	10.0
			Fluoranthene	206-44-0	10.0
			Fluorene	86-73-7	10.0
			Hexachlorobenzene	118-74-1	1.00
			Hexachlorobutadiene	87-68-3	1.00
			Hexachlorocyclopentadiene	77-47-4	10.0
			Hexachloroethane	67-72-1	2.00
			Indeno[1,2,3-cd]pyrene	193-39-5	2.00
			Isophorone	78-59-1	10.0
			Naphthalene	91-20-3	2.00
			Nitrobenzene	98-95-3	1.00
			N-Nitrosodi-n-propylamine	621-64-7	1.00
			N-Nitrosodiphenylamine	86-30-6	10.0
			Pentachlorophenol	87-86-5	20.0
			Phenanthrene	85-01-8	10.0
			Phenol	108-95-2	10.0
			Pyrene	129-00-0	10.0
			2,4,6-Tribromophenol (Surr)	118-79-6	
			2-Fluorobiphenyl	321-60-8	
			2-Fluorophenol (Surr)	367-12-4	
			Nitrobenzene-d5 (Surr)	4165-60-0	
			Phenol-d5 (Surr)	4165-62-2	
			Terphenyl-d14 (Surr)	1718-51-0	

Semivolatile Organic Compounds (GC/MS SIM / Isotope Dilution)	8270E_SIM_MS_ID	3510C_LVI	1,4-Dioxane	123-91-1	0.200
			1,4-Dioxane-d8	17647-74-4	0.400

Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL
Solid					
Organochlorine Pesticides (GC)	8081B	3510C_LVI	4,4'-DDD	72-54-8	0.0200
			4,4'-DDE	72-55-9	0.0200
			4,4'-DDT	50-29-3	0.0200
			Aldrin	309-00-2	0.0200
			alpha-BHC	319-84-6	0.0200
			beta-BHC	319-85-7	0.0200
			Chlordane (technical)	12789-03-6	0.500
			cis-Chlordane	5103-71-9	0.0200
			delta-BHC	319-86-8	0.0200
			Dieldrin	60-57-1	0.0200
			Endosulfan I	959-98-8	0.0200
			Endosulfan II	33213-65-9	0.0200
			Endosulfan sulfate	1031-07-8	0.0200
			Endrin	72-20-8	0.0200
			Endrin aldehyde	7421-93-4	0.0200
			Endrin ketone	53494-70-5	0.0200
			gamma-BHC (Lindane)	58-89-9	0.0200
			Heptachlor	76-44-8	0.0200
			Heptachlor epoxide	1024-57-3	0.0200
			Methoxychlor	72-43-5	0.0200
			Toxaphene	8001-35-2	0.500
			DCB Decachlorobiphenyl	2051-24-3	
			Tetrachloro-m-xylene	877-09-8	
Polychlorinated Biphenyls (PCBs) by Gas Chromatography	8082A	3510C_LVI	Aroclor 1016	12674-11-2	0.400
			Aroclor 1221	11104-28-2	0.400
			Aroclor 1232	11141-16-5	0.400
			Aroclor 1242	53469-21-9	0.400
			Aroclor 1248	12672-29-6	0.400
			Aroclor 1254	11097-69-1	0.400
			Aroclor 1260	11096-82-5	0.400
			Aroclor-1262	37324-23-5	0.400
			Aroclor 1268	11100-14-4	0.400
			Polychlorinated biphenyls, Total	1336-36-3	0.400
			DCB Decachlorobiphenyl	2051-24-3	
			Tetrachloro-m-xylene	877-09-8	
Herbicides (GC)	8151A	8151A_AP	2,4,5-T	93-76-5	1.20
			2,4-D	94-75-7	1.20
			Silvex (2,4,5-TP)	93-72-1	1.20
			2,4-Dichlorophenylacetic acid	19719-28-9	

Method Description	Method Code	Prep Method	Analyte Description	CAS Number	RL
Solid					
Chromium, Hexavalent	7196A		Cr (VI)	18540-29-9	10.0
Chromium, Trivalent (Colorimetric)	7196A_CR3		Cr (III)	16065-83-1	10.0
Cyanide, Total	9012B	9012B_Prep	Cyanide, Total	57-12-5	0.0100
Metals (ICP/MS)	6020B	3005A	Aluminum	7429-90-5	40.0
			Antimony	7440-36-0	2.00
			Arsenic	7440-38-2	2.00
			Barium	7440-39-3	4.00
			Beryllium	7440-41-7	0.800
			Cadmium	7440-43-9	2.00
			Calcium	7440-70-2	500
			Chromium	7440-47-3	4.00
			Cobalt	7440-48-4	4.00
			Copper	7440-50-8	4.00
			Iron	7439-89-6	120
			Lead	7439-92-1	1.20
			Magnesium	7439-95-4	200
			Manganese	7439-96-5	8.00
			Nickel	7440-02-0	4.00
			Potassium	7440-09-7	200
			Selenium	7782-49-2	2.50
			Silver	7440-22-4	2.00
			Sodium	7440-23-5	500
			Thallium	7440-28-0	0.800
			Vanadium	7440-62-2	4.00
			Zinc	7440-66-6	16.0
Mercury (CVAA)	7470A	7470A_Prep	Mercury	7439-97-6	0.200

MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
0.000233		mg/Kg	78	120	30	78	120	30		
0.000214		mg/Kg	70	133	30	70	133	30		
0.000301		mg/Kg	75	121	30	75	121	30		
0.000178		mg/Kg	80	120	30	80	120	30		
0.000206		mg/Kg	72	120	30	72	120	30		
0.000225		mg/Kg	76	120	30	76	120	30		
0.000181		mg/Kg	77	137	30	77	137	30		
0.000358		mg/Kg	77	136	30	77	136	30		
0.000246		mg/Kg	75	120	30	75	120	30		
0.000460		mg/Kg	80	124	30	80	124	30		
0.000361		mg/Kg	80	120	30	80	120	30		
0.000296		mg/Kg	70	123	30	70	123	30		
0.000423		mg/Kg	73	124	30	73	124	30		
0.000314		mg/Kg	70	120	30	70	120	30		
0.000365		mg/Kg	80	120	30	80	120	30		
0.000225		mg/Kg	80	120	30	80	120	30		
0.000368		mg/Kg	64	128	30	64	128	30		
0.00171		mg/Kg	75	120	30	75	120	30		
0.00156		mg/Kg	80	120	30	80	120	30		
0.00572		mg/Kg	58	122	30	58	122	30		
0.000258		mg/Kg	75	120	30	75	120	30		
0.000425		mg/Kg	61	125	30	61	125	30		
0.00100		mg/Kg	37	150	30	37	150	30		
0.000266		mg/Kg	57	133	30	57	133	30		
0.000387		mg/Kg	66	127	30	66	127	30		
0.000177		mg/Kg	80	120	30	80	120	30		
0.000281		mg/Kg	76	127	30	76	127	30		
0.000194		mg/Kg	72	120	30	72	120	30		
0.000522		mg/Kg	60	123	30	60	123	30		
0.000971		mg/Kg	79	126	30	79	126	30		
0.000435		mg/Kg	46	122	30	46	122	30		
0.000358		mg/Kg	80	123	30	80	123	30		
0.000273		mg/Kg	75	120	30	75	120	30		
0.000221		mg/Kg	65	132	30	65	132	30		
0.000257		mg/Kg	77	124	30	77	124	30		
0.000338		mg/Kg	45	129	30	45	129	30		
0.000199		mg/Kg	80	120	30	80	120	30		
0.000180		mg/Kg	79	120	30	79	120	30		
0.000285		mg/Kg	74	120	30	74	120	30		
0.00430		mg/Kg	57	120	30	57	120	30		

MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
0.000512		mg/Kg	74	125	30	74	125	30		
0.000499		mg/Kg	66	125	30	66	125	30		
0.00115		mg/Kg	78	120	30	78	120	30		
0.000174		mg/Kg	80	120	30	80	120	30		
0.000294		mg/Kg	72	120	30	72	120	30		
0.000175		mg/Kg	68	120	30	68	120	30		
0.000194		mg/Kg	80	120	30	80	120	30		
0.000288		mg/Kg	78	120	30	78	120	30		
0.000278		mg/Kg	80	120	30	80	120	30		
0.000276		mg/Kg	80	120	30	80	120	30		
0.000305		mg/Kg	73	120	30	73	120	30		
0.000234		mg/Kg	80	120	30	80	120	30		
0.000246		mg/Kg	78	120	30	78	120	30		
0.000266		mg/Kg	77	120	30	77	120	30		
0.000321		mg/Kg	80	120	30	80	120	30		
0.000406		mg/Kg	61	130	30	61	130	30		
0.000546		mg/Kg	54	122	30	54	122	30		
0.000174		mg/Kg	80	120	30	80	120	30		
		mg/Kg			30			30	65	138
		mg/Kg			30			30	71	128
		mg/Kg							50	150
		mg/Kg			30			30	71	126
0.0140		mg/Kg	76	120	30	76	120	30		
0.00991		mg/Kg	72	120	30	72	120	30		
0.0170		mg/Kg	79	120	30	79	120	30		
0.0102		mg/Kg	80	120	30	80	120	30		
0.0120		mg/Kg	70	120	30	70	120	30		
0.0132		mg/Kg	69	120	30	69	120	30		
0.0175		mg/Kg	80	137	30	80	137	30		
0.0135		mg/Kg	80	134	30	80	134	30		
0.0115		mg/Kg	75	120	30	75	120	30		
0.0106		mg/Kg	68	134	30	68	134	30		
0.0110		mg/Kg	80	120	30	80	120	30		
0.0125		mg/Kg	72	124	30	72	124	30		
0.00900		mg/Kg	74	120	30	74	120	30		
0.0125		mg/Kg	71	120	30	71	120	30		
0.0165		mg/Kg	80	120	30	80	120	30		
0.0167		mg/Kg	80	120	30	80	120	30		
0.110		mg/Kg	64	121	30	64	121	30		
0.0568		mg/Kg	70	120	30	70	120	30		
0.0651		mg/Kg	64	127	30	64	127	30		

MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
0.221		mg/Kg	52	127	30	52	127	30		
0.0101		mg/Kg	75	120	30	75	120	30		
0.00900		mg/Kg	68	122	30	68	122	30		
0.0277		mg/Kg	16	125	30	16	125	30		
0.0337		mg/Kg	59	127	30	59	127	30		
0.0165		mg/Kg	77	120	30	77	120	30		
0.0120		mg/Kg	80	120	30	80	120	30		
0.0150		mg/Kg	77	120	30	77	120	30		
0.0110		mg/Kg	80	120	30	80	120	30		
0.0185		mg/Kg	61	140	30	61	140	30		
0.0110		mg/Kg	73	120	30	73	120	30		
0.0201		mg/Kg	42	136	30	42	136	30		
0.0130		mg/Kg	73	120	30	73	120	30		
0.0111		mg/Kg	79	120	30	79	120	30		
0.0130		mg/Kg	76	125	30	76	125	30		
0.00750		mg/Kg	77	120	30	77	120	30		
0.0155		mg/Kg	39	140	30	39	140	30		
0.0150		mg/Kg	80	121	30	80	121	30		
0.00950		mg/Kg	80	120	30	80	120	30		
0.0160		mg/Kg	74	125	30	74	125	30		
0.0392		mg/Kg	40	130	30	40	130	30		
0.0107		mg/Kg	77	125	30	77	125	30		
0.0363		mg/Kg	75	127	30	75	127	30		
0.0105		mg/Kg	73	120	30	73	120	30		
0.0140		mg/Kg	79	120	30	79	120	30		
0.0135		mg/Kg	68	120	30	68	120	30		
0.0145		mg/Kg	68	120	30	68	120	30		
0.0160		mg/Kg	80	123	30	80	123	30		
0.0155		mg/Kg	77	126	30	77	126	30		
0.00850		mg/Kg	80	124	30	80	124	30		
0.0140		mg/Kg	80	120	30	80	120	30		
0.0180		mg/Kg	78	120	30	78	120	30		
0.0125		mg/Kg	80	120	30	80	120	30		
0.00900		mg/Kg	70	120	30	70	120	30		
0.0111		mg/Kg	73	120	30	73	120	30		
0.0110		mg/Kg	77	120	30	77	120	30		
0.0160		mg/Kg	61	143	30	61	143	30		
0.0100		mg/Kg	50	131	30	50	131	30		
0.0140		mg/Kg	80	120	30	80	120	30		
		mg/Kg			30			30	60	150
		mg/Kg			30			30	70	150
		mg/Kg			30			30	68	150

MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
		mg/Kg			30			30	64	157
0.0115		mg/Kg	64	120	30	64	120	30		
0.0103		mg/Kg	60	120	30	60	120	30		
0.0289		mg/Kg	27	120	30	27	120	30		
0.0198		mg/Kg	46	126	30	46	126	30		
0.0224		mg/Kg	62	120	30	62	120	30		
0.0337		mg/Kg	64	120	30	64	120	30		
0.0425		mg/Kg	65	120	30	65	120	30		
0.0212		mg/Kg	66	120	30	66	120	30		
0.0395		mg/Kg	46	120	30	46	120	30		
0.163		mg/Kg	41	129	30	41	129	30		
0.0356		mg/Kg	65	124	30	65	124	30		
0.0239		mg/Kg	67	121	30	67	121	30		
0.0153		mg/Kg	64	120	30	64	120	30		
0.0118		mg/Kg	63	120	30	63	120	30		
0.00925		mg/Kg	64	120	30	64	120	30		
0.0124		mg/Kg	63	120	30	63	120	30		
0.0252		mg/Kg	48	120	30	48	120	30		
0.0331		mg/Kg	64	120	30	64	120	30		
0.0206		mg/Kg	61	120	30	61	120	30		
0.0500		mg/Kg	10	120	30	10	120	30		
0.0785		mg/Kg	18	122	30	18	122	30		
0.135		mg/Kg	58	136	30	58	136	30		
0.0131		mg/Kg	60	120	30	60	120	30		
0.0186		mg/Kg	66	120	30	66	120	30		
0.0587		mg/Kg	10	120	30	10	120	30		
0.0117		mg/Kg	62	120	30	62	120	30		
0.0207		mg/Kg	61	120	30	61	120	30		
0.0380		mg/Kg	51	120	30	51	120	30		
0.0539		mg/Kg	42	132	30	42	132	30		
0.00943		mg/Kg	65	120	30	65	120	30		
0.00946		mg/Kg	64	120	30	64	120	30		
0.0162		mg/Kg	61	120	30	61	120	30		
0.0101		mg/Kg	67	120	30	67	120	30		
0.0195		mg/Kg	32	150	30	32	150	30		
0.0547		mg/Kg	28	150	30	28	150	30		
0.0249		mg/Kg	66	120	30	66	120	30		
0.00881		mg/Kg	73	123	30	73	123	30		
0.00856		mg/Kg	70	125	30	70	125	30		
0.00976		mg/Kg	66	120	30	66	120	30		
0.00649		mg/Kg	67	122	30	67	122	30		

MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
0.0258		mg/Kg	62	120	30	62	120	30		
0.0115		mg/Kg	60	120	30	60	120	30		
0.0175		mg/Kg	64	125	30	64	125	30		
0.0155		mg/Kg	62	127	30	62	127	30		
0.0515		mg/Kg	36	150	30	36	150	30		
0.0126		mg/Kg	64	120	30	64	120	30		
0.0139		mg/Kg	67	120	30	67	120	30		
0.0143		mg/Kg	66	128	30	66	128	30		
0.0110		mg/Kg	61	120	30	61	120	30		
0.0107		mg/Kg	63	120	30	63	120	30		
0.0752		mg/Kg	65	120	30	65	120	30		
0.0125		mg/Kg	66	120	30	66	120	30		
0.0175		mg/Kg	61	123	30	61	123	30		
0.0116		mg/Kg	61	120	30	61	120	30		
0.00968		mg/Kg	64	120	30	64	120	30		
0.0157		mg/Kg	66	120	30	66	120	30		
0.00704		mg/Kg	62	120	30	62	120	30		
0.0290		mg/Kg	13	120	30	13	120	30		
0.0114		mg/Kg	61	120	30	61	120	30		
0.0129		mg/Kg	71	137	30	71	137	30		
0.0956		mg/Kg	61	120	30	61	120	30		
0.00572		mg/Kg	63	120	30	63	120	30		
0.0183		mg/Kg	63	120	30	63	120	30		
0.0240		mg/Kg	56	120	30	56	120	30		
0.0272		mg/Kg	63	120	30	63	120	30		
0.0678		mg/Kg	61	126	30	61	126	30		
0.0135		mg/Kg	66	120	30	66	120	30		
0.0122		mg/Kg	63	120	30	63	120	30		
0.00823		mg/Kg	61	121	30	61	121	30		
		mg/Kg			30			30	24	137
		mg/Kg			30			30	38	128
		mg/Kg			30			30	31	133
		mg/Kg			30			30	31	120
		mg/Kg			30			30	35	132
		mg/Kg			30			30	44	147

0.00114		mg/Kg	70	138	30	70	138	30		
0.000790		mg/Kg	68	136	30	68	136	30		
0.00123		mg/Kg	66	149	30	66	149	30		
0.00101		mg/Kg	69	136	30	69	136	30		
0.000680		mg/Kg	72	134	30	72	134	30		
0.000750		mg/Kg	71	130	30	71	130	30		

MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
0.0162		mg/Kg	77	120	30	77	120	30		
0.00106		mg/Kg	69	135	30	69	135	30		
0.000410		mg/Kg	69	140	30	69	140	30		
0.000870		mg/Kg	69	134	30	69	134	30		
0.00102		mg/Kg	69	133	30	69	133	30		
0.00172		mg/Kg	71	137	30	71	137	30		
0.000840		mg/Kg	61	140	30	61	140	30		
0.000960		mg/Kg	67	141	30	67	141	30		
0.00158		mg/Kg	71	136	30	71	136	30		
0.00130		mg/Kg	60	146	30	60	146	30		
0.000620		mg/Kg	72	139	30	72	139	30		
0.000790		mg/Kg	74	138	30	74	138	30		
0.00100		mg/Kg	72	134	30	72	134	30		
0.00153		mg/Kg	60	150	30	60	150	30		
0.0242		mg/Kg	63	120	30	63	120	30		
		mg/Kg						30	17	150
		mg/Kg						30	21	150
0.0178	0.0178	mg/Kg	54	136	30	54	136	30		
0.0178	0.0178	mg/Kg								
0.0178	0.0178	mg/Kg								
0.0178	0.0178	mg/Kg								
0.0178	0.0178	mg/Kg								
0.0178	0.0178	mg/Kg								
0.0178	0.0178	mg/Kg	53	145	30	53	145	30		
0.0178	0.0178	mg/Kg								
0.0178	0.0178	mg/Kg								
0.0178	0.0178	mg/Kg								
		mg/Kg							20	150
		mg/Kg							20	150
0.00708		mg/Kg	69	150	30	69	150	30		
0.0121		mg/Kg	78	150	30	78	150	30		
0.00347		mg/Kg	78	150	30	78	150	30		
		mg/Kg			30			30	41	150
5.49		mg/Kg	80	120	20	75	125	20		
0.146		mg/Kg	80	120	20	75	125	20		
0.103		mg/Kg	80	120	20	75	125	20		
0.145		mg/Kg	80	120	20	75	125	20		
0.0570		mg/Kg	80	120	20	75	125	20		
0.113		mg/Kg	80	120	20	75	125	20		

MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
40.7		mg/Kg	80	120	20	75	125	20		
0.908		mg/Kg	80	120	20	75	125	20		
0.148		mg/Kg	80	120	20	75	125	20		
0.368		mg/Kg	80	120	20	75	125	20		
20.2		mg/Kg	80	120	20	75	125	20		
0.200		mg/Kg	80	120	20	75	125	20		
10.2		mg/Kg	80	120	20	75	125	20		
0.403		mg/Kg	80	120	20	75	125	20		
0.470		mg/Kg	80	120	20	75	125	20		
16.2		mg/Kg	80	120	20	75	125	20		
0.128		mg/Kg	80	120	20	75	125	20		
0.0890		mg/Kg	80	120	20	75	125	20		
45.7		mg/Kg	80	120	20	75	125	20		
0.0410		mg/Kg	80	120	20	75	125	20		
0.206		mg/Kg	80	120	20	75	125	20		
3.05		mg/Kg	80	120	20	75	125	20		
0.00800		mg/Kg	80	120	20	80	120	20		
0.848		mg/Kg								
0.131		mg/Kg	85	115		29	100	40		
		%								
		%								
0.238		ug/L	72	128	30	72	128	30		
0.367		ug/L	63	139	30	63	139	30		
0.311		ug/L	65	142	30	65	142	30		
0.204		ug/L	74	125	30	74	125	30		
0.264		ug/L	73	130	30	73	130	30		
0.264		ug/L	68	133	30	68	133	30		
0.357		ug/L	55	150	30	55	150	30		
0.365		ug/L	67	132	30	67	132	30		
0.374		ug/L	75	125	30	75	125	30		
0.376		ug/L	58	132	30	58	132	30		
0.212		ug/L	80	120	30	80	120	30		
0.430		ug/L	66	129	30	66	129	30		
0.353		ug/L	72	128	30	72	128	30		
0.326		ug/L	75	125	30	75	125	30		

MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
0.342		ug/L	80	120	30	80	120	30		
0.334		ug/L	80	120	30	80	120	30		
1.85		ug/L	65	142	30	65	142	30		
1.14		ug/L	72	134	30	72	134	30		
1.30		ug/L	77	130	30	77	130	30		
4.42		ug/L	60	133	30	60	133	30		
0.203		ug/L	71	126	30	71	126	30		
0.536		ug/L	58	128	30	58	128	30		
0.550		ug/L	33	150	30	33	150	30		
0.821		ug/L	35	150	30	35	150	30		
0.208		ug/L	65	131	30	65	131	30		
0.377		ug/L	80	120	30	80	120	30		
0.412		ug/L	71	134	30	71	134	30		
0.281		ug/L	73	121	30	73	121	30		
0.320		ug/L	54	150	30	54	150	30		
0.326		ug/L	78	125	30	78	125	30		
0.402		ug/L	43	149	30	43	149	30		
0.219		ug/L	78	121	30	78	121	30		
0.222		ug/L	74	125	30	74	125	30		
0.321		ug/L	64	142	30	64	142	30		
0.343		ug/L	76	121	30	76	121	30		
0.311		ug/L	38	144	30	38	144	30		
0.298		ug/L	78	120	30	78	120	30		
0.498		ug/L	79	126	30	79	126	30		
0.336		ug/L	79	125	30	79	125	30		
0.785		ug/L	50	147	30	50	147	30		
0.216		ug/L	72	131	30	72	131	30		
0.707		ug/L	63	138	30	63	138	30		
0.315		ug/L	74	127	30	74	127	30		
0.296		ug/L	78	120	30	78	120	30		
0.324		ug/L	69	135	30	69	135	30		
0.322		ug/L	68	129	30	68	129	30		
0.361		ug/L	78	120	30	78	120	30		
0.367		ug/L	77	129	30	77	129	30		
0.415		ug/L	82	127	30	82	127	30		
0.335		ug/L	78	120	30	78	120	30		
0.249		ug/L	70	127	30	70	127	30		
0.379		ug/L	78	120	30	78	120	30		
0.235		ug/L	70	126	30	70	126	30		
0.223		ug/L	71	127	30	71	127	30		
0.314		ug/L	73	121	30	73	121	30		
0.320		ug/L	62	134	30	62	134	30		

MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
0.171		ug/L	55	144	30	55	144	30		
0.654		ug/L	80	120	30	80	120	30		
		ug/L			30			30	70	128
		ug/L			30			30	76	120
		ug/L			30			30	77	132
		ug/L			30			30	80	120
1.19		ug/L	52	129	30	52	129	30		
1.24		ug/L	40	136	30	40	136	30		
0.629		ug/L	47	133	30	47	133	30		
0.746		ug/L	63	131	30	63	131	30		
0.880		ug/L	63	124	30	63	124	30		
0.857		ug/L	66	131	30	66	131	30		
1.07		ug/L	60	120	30	60	120	30		
0.619		ug/L	37	120	30	37	120	30		
2.63		ug/L	50	148	30	50	148	30		
0.997		ug/L	71	142	30	71	142	30		
0.826		ug/L	71	136	30	71	136	30		
1.18		ug/L	50	129	30	50	129	30		
0.377		ug/L	49	120	30	49	120	30		
0.527		ug/L	42	134	30	42	134	30		
0.671		ug/L	35	120	30	35	120	30		
0.474		ug/L	57	134	30	57	134	30		
0.747		ug/L	62	124	30	62	124	30		
0.644		ug/L	27	120	30	27	120	30		
1.43		ug/L	55	145	30	55	145	30		
1.94		ug/L	51	120	30	51	120	30		
2.99		ug/L	65	145	30	65	145	30		
0.745		ug/L	59	132	30	59	132	30		
0.575		ug/L	54	120	30	54	120	30		
1.88		ug/L	43	120	30	43	120	30		
1.28		ug/L	65	127	30	65	127	30		
0.651		ug/L	28	120	30	28	120	30		
1.22		ug/L	57	135	30	57	135	30		
3.98		ug/L	10	120	30	10	120	30		
1.08		ug/L	62	127	30	62	127	30		
0.823		ug/L	58	122	30	58	122	30		
2.33		ug/L	65	125	30	65	125	30		
1.30		ug/L	67	127	30	67	127	30		
1.35		ug/L	13	150	30	13	150	30		
2.10		ug/L	10	150	30	10	150	30		
0.592		ug/L	71	131	30	71	131	30		

MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
0.405		ug/L	75	148	30	75	148	30		
0.676		ug/L	70	140	30	70	140	30		
0.702		ug/L	52	143	30	52	143	30		
0.674		ug/L	71	140	30	71	140	30		
0.589		ug/L	63	122	30	63	122	30		
0.633		ug/L	61	125	30	61	125	30		
0.804		ug/L	65	144	30	65	144	30		
0.854		ug/L	67	141	30	67	141	30		
2.24		ug/L	10	120	30	10	120	30		
0.679		ug/L	68	132	30	68	132	30		
0.907		ug/L	70	132	30	70	132	30		
0.720		ug/L	53	150	30	53	150	30		
1.10		ug/L	64	125	30	64	125	30		
0.976		ug/L	67	131	30	67	131	30		
0.766		ug/L	67	129	30	67	129	30		
0.840		ug/L	71	139	30	71	139	30		
0.749		ug/L	51	150	30	51	150	30		
0.842		ug/L	69	137	30	69	137	30		
0.912		ug/L	67	125	30	67	125	30		
0.396		ug/L	62	135	30	62	135	30		
0.780		ug/L	10	147	30	10	147	30		
3.64		ug/L	10	135	30	10	135	30		
0.803		ug/L	10	138	30	10	138	30		
0.939		ug/L	59	150	30	59	150	30		
0.798		ug/L	65	128	30	65	128	30		
0.541		ug/L	39	126	30	39	126	30		
0.567		ug/L	66	127	30	66	127	30		
0.430		ug/L	63	133	30	63	133	30		
0.891		ug/L	66	128	30	66	128	30		
1.45		ug/L	60	140	30	60	140	30		
1.28		ug/L	68	126	30	68	126	30		
0.292		ug/L	10	80	30	10	80	30		
1.64		ug/L	60	137	30	60	137	30		
		ug/L			30			30	37	150
		ug/L			30			30	46	139
		ug/L			30			30	16	80
		ug/L			30			30	51	145
		ug/L			30			30	10	56
		ug/L			30			30	13	159
0.0720		ug/L	42	142	20	42	142	20		
		ug/L			20			20	10	150

MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
0.00600		ug/L	69	138	30	69	138	30		
0.00200		ug/L	68	130	30	68	130	30		
0.00400		ug/L	57	1505	30	57	150	30		
0.00300		ug/L	67	129	30	67	129	30		
0.00700		ug/L	70	125	30	70	125	30		
0.0150		ug/L	70	129	30	70	129	30		
0.0550		ug/L	45	120	30	45	120	30		
0.00200		ug/L	72	126	30	72	126	30		
0.00500		ug/L	44	120	30	44	120	30		
0.00300		ug/L	72	128	30	72	128	30		
0.00200		ug/L	73	127	30	73	127	30		
0.00400		ug/L	73	134	30	73	134	30		
0.00600		ug/L	61	128	30	61	128	30		
0.00400		ug/L	64	138	30	64	138	30		
0.00800		ug/L	74	133	30	74	133	30		
0.00800		ug/L	56	150	30	56	150	30		
0.0120		ug/L	73	132	30	73	132	30		
0.00300		ug/L	70	134	30	70	134	30		
0.00500		ug/L	75	126	30	75	126	30		
0.00400		ug/L	49	150	30	49	150	30		
0.110		ug/L	41	140	30	41	140	30		
		ug/L			30			30	30	131
		ug/L			30			30	34	120
0.119		ug/L	42	120	30	42	120	30		
0.119		ug/L								
0.119		ug/L								
0.119		ug/L								
0.119		ug/L								
0.107		ug/L								
0.107		ug/L	42	126	30	42	126	30		
0.107		ug/L								
0.107		ug/L								
0.119		ug/L								
		ug/L							18	145
									21	124
0.120		ug/L	35	150	30	35	150	30		
0.130		ug/L	25	150	30	25	150	30		
0.110		ug/L	39	150	30	39	150	30		
		ug/L			30			30	10	150

MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
8.14		ug/L			20	85	115	20		
8.14		ug/L			20	85	115	20		
0.00400		mg/L	85	115		90	110	35		
11.7		ug/L	80	120	20	75	125	20		
0.475		ug/L	80	120	20	75	125	20		
1.15		ug/L	80	120	20	75	125	20		
0.925		ug/L	80	120	20	75	125	20		
0.124		ug/L	80	120	20	75	125	20		
0.375		ug/L	80	120	20	75	125	20		
31.7		ug/L	80	120	20	75	125	20		
1.65		ug/L	80	120	20	75	125	20		
0.412		ug/L	80	120	20	75	125	20		
1.97		ug/L	80	120	20	75	125	20		
33.7		ug/L	80	120	20	75	125	20		
0.417		ug/L	80	120	20	75	125	20		
21.8		ug/L	80	120	20	75	125	20		
0.839		ug/L	80	120	20	75	125	20		
1.39		ug/L	80	120	20	75	125	20		
83.3		ug/L	80	120	20	75	125	20		
0.432		ug/L	80	120	20	75	125	20		
1.30		ug/L	80	120	20	75	125	20		
180		ug/L	80	120	20	75	125	20		
0.191		ug/L	80	120	20	75	125	20		
1.00		ug/L	80	120	20	75	125	20		
4.22		ug/L	80	120	20	75	125	20		
0.0910		ug/L	80	120	20	75	125	20		

ATTACHMENT 4

Roux's Standard Operating Procedures

Date: May 5, 2000
Revision: April 6, 2015

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to explain the quality control (QC) measures taken to ensure the integrity of the samples collected and to establish the guidelines for the collection of QC samples. The objective of the QC program is to ensure that water-quality data of known and reliable quality are developed.

Valid water-chemistry data are integral to adequately characterize water-quality conditions, therefore, the data will typically be confirmed by the implementation of QC samples to verify the collection of representative samples. Without checks on the sampling and analytical procedures, the potential exists for contradictory or incorrect results. The acceptance of water-quality data by regulatory agencies and in litigation-support investigations depends heavily on the proper QC program to justify the results presented. The QC sampling requirements must be determined by the project manager and be clearly defined in the work plan. If data validation (for in-house purposes or for compliance with the United States Environmental Protection Agency [USEPA] regulations) is stipulated as part of the hydrogeologic investigation, QC sampling must be conducted.

2.0 QUALITY CONTROL SAMPLES

2.1 Samples taken for analysis of compounds require the use of quality control samples to monitor sampling activities and laboratory performance. Types of quality control samples may include replicate (sometimes referred to as a duplicate) and/or replicate split, trip blank, field equipment blank, matrix spike and matrix spike duplicate, and fortification. A discussion pertaining to each quality control sample follows:

a. Replicate and Replicate Split - Replicate sample analysis is done to check on the reproducibility of results either within a laboratory or between laboratories. A replicate sample is called a split sample when it is collected with, or turned over to, a second party (e.g., regulatory agency, consulting firm) for an independent analysis. Replicate samples are aliquots (equal portions) from a sample in a common container.

To collect a replicate sample, water from the bailer or pump will be distributed first to fill one container and then to fill the second container. Adequate water should be available to fill the bottles completely before they are capped. If the water is insufficient to fill all the bottles at once, then incrementally fill each bottle with water from two or more bailer volumes or pump cycles.

For some test substances, water may have to be accumulated in a common container and then decanted slowly into the sample bottles. The work plan

should be checked for a description of how replicate samples are to be collected. Additionally, in the case of wells that recover slowly and produce insufficient water to fill all the replicate sample containers, the containers should be filled incrementally and kept on ice in the cooler in between filling periods.

b. Trip Blank - A trip blank sample is a 40-ml vial that is filled with "clean" (e.g., distilled/deionized) water in the laboratory, and travels unopened with the sample bottles. (The USEPA now uses the phrase "demonstrated analyte free water.") It is opened in the laboratory and analyzed along with the field samples for the constituent(s) of interest to detect if contamination has occurred during field handling, shipment, or in the laboratory. Trip blanks are primarily used to check for "artificial" contamination of the sample caused by airborne volatile organic compounds (VOCs) but may also be used to check for "artificial" contamination of the sample by a test substance or other analyte(s). Two sealed laboratory filled 40-ml vials will be submitted per trip blank set. A trip blank set will be included in each cooler containing VOC samples, or other analyte(s) of interest, per day.

c. Field Equipment Blank - A field equipment blank (field blank) sample is collected to check on the sampling procedures implemented in the field. A field blank is made with "clean" (e.g., distilled/deionized/demonstrated analyte free) water by exposing it to sampling processes (i.e., the clean water must pass through the actual sampling equipment). For example, if samples are being collected with a bailer, the field blank would be made by pouring the clean water into a bailer which has been decontaminated and is ready for sampling, and then pouring from the bailer into the sample containers. If a metals equipment blank is to be made, and the water was filtered, then the sample must be filtered (i.e., exposed to the sampling process). One equipment blank would be incorporated into the sampling program for each day's collection of samples and analyzed for the identical suite of constituents as the sample.

A special type of field blank may be needed where ambient air quality may be poor. This field blank sample would be taken to determine if airborne contaminants will interfere with constituent identification or quantification. This field blank sample is a sample bottle that is filled and sealed with "clean" (e.g., distilled/deionized/demonstrated analyte free) water in the analytical laboratory, and travels unopened with the sample bottles. It is opened in the field and exposed to the air at a location(s) to check for potential atmospheric interference(s). The field blank is resealed and shipped to the contract laboratory for analysis.

d. Matrix Spike and Matrix Spike Duplicate - Spikes of compounds (e.g., standard compound, test substance, etc.) may be added to samples in the laboratory to determine if the ground-water matrix is interfering with

constituent identification or quantification, as well as a check for systematic errors and lack of sensitivity of analytical equipment. Samples for spikes are collected in the identical manner as for standard analysis, and shipped to the laboratory for spiking. Additional volume is required for MS/MSD samples. Matrix spike duplicate sample collection, and laboratory spiking and analysis is done to check on the reproducibility of matrix spike results and should be collected, when possible, from a location expected to be relatively free from contamination

e. Fortification - A fortification, which is performed in the field, is used to check on the laboratory's ability to recover the test substance (analyte) added as well as its stability between fortification and analysis.

A field fortification (spike) is prepared by filling the container(s) with field or distilled/deionized/demonstrated analyte free water (as specified by the laboratory) to a predetermined volume (as specified by the laboratory) and adding the spike (supplied by the laboratory). The predetermined volume of water is measured with a clean (decontaminated) graduated cylinder. Field spikes will be prepared following the collection, labeling, and sealing of nonspiked samples in a separate cooler. The spike is kept at a safe distance from the sampling point (e.g., in the hotel room).

f. Temperature Blank - A temperature blank is a container of water shipped with each cooler of samples requiring preservation by cooling to 6EC (ice). The temperature of the blank is measured at the time of sample receipt by the laboratory.

2.2 The work plan must be referred to for details regarding the type of QC samples to be collected and the QC sample collection method.

3.0 PROCEDURE

3.1 Implement QC sampling as outlined above, depending on the type of QC sample(s) specified in the work plan.

3.2 Ensure unbiased handling and analysis of replicate and blank QC samples by concealing their identity by means of coding so that the analytical laboratory cannot determine which samples are included for QC purposes. Attempt to use a code that will not cause confusion if additional samples are collected or additional monitoring wells are installed. For example, if there are three existing monitoring wells (MW-1, 2 and 3), do not label the QC blank MW-4. If an additional monitoring well were installed, confusion could result.

3.3 Label matrix spike and field fortification (spike) QC samples so that the analytical laboratory knows which samples are to be spiked in the laboratory and which samples were fortified (spiked) in the field, respectively. In certain situations, the field fortification will be "blind" or undisclosed to the laboratory to independently verify their analytical ability.

3.4 Verify that each sample is placed in an individual "zip-lock" bag, wrapped with "bubble wrap," and placed in its appropriate container (holder) 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. Consult the site work plan to determine if a particular ice is specified as the preservative for transportation (e.g., the USEPA prefers the use of wet ice because they claim that blue ice will not hold the samples at 4° Centigrade/Celsius).

3.5 Document the QC samples on the appropriate field form and in the field notebook. On the chain-of-custody form, replicate and blank QC samples will be labeled using the codes (Number 3.2, above), and matrix spike and field fortification QC samples will be identified as such (Number 3.3, above).

3.6 Follow standard shipping procedures for samples (i.e., retain one copy of the chain-of-custody form, secure the cooler with sufficient packing tape and a custody seal, forward the samples via overnight [express] mail, by courier or hand deliver to the designated analytical laboratory preferably within 24 hours but no later than 48 hours after sampling). However, check the site work plan for information on the analyte(s), as some have to be analyzed immediately (e.g., CN).

END OF PROCEDURE

Date: May 5, 2000

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide procedures and standards for record keeping and maintenance, for all field activities conducted by Roux Associates, Inc. (Roux Associates).

Strict quality assurance/quality control (QA/QC) is necessary to properly and accurately document and preserve all project-related information. Quality assurance is implemented to corroborate that quality control procedures are followed. Quality control provides a means to monitor investigation activities (e.g., sampling and laboratory performance) as a check on the quality of the data.

Valid data and information are integral to all aspects of Roux Associates' field activities. These aspects include, but are not necessarily limited to, activities that involve: drilling; sediment, sludge, and soil sampling (lithologic, and soil-quality and analysis); well construction and development; aquifer testing and analysis; water-quality sampling and analysis (surface water and ground water); free-product sampling and analysis; air-quality sampling and analysis; geophysical testing; demolition activities; waste removal operations; engineering installations; etc. The data will be confirmed by QA/QC methods established and set forth in the work plan/scope of work. Without checks on the field and analytical procedures, the potential exists for contradictory results, and associated incomplete or incorrect results from the interpretation of potentially questionable data.

Documentation will be entered in the field notebook and must be transcribed with extreme care, in a clear and concise manner, as the information recorded will become part of the permanent legal record. Because field notes are the legal record of site activities, they must be taken in a standard and consistent manner. If abbreviations are used, then they must first be spelled out for clarity (i.e., to avoid ambiguity and misunderstanding). All entries must be dated and initialed, and the time (military time) of the entry included. Field notebooks and forms must be assigned to an individual project and properly identified (i.e., client name, project number, location and name of site, individual recording information, dates, times, etc.). Change of possession of field notebooks or forms must be documented with the date and time, and initialed by both individuals. Any cross outs of text (from grammatical errors or misspellings) must be followed by the field personnel's initials. Following each day's entries, the field notebook or form must be photocopied in the event that the original documentation is lost or stolen. All field notebooks must have the company name and address legibly printed in indelible ink along with the message "If found, then please forward to Roux Associates, Inc. at the above address - REWARD OFFERED."

Information must be recorded while onsite because it may be difficult to recall details at a later date. Furthermore, information must be documented immediately as it provides unbiased information which will be used for writing the report when the field activities

are completed. Project-related documentation is an irreplaceable, important record for other individuals who may become involved in the project, and provides the project manager with a complete history of project-related activities. Written information must be accompanied by maps, sketches, and photographs where appropriate, especially if these supplemental sources of information assist in the documentation process. A new page must be used in the field notebook for each new day's entries (i.e., unused portions of a previous page must have an "X" placed through it). The end of the day's records must be initialed and dated.

As part of record keeping and QA/QC activities, state and federal regulatory agencies should be contacted to check if special or different protocols are required and/or if particular or unconventional methods are required for the given field activity. Thus, the record keeping and QA/QC activities implemented by Roux Associates are based on technically sound standard practices and incorporate Roux Associates own, extensive experience in conducting hydrogeologic field activities.

2.0 MATERIALS

In order to track investigation activities, specific materials are required. These materials include the following:

a. A bound, waterproof field notebook.

b. Appropriate Roux Associates' forms (e.g., daily log, geologic log, monitoring well construction log, well sampling data form, location sketch, chain of custody, telephone conversation record, meeting notes, etc.).

c. Appropriate labels (e.g., sample, Roux Associates' Custody Seal, etc.)

d. Work plan/scope of work.

e. Health and safety plan (HASP).

f. Appropriate Roux Associates' SOPs.

g. Black pens, and indelible markers.

h. Camera and film.

3.0 DOCUMENTATION

3.1 Before the Roux Associates personnel leave the field, they must ensure that their field notes include comprehensive descriptions of the hydrogeologic conditions, and all investigation-related activities and results (onsite and offsite). This will safeguard against the inability to reconstruct and comprehend all aspects of the field investigation after its completion, and will serve to facilitate the writing of an accurate report. Properly documented information provides the QA/QC tracking (back-up) required for all Roux Associates' projects. General types of

information that must be recorded (where pertinent to the investigation being conducted) include, but may not necessarily be limited to, the following:

a. List of Roux Associates personnel on site.

b. Name, date, and time of arrival on site by Roux Associates personnel, including temporary departures from, and returns to, the site during the work day.

c. Client and project number.

d. Name and location of study area.

e. Date and time of arrival on site by non-Roux Associates personnel (names and affiliation) and equipment (e.g., subcontractors and facility personnel, and drilling equipment, respectively, etc.), including temporary departures from, and returns to, the site during the work day, and departure at the end of the work day.

f. List of non-Roux Associates personnel on site.

g. Weather conditions at the beginning of the day as well as any changes in weather that occur during the working day.

h. Health and safety procedures including level of protection, monitoring of vital signs, frequency of air monitoring, and any change (i.e., downgrade or upgrade) in the level of protection for Roux Associates and other on-site personnel (e.g., subcontractors, facility personnel, etc.).

i. Health and safety procedures not in compliance with the HASP (for all on-site personnel).

j. Site reconnaissance information (e.g., topographic features, geologic features, surface-water bodies, seeps, areas of apparent contamination, facility/plant structures, etc.).

k. Air monitoring results (i.e., photoionization detector [PID], etc. measurements).

l. Task designation and work progress.

m. Work-related and site-related discussions with subcontractors, regulatory agency personnel, plant personnel, the general public, and Roux Associates personnel.

n. Delays, unusual situations, problems and accidents.

o. Field work not conducted in accordance with the work plan/scope of work, and rationale and justification for any change(s) in field procedures

including discussions with personnel regarding the change(s) and who authorized the change(s).

p. QA/QC procedures not conducted in accordance with the QA/QC procedures established in the work plan/scope of work and rationale and justification for any change(s) in QA/QC procedures including discussions with personnel regarding the change(s) and who authorized the change(s).

q. Equipment and instrument problems.

r. Decontamination and calibration procedures.

s. Activities in and around the site and work area by any and all on-site personnel which may impact field activities.

t. Sketches, maps, and/or photographs (with dates and times) of the site, structures, equipment, etc. that would facilitate explanations of site conditions.

u. Contamination evidenced as a result of work-related activities (e.g., visible contaminants [sheen] in drilling fluids or on drilling equipment; sheen on, or staining of, sediments; color of, or separate [nonaqueous] phase on, water from borehole or well; vapors or odors emanating from a borehole or well; etc.); make all observations as objectively as possible (e.g., grey-blue, oil-like sheen; black and orange, rust-like stain; fuel-like odor; etc.) and avoid using nontechnical or negative-sounding terms (e.g., slimy, goopy, foul-smelling).

v. Date and time of final departure from the site of all personnel at the end of the work day.

3.2 In addition to the general types of information that must be recorded (as presented in Section 3.1), task-specific information must also be properly documented. Task-specific information which is required is provided in each respective task-oriented SOP, and the documentation procedures outlined in each SOP must be followed.

END OF PROCEDURE

Date: ~~May 5, 2000~~March 4, 2015

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(SOP-3.1), for Evaluation and Validation of Data (SOP-3.4), and for Field Record Keeping and Quality Assurance/Quality Control (SOP-3.2) 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. A plastic (garbage) bag should line the inside of the cooler and all ice and samples should be placed inside to prevent leaking during transportation. 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, preferably with bubble wrap, 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). If additional coolers are required, then they should be purchased. The chain-of-custody form should be properly completed, placed

Commented [LT1]: Should we include the use of a garbage bag?

in a "zip-lock" bag, and placed in the cooler. One copy must be maintained for the project files. The plastic (garbage) bag encompassing all samples and ice shall be secured tightly with a zip tie. 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).

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

d. Black pen and indelible marker.

e. Packing tape, large plastic (garbage) bag, zip ties, "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 nitrile 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.

Commented [LT2]: Should we include taping over the labels to prevent the ink from running?

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: January 10, 2011
Revision: May 7, 2015

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to establish guidelines for sampling monitoring wells for dissolved constituents in groundwater using low flow (low stress) techniques. Low flow purging and sampling results in the collection of groundwater samples from monitoring wells that are representative of groundwater conditions in the geological formation by minimizing stress on the geological formation, and minimizing the disturbance of sediment in the bottom of the well. This procedure also minimizes aeration of the groundwater during sample collection, which improves the representativeness of the sample for VOC analysis.

As part of the SOP for the sampling of groundwater monitoring wells, sample collection equipment and devices must be considered, and equipment decontamination and pre-sampling procedures (e.g., measuring water levels, and purging wells) must be implemented. The sampling objectives will be provided in specific investigation work plans that will be approved by the applicable regulatory agency. Unless otherwise specified in an approved work plan, all groundwater purging and sampling should typically be performed using low stress (low flow) sample collection techniques.

2.0 EQUIPMENT AND MATERIALS

2.1 In order to sample groundwater 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. Safety first. Obtain the appropriate Job Safety Analysis (JSA) and personal protection equipment (PPE), as specified in the site Health and Safety Plan (HASP).

b. Adjustable rate, positive displacement groundwater sampling pump (e.g., low-flow centrifugal submersible pump or bladder pump constructed of stainless steel, Teflon or other inert material).

c. Teflon or Teflon-lined polyethylene tubing with ¼-inch or 3/8-inch inner diameter to collect samples for organic analysis. Teflon or Teflon-lined polyethylene tubing, PVC, Tygon or polyethylene tubing with ¼-inch or 3/8-inch inner diameter to collect samples for inorganic analysis. Sufficient tubing of the appropriate material must be available so that each well has dedicated tubing.

d. Appropriate power source (compressor or compressed gas tank, controller, generator, etc.).

e. Monitoring instrument for water quality indicator parameters (Horiba U-22 or equivalent) including temperature, dissolved oxygen, turbidity, specific conductance, pH and redox potential (Eh).

f. Flow measurement supplies (e.g., graduated cylinder and stop watch or in-line flow meter).

g. Electronic interface probe with a minimum of 0.01 foot accuracy.

h. Decontamination supplies including Non-phosphate laboratory-grade detergent, distilled/deionized water and potable water.

i. Roux Associates' low flow well sampling/purging data form and field notebook.

j. Well construction data logs, field data from previous sampling event, and site map with well locations.

k. Well security keys.

l. Plastic sheeting.

m. Paper towels, clean rags.

n. Teflon™ tape, .

o. Calculator.

p. Black pen and water-proof marker.

q. Tools (e.g., wrenches, screwdrivers, hammer, pliers, flashlight, etc.) or alternatives recommended in the JSAs.

r. Disposable Nitrile gloves.

s. Laboratory-supplied sample containers with labels.

t. Cooler(s).

u. Ice (wet, blue packs).

v. Masking, duct, and packing tape.

w. Chain-of-custody form(s) and custody seal(s).

x. Field Sampling Plan (FSP) and Quality Assurance Project Plan (QAPP) or equivalent summary.

y. Site health and safety plan (HASP).

z. Packing material (e.g., bubble wrap)

aa. "Zip-lock" plastic bags.

bb. Overnight (express) mail forms (or schedule courier pickup if available).

3.0 DECONTAMINATION

3.1 Make sure all equipment is decontaminated and cleaned before use (refer to the Roux SOP-9.1 for Decontamination of Field Equipment for detailed decontamination methods; a summary of pump decontamination procedures is provided below). Use new, clean materials when decontamination is not appropriate (e.g., tubing, disposable gloves). Document, and initial and date the decontamination procedures on the appropriate field form and in the field notebook.

A. Before sampling:

The manufacturer recommendations on pump disassembly for cleaning, if any, should take precedence to the disassembly recommendations presented below.

1) Wear disposable Nitrile and cut proof gloves.
2) Pre-rinse: Operate the pump first in an appropriate container with potable water for 5 minutes.
3) Wash: Operate the pump in an appropriate container with a non-phosphate, laboratory-grade detergent such as Alcanox and potable water solution for 5 minutes.
4) Rinse: Operate the pump in an appropriate container for 5 minutes with potable water.
5) Disassemble pump (refer to manufacturer's manual).
6) Wash pump parts: Place the disassembled parts of the pump into an appropriate container with non-phosphate detergent solution. Scrub the disassembled pump parts with an appropriate brush.
7) Rinse pump parts with potable water.
8) Rinse the following parts with distilled/deionized water: inlet screen, shaft, suction interconnector, motor lead assembly, and stator housing.
9) Rinse impeller with potable water.

B. After each sample collection, decontaminate by:

1) Wear disposable Nitrile gloves.

2) Pre-rinse: Operating the pump first with potable water for 5 minutes.

3) Wash: Operate the pump in an appropriate container with a non-phosphate, laboratory-grade detergent such as Alcanox and potable water solution for 5 minutes.

4) Rinse: Operate the pump in an appropriate container for 5 minutes with potable water

5) Final Rinse: Operate the pump in an appropriate container to pump out 1 to 2 gallons of distilled/deionized water

6) Wipe pump-related equipment (e.g., electrical lines, cables) 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 pumps are the minimum that must be performed. Check the work plan to determine if additional chemicals specified by individual state regulatory agencies also must 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., water quality meter, photoionization detector, flame ionization detector) according to the Manufacturer's calibration instructions. Document, initial and date the calibration procedures on the appropriate field form and in the field notebook.

5.0 PROCEDURE

5.1 As practicable under field conditions, start at the well that is known or believed to have the least contaminated groundwater and proceed systematically to the well that is known or believed to have the most contaminated groundwater.

5.2 Document, initial and date well identification, pre-sampling information, and problems encountered on the appropriate field form and in the field notebook as needed.

5.3 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).

5.4 Place plastic sheeting around the well to protect sampling equipment from potential cross contamination.

5.5 When vapor concerns are present, measure the VOCs at the rim of the unopened well utilizing a photoionization detector (PID) or flame ionization detector (FID) instrument and record the reading in the field notebook.

5.6 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. Never inhale the vapors.

5.7 Measure the depth to water (DTW) to 0.01 ft from the measuring point (MP) on the well (top of well casing) using an electronic interface probe or water level meter. Calculate the water-level elevation. Document, initial and date the information on the appropriate field form and in the field notebook.

5.9 Decontaminate the interface probe or water level meter with a non-phosphate, laboratory-grade detergent solution followed by a distilled/deionized water rinse.

5.10 Install Pump: Slowly lower the pump, safety cable, tubing and electrical lines into the well at midpoint of the immerged well screen or at the most permeable zone within the immerged well screen. The pump intake is to be kept at least two feet above the bottom of the well to prevent disturbance and re-suspension of any sediment or a sinking separate phase product (DNAPL) present in the bottom of the well.

If separate-phase product was identified in a measurable quantity, then the tubing should be installed using PVC with a tethered cap inserted into the well to bypass the LNAPL. The sample tubing should be inserted through the PVC, pushing out the cap at a depth below the LNAPL in order to sample the groundwater below the LNAPL.

5.11 Measure Water Level: Before starting the pump, measure the water level again with the pump in the well. Leave the electronic interface probe in the well.

5.12 Purge well: Start pumping the well at 200 to 500 milliliters per minute (mL/min). The water level should be monitored approximately every five minutes. Ideally, a steady flow rate should be maintained that results in a stabilized water level (drawdown of 0.3 ft or less). Pumping rates should be, if needed, reduced to the minimum capabilities of the pump to ensure stabilization of the water level. The flow rate should, however, be sufficient to maintain pump suction and to avoid cascading or air bubble formation in the tubing. Record each adjustment made in the pump rate and the water level measured immediately after each adjustment.

5.13 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.

5.14 Monitor Indicator Parameters: During purging of the well, monitor and record the field indicator parameters (turbidity, temperature, specific conductance, pH, redox potential, and dissolved oxygen) approximately every five minutes. The well is considered stabilized and ready for sample collection when the indicator parameters have stabilized for three consecutive readings as follows:

±0.1 for pH

±3% for specific conductance and temperature

±10 mV for redox potential

±10% for DO and turbidity

And turbidity to be less than 50 NTU prior to sampling

The pump must not be removed from the well between purging and sampling.

ADDRESSING POTENTIAL PROBLEMS

Insufficient Yield

If the formation cannot produce enough water to sustain purging, then the well should be pumped to "near-dry" conditions (i.e., leaving some water in the well). Purging should be interrupted before the water level in the well drops below the top of the pump, as this may induce cascading of the sand pack. Pumping the well dry should be avoided to the extent possible in all cases. When preparing to conduct a sampling round, review drilling, well 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.

Sampling should commence as soon as the volume in the well has recovered sufficiently to allow collection of samples. 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.
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 in the field notebook.

Failure to Stabilize Key Indicator Parameters

If one or more key indicator parameters fail to stabilize after 4 hours or after 5 well volumes have been purged, one of four options should be considered:

1) Continue purging in an attempt to achieve stabilization.
2) Discontinue purging, do not collect samples, and document attempts to reach stabilization in the field notebook.
3) Discontinue purging, collect samples, and document attempts to reach stabilization in the field notebook.
4) Secure the well, purge and collect samples the next day (preferred)

The key indicator parameter for VOCs is dissolved oxygen.

The key indicator parameter for all other samples is turbidity.

Regardless of the option that is followed, the sampling procedure must be fully documented in the field notebook.

5.15 Collect Samples: Collect samples at a flow rate between 100 and 250 mL/min and such that drawdown of the water level within the well does not exceed the maximum allowable drawdown of 0.3 ft.

VOC samples must be collected first. If a bladder pump is used, best practice would have it to match one compression stroke to the fill one VOC sampling vial.

Remove each appropriate container's cap only when ready to fill each with the water sample, and then replace and secure the cap immediately.

5.16 During sample collection, make sure that the pump discharge line or the bailer does not contact the sample container. Fill each appropriate, pre-labeled sample container by allowing the ground water to flow from the tubing gently down the inside of the container 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 an 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.

5.17 Fill containers for VOCs until a convex surface is observed 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.

5.18 Consult the work plan if filtered sampled should also be collected for dissolved metals analysis.

5.19 Collect quality control (QC) samples as required in the work plan to monitor sampling and laboratory performance.

5.20 Note and record the "final" physical appearance of the water (after purging and sampling) on an appropriate field form and in the field notebook.

5.21 Remove pump and tubing.

5.22 Measure the total depth of the well from the MP with an interface probe. Document, initial and date the information on the appropriate field form and in the field notebook.

5.23 Wipe the well cap/j-plug with a clean rag, replace the well cap/j-plug and protective cover. Lock the protective cover.

5.24 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.

5.25 Decontaminate field equipment following Roux SOP-9.1 for Decontamination of Field Equipment. Wrap decontaminated equipment with a suitable material (e.g., clean plastic bag or aluminum foil). Discard rags, gloves, etc. in a manner consistent with site conditions.

5.26 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.

5.27 Samples will be delivered according to the QAPP schedule.

6.0 REFERENCES

U.S. Environmental Protection Agency – Region I. Low Stress (Low Flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells. Revised January 19, 2010.

END OF PROCEDURE

Date: ~~May 5, 2000~~March 5, 2015

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. Safety first. Obtain the appropriate Job Safety Analysis (JSA) and personal protection equipment (PPE), as specified in the site Health and Safety Plan (HASP).

~~a.~~b. A work plan which outlines soil sampling requirements.

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

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

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

~~e.~~f. Stainless steel spoons or spatulas.

~~f.~~g. Disposable nitrile sampling gloves.

~~g.~~h. Laboratory-supplied sample containers with labels.

~~h.~~i. Cooler with blue or wet ice.

~~i.~~j. Plastic sheeting.

~~j.~~k. Black pen and indelible marker.

~~k.~~l. Zip-lock bags and packing material (e.g., bubble wrap).

~~l.~~m. Tape measure.

~~m.~~n. Paper towels or clean rags.

~~n.~~o. Masking and packing tape.

~~o.~~p. Overnight (express) mail forms or schedule currier pickup.

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: January 9, 2011
Revision: May 12, 2015

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, Geoprobe™ core samples, and Sonic core 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 Geoprobe™ or other direct push sampling method, or a decontaminated bucket auger or power auger may be needed to advance the hole to the point of sample collection. If augering to depth, once the sampling depth is reached a clean bucket auger should be used to collect the sample. To collect samples at depths of greater than approximately six feet, the use of a drill rig (direct push, split spoon, etc) will usually be necessary. In some situations, such as an excavation or trench, sample locations are accessed with the use of a backhoe.

3.0 EQUIPMENT AND MATERIALS

a. Safety first. Obtain the appropriate Job Safety Analysis (JSA) and personal protection equipment (PPE), as specified in the site Health and Safety Plan (HASP).

b. A work plan which outlines soil sampling requirements.

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

d. Decontamination supplies (including: non-phosphate laboratory grade detergent, buckets, brushes, potable water, distilled water, plastic sheeting, etc.).

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

f. Stainless steel spoons or spatulas.

g. Disposable Nitrile sampling gloves and cut-proof gloves.

h. Laboratory-supplied sample containers with labels.

i. Cooler with blue or wet ice.

j. Plastic sheeting.

k. Black pen and indelible marker.

l. Zip-lock bags and packing material.

m. Tape measure.

n. Paper towels or clean rags.

o. Masking and packing tape.

p. Overnight (express) mail forms or schedule courier pickup.

4.0 DECONTAMINATION

All reusable sampling equipment will be thoroughly cleaned according to the decontamination SOP (ROUX SOP 9.1). Where possible, thoroughly pre-cleaned and wrapped sampling equipment should be used and dedicated to individual sampling locations. Disposable items such as sampling gloves 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 ROUX SOP 9.1 decontamination procedures. 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. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised

When sampling shallow soils, if a thick, matted root zone, gravel, concrete, etc. is present at or near the surface, it should be removed before the sample is collected. The depth measurement for the sample begins at the top of the soil horizon, immediately following any removed materials.

When using direct push sampling methods, it is important to maintain the proper orientation of the sample when the sampling liners are removed from the sample tubes define precisely the depth at which an aliquot was collected. This is particularly important when multiple sample depths are collected from the same push.

When utilizing a split spoon sampling method, always discard the top several inches of material in the spoon before removing any portion for sampling. This material normally consists of borehole wall material that has sloughed off of the boregole wall after removal of the drill string prior to inserting the split spoon.

When utilizing a backhoe to collect soil samples from a trench or excavation, measures should be taken to ensure that the bucket is decontaminated and that no paint, grease, and rust is present prior to sample collection.

5.3 Place the sample in a laboratory-supplied, pre-cleaned sample container. Cut-proof gloves should be worn at all times when handling glassware. 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.

If samples are to be analyzed for VOCs, they should be collected in a manner that minimizes disturbance of the sample. ***Samples for VOC analysis must not be homogenized.***

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 physical characteristics (color, odor, moisture, texture, density, consistency, organic content, layering, grain size, etc.). Refer to Soil Classification and logging SOP.

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 or in a Zip-lock bag and homogenized. make sure that each composite location (aliquot) consist of equal volumes,(i.e., same number of equal spoonfuls). 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 with ice, at 4°C. Store the sample cooler in a secure location.

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 signed and dated custody seal is placed on the cooler prior to shipment.

5.9 Samples collected from Monday to Friday are typically 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, sample 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 ROUX SOP 9.1 decontamination procedures. Following the final decontamination (after all samples are collected), the sampling equipment is wrapped with plastic. Discard any gloves, plastic, etc. in an appropriate manner that is consistent with site conditions.

END OF PROCEDURE

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish guidance to obtain accurate and consistent descriptions of soil characteristics during soil-sampling operations. Consistency with description is essential for creating subsurface interpretations.

This SOP discusses the use of the Unified Soil Classification System (USCS) as a guideline for classifying and describing lithology encountered in boreholes, test pits, and surface sampling.

The Unified Soil Classification System (USCS) is based on numerical values of certain soil properties that are measured by laboratory tests (ASTM D 2487). It is possible, however, to estimate these values in the field with reasonable accuracy using visual-manual procedures (ASTM D 2488). In addition, some elements of a complete soil description, such as the presence of cobbles or boulders, changes in strata, and the relative proportions of soil types in a bedded deposit can be obtained only in the field.

2.0 MATERIALS/EQUIPMENT

a) Indelible pens

b) Tape measure or ruler

c) Field logbook

d) Squirt bottle/Spray bottle with water

e) Rock- or soil-color chart (e.g., Munsell)

f) Grain-size chart

g) Hand lens

h) Unified Soil Classification System index charts and tables to help with soil classification

i) Workplan or event-specific field instructions. Planned borehole depth, proposed well construction/specifications, and field sampling summary table, if available.

3.0 DECONTAMINATION

All reusable sampling equipment will be thoroughly cleaned according to the decontamination ROUX SOP 9.1. 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 consistent with site conditions

4.0 PROCEDURE

This section covers several aspects of the soil characterization: instructions for completing soil boring logs.

4.1 Record the following general site information in the field book:

a. Boring/Well ID. Enter the boring/well number. A numbering system should be chosen that does not conflict with information recorded for previous exploratory work done at the site. Number the sheets consecutively for each boring.

b. Client/Project Name

c. Site Location.

d. Drilling Contractor. Enter the name of the drilling company and the city and state where the company is based.

e. Drilling Method and Equipment. Identify the bit size and type, drilling fluid (if used), and method of drilling (e.g., rotary, hollow-stem auger, sonic). Information on the drilling equipment (e.g., CME 55, Mobile B61) should be noted.

f. Date and Time. Include Start time and End time.

g. Name and/or initials of the field personnel logging the soil.

4.2 Record the following technical data in the field book and/or log forms:

a. Depth Below Surface.

b. Sample Interval.

c. PID readings (if specified by work plan). Refer to ROUX SOP 5.4 for soil screening procedures.

d. Blow Counts.

e. Sample Recovery.

f. Soil Description.

g. Soil Classification.

4.3 Soil Classification

Soil descriptions should be precise and comprehensive without being verbose. The correct overall impression of the soil should not be distorted by excessive emphasis on insignificant details. In general, similarities rather than differences between consecutive samples should be stressed.

Field descriptions should include as a minimum:

a. Color: The basic color of the soil.

b. Soil name: The basic name of the predominant constituent and a single word modifier indicating the major subordinate constituent.

c. Particle Size Distribution: An estimate of the percentage and grain-size range of each of the soil's subordinate constituents (trace, little, some). This parameter is critical for assessing hydrogeology of the site and should be carefully and fully documented.

d. Gradation or Plasticity: For granular soil (sands or gravels) that should be described as well-graded, poorly graded, uniform, or gap-graded. Cohesive soil (silts or clays) should be described as non-plastic, low plastic, medium plastic, or highly plastic.

e. Other Components: Examine the soil sample to identify other components that may be present including roots and root mass, vegetation, peat, organic matter, shells or fossils, accessory minerals slag, cinder or charcoal, trash, rubbish, fill, bricks, glass.

f. Moisture Content. The amount of soil moisture described as dry, moist, or wet/saturated.

g. Odor: The odor should only be described if it is organic or unusual. An organic odor will have distinctive decaying vegetation smell. Unusual odors, petroleum product, chemical, and the like should be noted.

h. Unusual characteristics: If staining or discoloration of soils is present it should be noted.

4.4 The soil logs should also include a complete description of any tests run in the borehole; placement and construction details of piezometers, wells, and other monitoring equipment; abandonment records; geophysical logging techniques used; and notes on readings obtained by air monitoring instruments.

4.5 Backfilling

When a boring is completed, the boring shall be abandoned with procedures specified in the workplan. The destruction of the hole shall be noted on the log Proper borehole abandonment procedures are documented in ROUX SOP 5.4.

END OF PROCEDURE

Date: January 9, 2011
Revision: April 17, 2015

1.0 PURPOSE

The purpose for this standard operating procedure (SOP) is to establish the guidelines for measuring field parameters of groundwater samples. The parameters are measured in the field using a water quality meter (Horiba U-22 or equivalent). The water quality parameters include pH, temperature, conductivity, ORP, dissolved Oxygen and turbidity. The manufacturer's instrument manual for each water quality meter, which is maintained with the instrument, will be referred to for calibration, use, repair, maintenance, or trouble-shooting operations.

The pH will be measured in standard units (SU) and can be recorded with or without the SU designation. The conventional means of recording a pH value is without a unit designation (e.g., 7.0); however, the SU designation may be used provided the term is defined as standard units when first referenced. The pH is measured in the field to provide the pH of the water under ambient (in situ) conditions. The pH is a measure of the acidic (<7.0) or basic (>7.0) nature of the water and is used to assist in evaluating the mobility of contaminants. In addition, pH measurements can be used during well purging to help determine when sufficient groundwater has been purged (removed) from a well (i.e., the standing water in the well has been removed and replaced with "fresh" water from the aquifer).

Conductivity will be measured and recorded in micromhos/centimeter (µmhos/cm). Some water quality meters measure directly in µmhos/cm while others have to be converted to this unit. The specific conductivity is measured in the field as a measure of the total dissolved solids (TDS) in the water samples. TDS data can then be used as a qualitative measure of contamination and to assist in evaluating electrical resistivity and borehole geophysical data. In addition, specific conductivity measurements can be used during well purging to help determine when sufficient groundwater has been purged (removed) from a well (i.e., the standing water in the well has been removed and replaced with "fresh" water from the aquifer).

Temperature will be measured and recorded in degrees Celsius/Centigrade (° C). Temperature data is collected in the field to determine the temperature of the water sample under ambient (in-situ) conditions. Temperature data can be used to evaluate the mobility of compounds in groundwater and flow conditions. In addition, temperature measurements can be used during well purging to help determine when sufficient groundwater has been purged (removed) from a well (i.e., the standing water in the well has been removed and replaced with "fresh" water from the aquifer).

ORP will be measured and recorded in millivolts (mV). ORP data is collected in the field for monitoring and controlling chemical addition of oxidizing or reducing agents. In addition, ORP data can be used during well purging to help determine when sufficient groundwater has been purged (removed) from a well (i.e., the standing water in the well has been removed and replaced with "fresh" water from the aquifer).

Dissolved Oxygen (DO) will be measured and recorded in mg/L. DO data is collected in the field for monitoring the amount of oxygen dissolved in a unit volume of water. In addition, DO data can be used during well purging to help determine when sufficient groundwater has been purged (removed) from a well (i.e., the standing water in the well has been removed and replaced with "fresh" water from the aquifer).

Turbidity will be measured and recorded in Nephelometric Turbidity Units (NTU). Turbidity data is collected to measure the presence of suspended particulates in water.

2.0 CALIBRATION

Prior to calibration, all water quality meters must be cleaned and the battery checked according to the manufacturer's instructions. Document, initial and date the pre-calibration procedure on the appropriate field form, and in the field notebook.

2.1 Safety first. Obtain the appropriate Job Safety Analysis (JSA) and personal protection equipment (PPE), as specified in the site Health and Safety Plan (HASP).

2.2 Calibration of the water quality meter(s) is to be performed at the beginning and end of each day's use in accordance with the manufacturer's specific calibration instructions. At the end of each day, a calibration check is performed to verify that the instrument(s) remained in calibration throughout the day. This check is to be performed while the instrument(s) is in measurement mode, not calibration mode. Usual procedures are given below.

2.3 Recalibration must occur if: 1) the parameter being measured is outside the previous calibration range (if meter readings are zero or a negative number for DO, pH, conductivity or turbidity [negative value only]); 2) the procedure or use conditions warrant frequent calibrations; 3) four or more hours have elapsed; or 4) the instrument has been moved from one area to another (e.g., offsite or out of the study area).

2.4 The volume of the calibration solutions must be sufficient to cover both the probe and temperature sensor (see manufacturer's instructions for the volume to be used). Note that most instrument manual's state there is no calibration for the temperature sensor, but the temperature sensor must be checked to determine its accuracy. If the accuracy check date/information is not included with the instrument or the last check was over a year, the temperature sensor accuracy needs to be checked at the beginning of the water quality measuring event.

2.5 A minimum of two standards should be used to bracket the instruments measurement range for all parameters except ORP (refer to manufacturer's instructions).

2.6 The following information is documented in the calibration logbook and /or field notebook at the time of calibration:

a. Date.

b. Water Quality meter identification.

c. Pre-cleaning and battery check.

d. Calibration results.

e. Initials of the individual performing calibration.

3.0 PROCEDURE

3.1 A warm-up period may or may not be necessary for the instrument, depending on instrument requirements. The manufacturer's instrument manual must be followed.

3.2 See SOPs 4.3 and 4.4 for information regarding well sampling/purging procedures. Follow instruction manual for proper set up and deployment of monitoring device.

3.3 The groundwater quality parameters will be continuously measured by the water quality meter utilizing a flow-through-cell. If the accumulation of sediment buildup in the cell is observed, continue pumping and disconnect the cell for cleaning, then reconnect after cleaning and continue monitoring activities. If air bubbles accumulation is observed in the cell, place the meter at a 45 degree angle with the port facing upward to help evacuate any air bubble buildup.

3.5 Pertinent data are documented in the field notebook and/or appropriate field form, initialed and dated.

3.6 Follow decontamination procedures documented in SOP 9.1 after each use to avoid potential cross contamination. Ensure that the electrodes are rinsed with distilled or deionized water and the unit stored properly in accordance with the manufacturer's instructions (e.g., capping and storing in a buffer such as altex electrode storage solution). The electrodes are not to be stored in potable water, or distilled or deionized water.

END OF PROCEDURE

Date: January 9, 2011
Revision: May 5, 2015

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 field investigation activities (i.e. drilling, soil and water sampling).

The objective of decontamination is to ensure that all field samplingequipment 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.

The decontamination line is setup so that the first station is used to clean the most contaminated item. It progresses to the last station where the least contaminated item is cleaned. A site is typically divided up into the following boundaries: Hot Zone or Exclusion Zone (EZ), the Contamination Reduction Zone (CRZ), and the Support or Safe Zone (SZ). The decontamination line should be setup in the Contamination Reduction Corridor (CRC).

In considering decontamination procedures, state and federal regulatory agency requirements must be considered because of potential variability between state and federal requirements. 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.

The equipment and materials list for decontamination activities may include, but not necessarily be limited to, the following:

a. A work plan and health and safety plan which outlines decontamination procedures and requirements.

b. Field notebook and field form(s).

c. Decontamination solutions, including as necessary: non-phosphate, laboratory-grade detergent; distilled/deionized water; potable water; cleaning solvents if needed [e.g., hexane, acetone, nitric acid).

d. Long and short handled brushes,

e. Bottle brushes

f. Drop cloth/plastic sheeting

g. Paper towels

h. Plastic or galvanized tubs or buckets

i. Pressure washers or steam cleaners

j. Solvent sprayers

k. Trash / bilge pumps

l. Aluminum foil

m. 55-gallon drums.

2.0 PROCEDURE FOR DRILLING EQUIPMENT

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

2.1 Safety first. Obtain the appropriate Job Safety Analysis (JSA) and personal protection equipment (PPE), as specified in the site Health and Safety Plan (HASP). Prior to mobilization to a site, the expected types of contamination should be evaluated to determine if the field cleaning and decontamination activities will generate rinsates and other waste waters that might be considered RCRA hazardous waste or may require special handling.

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

2.3 The augers, drilling casings, rods, samplers, tools, and any piece of equipment that can come in contact (directly or indirectly) with the soil, requires proper decontamination on-site prior to commencing drilling. The project work plan or HASP, and appropriate regulatory requirements, should be consulted to determine site-specific decontamination requirements.

2.4 The same decontamination procedures used prior to drilling will be followed between boreholes (at a fixed on-site location[s], if appropriate) and before leaving the site at the end of the investigation.

2.5 All on-site steam cleaning or (decontamination) activities will be monitored and documented by a member(s) of the staff of Roux Associates, Inc. and should be performed on a decontamination pad that meets the following specifications:

1. The pad should be constructed in an area known or believed to be free of surface contamination.
2. A temporary pad should be lined with a water impermeable material with no seams within the pad. This material should be either easily replaced disposable) or repairable.The pad should be regularly inspected to ensure there are no leaks.

3. Water should be removed from the decontamination pad frequently.

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

2.7 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 federalregulatory requirements.

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 Safety first. Obtain JSA and PPE, as specified in the site HASP.

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

3.3 Steam clean the sampler or rinse with potable water. If soil-sampling activities are conducted in the presence of thick, sticky oils (e.g., PCB oil) 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).

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

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

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

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

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

3.9 Reassemble the cleaned sampler, as necessary.

3.10 After equipment has been cleaned, all individuals involved in equipment handling should don 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 Safety first. Obtain the JSAs and PPE, as specified in the site HASP.

4.2 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 sampling equipment. Discard all used sampling tubes and cords in an appropriate manner. Disconnect all power sources from electrical equipment (i.e. pumps). Scrub each piece of equipment with a brush and solution.

d. Rinse all sampling equipment with copious amounts of potable, distilled or deionized water, Reassemble equipment as per the manufacturer's instructions.

f. Air dry.

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

4.3 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.

END OF PROCEDURE

APPENDIX C

Community Air Monitoring Plan

New York State Department of Health (NYSDOH)
Generic Community Air Monitoring Plan (CAMP)

Overview

A Community Air Monitoring Plan (CAMP) requires real-time monitoring for volatile organic compounds (VOCs) and particulates (i.e., dust) at the downwind perimeter of each designated work area when certain activities are in progress at contaminated sites. The CAMP is not intended for use in establishing action levels for worker respiratory protection. Rather, its intent is to provide a measure of protection for the downwind community (i.e., offsite receptors including residences and businesses and onsite workers not directly involved with the subject work activities) from potential airborne contaminant releases as a direct result of investigative and remedial work activities. The action levels specified herein require increased monitoring, corrective actions to abate emissions, and/or work shutdown. Additionally, the CAMP helps to confirm that work activities did not spread contamination offsite through the air.

The generic CAMP presented below will be sufficient to cover many, if not most sites. Specific requirements should be reviewed for each situation in consultation with NYSDOH to ensure proper applicability. In some cases, a separate site-specific CAMP or supplement may be required. Depending upon the nature of contamination, chemical-specific monitoring with appropriately sensitive methods may be required. Depending upon the proximity of potentially exposed individuals, more stringent monitoring or response levels than those presented below may be required. Special requirements will be necessary for work within 20 feet of potentially exposed individuals or structures and for indoor work with co-located residences or facilities. These requirements should be determined in consultation with NYSDOH.

Reliance on the CAMP should not preclude simple, common-sense measures to keep VOCs, dust, and odors at a minimum around the work areas.

Community Air Monitoring Plan

Depending upon the nature of known or potential contaminants at each site, real-time air monitoring for VOCs and/or particulate levels at the perimeter of the exclusion zone or work area will be necessary. Most sites will involve VOC and particulate monitoring; sites known to be contaminated with heavy metals alone may only require particulate monitoring. If radiological contamination is a concern, additional monitoring requirements may be necessary per consultation with appropriate DEC/NYSDOH staff.

Continuous monitoring will be required for all ground intrusive activities and during the demolition of contaminated or potentially contaminated structures. Ground intrusive activities include, but are not limited to, soil/waste excavation and handling, test pitting or trenching, and the installation of soil borings or monitoring wells.

Periodic monitoring for VOCs will be required during non-intrusive activities such as the collection of soil and sediment samples or the collection of groundwater samples from existing monitoring wells. “Periodic” monitoring during sample collection might reasonably consist of taking a reading upon arrival at a sample location, monitoring while opening a well cap or overturning soil, monitoring during well baling/purging, and taking a reading prior to leaving a sample location. In some instances, depending upon the proximity of potentially exposed individuals, continuous monitoring may be required during sampling activities. Examples of such situations include groundwater sampling at wells on the curb of a busy urban street, in the midst of a public park, or adjacent to a school or residence.

VOC Monitoring, Response Levels, and Actions

VOCs must be monitored at the downwind perimeter of the immediate work area (i.e., the exclusion zone) on a continuous basis or as otherwise specified. Upwind concentrations should be measured at the start of each workday and periodically thereafter to establish background conditions, particularly if wind direction changes. The monitoring work should be performed using equipment appropriate to measure the types of contaminants known or suspected to be present. The equipment should be calibrated at least daily for the contaminant(s) of concern or for an appropriate surrogate. The equipment should be capable of calculating 15-minute running average concentrations, which will be compared to the levels specified below.

1. If the ambient air concentration of total organic vapors at the downwind perimeter of the work area or exclusion zone exceeds 5 parts per million (ppm) above background for the 15-minute average, work activities must be temporarily halted and monitoring continued. If the total organic vapor level readily decreases (per instantaneous readings) below 5 ppm over background, work activities can resume with continued monitoring.
2. If total organic vapor levels at the downwind perimeter of the work area or exclusion zone persist at levels in excess of 5 ppm over background but less than 25 ppm, work activities must be halted, the source of vapors identified, corrective actions taken to abate emissions, and monitoring continued. After these steps, work activities can resume provided that the total organic vapor level 200 feet downwind of the exclusion zone or half the distance to the nearest potential receptor or residential/commercial structure, whichever is less - but in no case less than 20 feet, is below 5 ppm over background for the 15-minute average.
3. If the organic vapor level is above 25 ppm at the perimeter of the work area, activities must be shutdown.
4. All 15-minute readings must be recorded and be available for State (DEC and NYSDOH) personnel to review. Instantaneous readings, if any, used for decision purposes should also be recorded.

Particulate Monitoring, Response Levels, and Actions

Particulate concentrations should be monitored continuously at the upwind and downwind perimeters of the exclusion zone at temporary particulate monitoring stations. The particulate monitoring should be performed using real-time monitoring equipment capable of measuring particulate matter less than 10 micrometers in size (PM-10) and capable of integrating over a period of 15 minutes (or less) for comparison to the airborne particulate action level. The equipment must be equipped with an audible alarm to indicate exceedance of the action level. In addition, fugitive dust migration should be visually assessed during all work activities.

1. If the downwind PM-10 particulate level is 100 micrograms per cubic meter (mcg/m^3) greater than background (upwind perimeter) for the 15-minute period or if airborne dust is observed leaving the work area, then dust suppression techniques must be employed. Work may continue with dust suppression techniques provided that downwind PM-10 particulate levels do not exceed 150 mcg/m^3 above the upwind level and provided that no visible dust is migrating from the work area.
2. If, after implementation of dust suppression techniques, downwind PM-10 particulate levels are greater than 150 mcg/m^3 above the upwind level, work must be stopped and a re-evaluation of activities initiated. Work can resume provided that dust suppression measures and other controls are successful in reducing the downwind PM-10 particulate concentration to within 150 mcg/m^3 of the upwind level and in preventing visible dust migration.
3. All readings must be recorded and be available for State (DEC and NYSDOH) and County Health personnel to review.

APPENDIX D

Site-Specific Health and Safety Plan

Site-Specific Health and Safety Plan

11-24 Wyckoff Avenue
Queens, New York 11385

December 19, 2025

Prepared for:

1124 Wyckoff LLC
2483 Coney Island Avenue
Brooklyn, New York 11223

Prepared by:

Roux Environmental Engineering and Geology, D.P.C.
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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**
Office Manager (OM)	Jeffrey Wills		Direct: (631) 630-2366 Mobile: (516) 637-0213
Project Principal (PP)	Noelle Clarke		Direct: (631) 630-2341 Mobile: (631) 807-6523
Project Manager (PM)	Valerie Sabatasso		Direct: (631) 630-2364 Mobile: (516) 509-9852
Site Supervisor (SS)	*Varies*		
Site Health and Site Safety Officer (SHSO)	*Varies*		
Office Health and Safety Manager (OHSM)	Nevin Pahlad, CSP, CHMM		Direct: (631) 630-2426 Mobile: (347) 885-6930
Corporate Health and Safety Director (CHSD)	Brian Hobbs, CIH, CSP		631-807-0193
WorkCare, Inc. (Formally AllOne Health)	Occupational Health Care Management Provider		800-350-4511
Client Emergency Contact	Chris Roberts		917-620-3598
Outside Assistance			
Agency	**Contact**	**Telephone**	**Address/Location**
Ambulance/emergency medical services (EMS)	FDNY EMS Station 57 (in NYC Health + Hospitals/Woodhull)	(718) 999-2000 **or 911**	131 Throop Ave, Brooklyn, New York
Police	NYPD – 83rd Precinct	(718) 574-1605 **or 911**	480 Knickerbocker Ave, Brooklyn, NY 11237
Fire	FDNY Engine 277 & Ladder 112	(718) 965-8277 **or 911**	582 Knickerbocker Ave, Brooklyn, NY 11221
Site Address	11-24 Wyckoff Avenue, Queens, New York 11385		

Directions to NYC Health + Hospitals/Woodhull located at 760 Broadway, Brooklyn, New York 11206:

1. Head northwest on Wyckoff Ave toward Cody Ave;

2. Turn left onto Halsey St;
3. Turn right onto Bushwick Ave;
4. Turn left onto Hart St;
5. Turn right onto Broadway;
6. Turn left onto Marcus Garvey Blvd;
7. Destination will be on the right.

Direction to Northwell Health-GoHealth Urgent Care located at 55-05 Myrtle Avenue, Queens, New York 11385:

1. Head northwest on Wyckoff Ave toward Cody Ave;
2. Wyckoff Ave turns right and becomes Madison St;
3. Destination will be on the left.

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 soil vapor, soil, and groundwater sampling activities being performed at the Site belonging to 1124 Wyckoff LLC (Wyckoff), located at 11-24 Wyckoff Avenue, Queens, New York (Site; **Figure 1**). These activities fall within the scope of operations covered by 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 sampling 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 Sarah Stern. The Site Supervisor (SS) and Site Health and Safety Officer (SHSO) for this project is to be determined and may vary based upon the task being implemented.

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 a 0.34-acre parcel and located at 11-24 Wyckoff Avenue (Block 3542, Lot 50) in the Ridgewood neighborhood of Queens, New York. The Site is presently developed with a 1-story industrial/ manufacturing style building. The building is "U" shaped and has an asphalt capped driveway/parking area located in the center area. The building currently has an active sub-slab depressurization system (SSDS) with monitoring points installed throughout the building. The Site has a single tenant (Telfar) which operates as a fashion company. The areas surrounding the Site are generally developed with industrial, manufacturing, and residential properties: CubeSmart Self Storage, single family residential homes, and L train subway (below grade) to the north; an industrial/manufacturing building to the south; unspecified storage/parking to the east; and single family residential homes to the west. The regional direction of groundwater flow in the vicinity of the Subject Property is presumed to be generally towards the south in the direction of Jamaica Bay and the Atlantic Ocean.

2.2 Site History

A review of historical sources, including historical aerial photographs, historical topographic maps, historical Certified Sanborn Maps, City Directory Abstract and interviews with Key Subject Property Managers indicate that the Site was developed prior to 1902. Until approximately 1980, the Site historically was developed with auto houses and as an auto laundry. The Site was also historically adjoined to the east with a gasoline filling station. The Site was operated as a knitting mill from approximately 1980-2006.

2.3 Known and Potential Releases of Hazardous Substances at the Site

Based on the information gathered during the Phase I ESA process conducted by Roux, contaminants of concern include chlorinated solvents in the soil, groundwater, and soil vapor from the Site Property's historical uses as part of the knitting mill, textile, and auto laundry operations. There is still potential that there is a source of chlorinated solvents in the soil that has not been identified. Additionally, the Site was adjoined to/surrounded by gasoline filling stations which contained buried gasoline tanks, and the Site was also located in an industrial/manufacturing-use area, all which have the potential to adversely impact the environmental quality of the Site. The Site was identified to have poor quality fill material with elevated semi-volatile organic compounds, specifically PAHs, in the vicinity of a previously closed-in-place 2,000-gallon underground storage tank (UST).

3. Scope of Work

In general, the scope of work will include the following tasks:

- Monitoring of the onsite SSDS;
- Investigation of soil vapor, soil, and groundwater to acquire data on environmental conditions; and
- Oversight of subcontractor activities including drilling activities using hand tools, a rotary-sonic drill rig, and a Geoprobe direct push drill rig.

If there are any changes with the scope a revision of the HASP will be required to address any new hazards.

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 is controlled by closed doors. When the Site is not operating, access to the Site is controlled by the following locked doors.

4.3 Buddy System

This section is not applicable for all components of the SOW described in Section 3. Some Site inspections, soil vapor, soil, and groundwater sampling, are completed by a single Roux employee. However, when completing these tasks, the single Roux employee may be accompanied either by Roux subcontractors or the Site caretaker/other representatives from Telfar. Any time Roux is on-site, Telfar is made aware and communications with Telfar 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: cell phones and visual hand signals.

As applicable, hand signals will be used according to the following:

Hand Signals

SIGNAL	MEANING
Hand gripping throat	Out of air, can't breathe
Grip partner's wrist	Leave area immediately
Hands on top of head	Need assistance
Thumbs up	I'm 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

The SOW does not always require the implementation of work zones; however, should the need arise, this section provides details for the proper execution of work zones at this Site**.**

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 be determined prior to the start of work. 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 and consistent with Roux's COVID-19 Interim Health and Safety Guidance which can be found in **Appendix C**.

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.

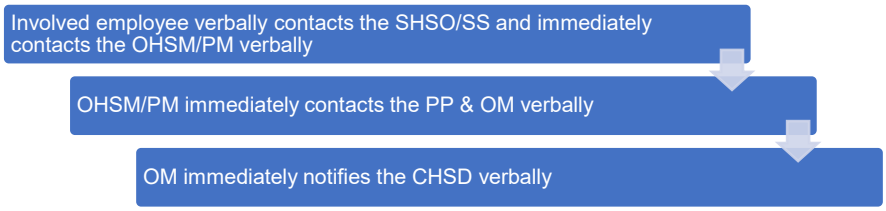
6.2 Emergency Alerting and Evacuation

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. (Formally AllOne Health), 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 Northwell Health-GoHelath Urgent Care and NYC Health + Hospitals/Woodhull 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 27 to 85 degrees Fahrenheit during the year in Queens, 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°F, and 10 degrees hotter than the average temperature of the five preceding days); and
- Implementing high heat procedures when the temperature reaches 95°F.

Roux's Heat Illness Prevention Plan is implemented when the when ambient temperatures exceed 80°F. Roux's Heat Illness Prevention Program can be found within **Appendix D**. 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 95°F. 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 105^{o}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 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 E**. 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 $\geq$ 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.
- A pre-calibrated multi-gas meter with combustible Lower Explosive Limit (LEL), oxygen (O2), carbon monoxide (CO), and hydrogen sulfide (H2S) sensors shall be used to monitor the potential for oxygen-deficient atmospheres, explosive concentrations of organic vapors, and toxic gases during intrusive operations. The calibration for this device will be performed using a known gas composition calibration mixture.

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. 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 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. 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.

Air Monitoring Summary and Action Levels Oxygen	
O_2 Reading in Breathing Zone (%)[1]	**Action**
20.9% O_2	Oxygen level normal
< 19.5% O_2	Oxygen deficient Interrupt task/Evacuate area
>23.5% O_2	Oxygen enriched Interrupt task/Evacuate area

1. Action levels based on USEPA Standard Operating Safety Guides; Table 5-1

Air Monitoring Summary and Action Levels Carbon Monoxide	
CO Reading in Breathing Zone (ppm)[1]	**Action**
<12.5 ppm	Inspect exhaust system for leaks or other sources of CO. Monitor initially and every 15 minutes during use of CO-generating equipment.
12.5-25 ppm	Ventilate area. Monitor continuously and record measurements. Contact PM.
>25 ppm	Cease Field Operations. Ventilate area.

1. Based upon the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Value (TLV) of 25 ppm as an 8-hour time weighted average (TWA) and OSHA's Permissible Exposure Limit (PEL) of 50 ppm as an 8-hour TWA concentration.

Air Monitoring Summary and Action Levels Combustible Gases	
Lower Explosive Limit (LEL) Reading	**Action**
< 4% LEL	Site activities will continue with normal monitoring
4% – 20% LEL	Stop work until levels dissipate to <4% LEL
> 20% LEL	Potential explosion hazard. Halt all site activities, research source of release, aerate work area, suppress source.
Air Monitoring Summary and Action Levels Hydrogen Sulfide	
Hydrogen Sulfide (H_2S) Reading	**Action**
<10 ppm	Site activities will continue with normal monitoring
≥10 ppm	Stop work until levels dissipate to <10 ppm; use mechanical ventilation if possible. Consult with CHSD if unable to reduce concentrations below 10 ppm.

8.6.2 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. The worker will, to his/her 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

There will be no confined space entry as part of this Scope of Work. Should confined space entry be needed, 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 SPSAs 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 SPSAs 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 will review all available Site maps showing buried utility lines to identify potential hazards.

8.13.2 Subsurface Work

Subsurface work activities will require adherence to Roux's Corporate Subsurface Utility Clearance Management program found within **Appendix F**.

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 G**. 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.

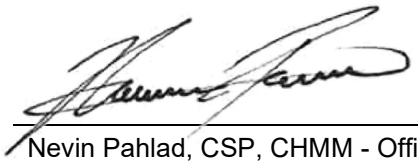
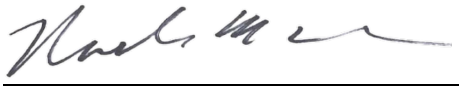
I have read and understand this Site-Specific Health and Safety Plan. I will comply with the provision contained therein.

Site/Project: 11-24 Wyckoff Avenue, Queens, New York

Name & Company	Signature	Date

10. Approvals

By their signature, the undersigned certify that this HASP is approved and will be utilized at the 11-24 Wyckoff Avenue, Queens, New York Site.

TBD – Site Health and Safety Officer	Date
 Nevin Pahlad, CSP, CHMM - Office Health and Safety Manager	December 19, 2025 Date
Valerie Sabatasso – Project Manager	December 19, 2025 Date
 Noelle Clarke – Project Principal	December 19, 2025 Date

TABLES

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
VOLATILE ORGANIC COMPOUNDS (VOCs)									
1,1,1-Trichloroethane	71-55-6	TWA 350 ppm 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. BP: 165°F Fl.Pt. = NA LEL: 7.5% UEL: 12.5% Combustible Liquid, but burns with difficulty
1,1,2,2-Tetrachloroethane	79-34-5	TWA 1 ppm	Ca TWA 1 ppm (7 mg/m^3)	TWA 5 ppm (35 mg/m^3)	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 BP: 296°F Fl.Pt. = NA LEL: NA UEL: NA Noncombustible Liquid
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1	TWA 1000 ppm STEL 1250 ppm	TWA 1000 ppm (7600 mg/m3) 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.] BP: 118°F Fl.Pt. = NA LEL: NA UEL: NA Noncombustible Liquid at ordinary temperatures, but the gas will ignite and burn weakly at 1256°F.
1,1,2-Trichloroethane	79-00-5	TWA 10 ppm	Ca TWA 10 ppm (45 mg/m3) [skin]	TWA 10 ppm (45 mg/m3)	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 BP: 237°F Fl.Pt. = NA LEL: 6% UEL: 15.5% 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. BP: 135°F Fl.Pt. = 2°F LEL: 5.4% UEL: 11.4% Class IB Flammable Liquid Fl.P. below 73°F and BP at or above 100°F.
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 BP: 89°F Fl.Pt. = -2°F LEL: 6.5% UEL: 15.5% 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				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 Fl.Pt. = 234.9°F
1,2,4-Trichlorobenzene	120-82-1	STEL 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 BP: 416°F Fl.Pt. = 222°F LEL (302°F): 2.5% UEL (302°F): 6.6% Class IIIB Combustible Liquid: Fl.P. at or above 200°F. Combustible 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
1,2-Dibromo-3-chloropropane	96-12-8		Ca	[1910.1044] 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.] BP: 384°F Fl.Pt. = (oc) 170°F LEL: NA UEL: NA Class IIIA Combustible Liquid: Fl.P. at or above 140°F and below 200°F.
1,2-Dibromoethane	106-93-4	None listed	Ca TWA 0.045 ppm C 0.13 ppm [15-minute]	TWA 20 ppm C 30 ppm 50 ppm [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] BP: 268°F Fl.Pt. = 50°F LEL: NA UEL: NA Noncombustible Liquid
1,2-Dichlorobenzene	95-50-1	TWA 25 ppm 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] BP: 357°F Fl.Pt. = 1°F LEL: 2.2% UEL: 9.2% 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) ST 2 ppm (8 mg/m3)	TWA 50 ppm C 100 ppm 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.] BP: 182°F Fl.Pt. = 56°F LEL: 6.2% UEL: 16% 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	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] BP: 206°F Fl.Pt. = 60°F LEL: 3.4% UEL: 14.5% 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. BP: 343°F Fl.Pt. = 146°F LEL: 2.02% 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] BP: 345°F Fl.Pt. = 150°F LEL: 2.5% UEL: NA Combustible Solid, but may take some effort to ignite.
1,4-Dioxane	123-91-1	TWA 20 ppm	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. BP: 214°F Fl.Pt. = 55°F LEL: 2.0% UEL: 22% 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
2-Butanone	78-93-3	TWA 200 ppm STEL 300 ppm	TWA 200 ppm (590 mg/m3) 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 BP: 175°F Fl.Pt. = 16°F LEL (200°F): 1.4% UEL (200°F): 11.4% Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F
2-Hexanone	591-78-6	TWA 5 ppm STEL 10 ppm	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 BP: 262°F Fl.Pt. = 77°F LEL: NA UEL: 8.0% 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 STEL 75 ppm	TWA 50 ppm (205 mg/m3) 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 BP: 242°F Fl.Pt. = 64°F LEL (200°F): 1.2% UEL (200°F): 8.0% Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F
Acetone	67-64-1	TWA 250 ppm 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 Fl.Pt. = 0°F LEL: 12.8% UEL: 2.5% Class IB Flammable liquid: Fl.P. below 73°F and BP at or above 100°F.
Benzene	71-43-2	TWA 0.5 ppm STEL 2.5 ppm	Ca TWA 0.1 ppm ST 1 ppm	TWA 1 ppm 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] BP: 176°F Fl.Pt. = 12°F LEL: 1.2% UEL: 7.8% 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.] BP: 155°F Fl.Pt. = NA LEL: NA UEL: NA 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 BP: 189°F Fl.Pt. = NA LEL: NA 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.] BP: 301°F Fl.Pt. = NA LEL: NA UEL: NA 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
Bromomethane	74-83-9	TWA 1 ppm	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.] BP: 38°F Fl.Pt. = NA (Gas) LEL: 10% UEL: 16.0% Flammable Gas, but only in presence of a high energy ignition source.
Carbon disulfide	75-15-0	TWA 1 ppm	TWA 1 ppm (3 mg/m3) ST 10 ppm (30 mg/m3) [skin]	TWA 20 ppm C 30 ppm 100 ppm (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.] BP: 116°F Fl.Pt. = -22°F LEL: 1.3% UEL: 50.0% Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F.
Carbon tetrachloride	56-23-5	TWA 5 ppm STEL 10 ppm	Ca ST 2 ppm (12.6 mg/m3) [60-minute]	TWA 10 ppm C 25 ppm 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 BP: 170°F Fl.Pt. = NA LEL: NA UEL: NA Noncombustible Liquid
Chlorobenzene	108-90-7	TWA 10 ppm	TWA 75 ppm	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 BP: 270°F Fl.Pt. = 82°F LEL: 1.3% UEL: 9.6% Class IC Flammable Liquid: Fl.P. at or above 73°F and below 100°F.
Chloroethane	75-00-3	TWA 100 ppm	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.] BP: 54°F Fl.Pt. = NA (gas), -58°F (liquid) LEL: 3.8% UEL: 15.4% Flammable Gas
Chloroform	67-66-3	TWA 10 ppm	Ca ST 2 ppm (9.78 mg/m3) [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 BP: 143°F Fl.Pt. = -82°F LEL: NA UEL: NA Noncombustible Liquid
Chloromethane	74-87-3	TWA 50 ppm STEL 100 ppm	Ca	TWA 100 ppm C 200 ppm 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.] BP: -12°F Fl.Pt. = NA (Gas) LEL: 8.1% UEL: 17.4% Flammable Gas

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
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. BP: 118-140°F Fl.Pt. = 36-39°F LEL: 5.6% UEL: 12.8% 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. BP: 219.7°F Fl.Pt. = NA LEL: NA 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.] BP: 177°F Fl.Pt. = 0°F LEL: 1.3% UEL: 8.0% 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 BP: 246-248°F Fl.Pt. = Greater than 200°F LEL: NA UEL: NA
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.] BP: -22°F Fl.Pt. = NA LEL: NA UEL: NA Nonflammable Gas
Ethylbenzene	100-41-4	TWA 20 ppm	TWA 100 ppm (435 mg/m^3) 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. BP: 277°F Fl.Pt. = 55°F LEL: 0.8% UEL: 6.7% Class IB Flammable Liquid below 73°F and BP at or above 100°F
Isopropylbenzene	98-82-8	TWA 1 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 BP: 306°F Fl.Pt. = 96°F LEL: 0.9% UEL: 6.5% Class IC Flammable Liquid: Fl.P. at or above 73°F and below 100°F
m&p-Xylenes	179601-23-1								
Methyl Acetate	79-20-9	TWA 200 ppm STEL 250 ppm	TWA 200 ppm (610 mg/m3) 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 BP: 135°F Fl.Pt. = 14°F LEL: 3.1% UEL: 16% 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
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 BP: 214°F Fl.Pt. = 25°F LEL: 1.2% UEL: 6.7% Class IB Flammable Liquid: Fl.P. below 73°F and BP at or above 100°F.
Methylene chloride	75-09-2	TWA 50 ppm	Ca	[1910.1052] TWA 25 ppm 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.] BP: 104°F Fl.Pt. = NA LEL: 13% UEL: 23% Combustible Liquid
Methyl-t-butyl ether	1634-04-4	TWA 50 ppm				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. BP: 131°F Fl.Pt. = -14°F LEL: NA UEL: NA
o-Xylene	95-47-6	TWA 100 ppm STEL 150 ppm (All isomers)	TWA 100 ppm (435 mg/m3) 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 BP: 292°F Fl.Pt. = 90°F LEL: 0.9% UEL: 6.7% Class IC Flammable Liquid: Fl.P. at or above 73°F and below 100°F
Styrene	100-42-5	TWA (20 ppm) STEL (40 ppm)	TWA 50 ppm (215 mg/m3) ST 100 ppm (425 mg/m3)	TWA 100 ppm C 200 ppm 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 BP: 293°F Fl.Pt. = 88°F LEL: 0.9% UEL: 6.8% Class IC Flammable Liquid: Fl.P. at or above 73°F and below 100°F
Tetrachloroethene	127-18-4	TWA 25 ppm STEL 100 ppm	Ca Minimize workplace exposure concentrations	TWA 100 ppm 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 BP: 250°F Fl.Pt. = NA LEL: NA UEL: NA Noncombustible Liquid, but decomposes in a fire to hydrogen chloride and phosgene
Sodium Hydroxide	1310-73-2	STEL C 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). BP: 2534°F Fl.Pt. = NA LEL: NA UEL: NA 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	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.] BP = 554°F Fl.Pt. = NA LEL = NA UEL = NA Noncombustible Liquid, but capable of igniting finely divided combustible materials.

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
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. BP: 118-140°F Fl.P: 36-39°F LEL: 5.6% UEL: 12.8% 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 BP: 234°F Fl.P: NA LEL: NA UEL: NA
Toluene	108-88-3	TWA 20 ppm	TWA 100 ppm (375 mg/m^3) ST 150 ppm (560 mg/m^3)	TWA 200 ppm C 300 ppm 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. BP: 232°F Fl.P: 40°F LEL: 1.1% UEL: 7.1% Class IB Flammable Liquid Fl.P. below 73°F and BP at or above 100°F.
Trichloroethene (TCE)	79-01-6	TWA 10 ppm STEL 25 ppm	Ca	TWA 100 ppm C 200 ppm 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. BP: 189°F Fl.Pt. = NA LEL(77°F): 8.0% UEL(77°F): 10.5% 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) BP: 75°F Fl.P: NA LEL: NA UEL: NA Noncombustible Liquid Nonflammable Gas
Vinyl Chloride (chloroethylene)	75-01-4	TWA 1 ppm	Ca	TWA 1 ppm 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.] BP: 7°F Fl.Pt. = NA (Gas) LEL: 3.6% UEL: 33.0% Flammable Gas
Xylene (m, o & p isomers)	108-38-3, 95-47-6, 106-42-3	TWA 100 ppm STEL 150 ppm	TWA 100 ppm (435 mg/m^3) ST 150 ppm (655 mg/m^3)	TWA 100 ppm (435 mg/m^3)	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 BP: 282°F, 292°F, 281°F Fl. Pt. 82°F, 90°F, 81°F LEL: 1.1%, 0.9%, 1.1% UEL: 7.0%, 6.7%, 7.0% Class IC Flammable Liquid at or above 73°F and below 100°F.
Zinc Oxide (dust)	7440-66-6	TWA 2 mg/m3 STEL 10 mg/m^3	TWA 5 mg/m^3 C 15 mg/m^3	TWA 15 mg/m^3 (total dust) TWA 5 mg/m^3 (resp dust)	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. BP: NA Fl.Pt. = NA LEL: NA UEL: NA Noncombustible Solid
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 BP: NA Fl.Pt. = NA LEL: NA 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
2-Methylnapthalene	91-57-6	TWA 0.5 ppm	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 Combustible solid BP: 466-468 ° F Fl.Pt. = 208 ° F LEL: NA 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 BP: 534 ° F Fl.Pt. = NA LEL: 0.6% UEL: NA
Acenaphthylene	208-96-8	https://cameochemicals.noaa.gov/chemical/16157				Inhalation, ingestion, skin and/or eye contact			Colorless crystalline solid BP: 509 to 527 ° F at 760 mm Hg Fl.Pt. = NA LEL: NA UEL: NA
Anthracene	120-12-7	None listed	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 BP: NA Fl.Pt. = NA LEL: NA UEL: NA 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. BP: 815° F at 760 mm Hg Fl.Pt. = NA LEL: NA UEL: NA
Benzo[a]pyrene	50-32-8	None listed	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 BP: NA Fl.Pt. = NA LEL: NA UEL: NA 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 BP: NA Fl.Pt. = NA LEL: NA 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. BP: NA Fl.Pt. = NA LEL: NA 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 BP: 896° F Fl.Pt. = NA LEL: NA UEL: NA
Chrysene	218-01-9	None listed	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 BP: NA Fl.Pt. = NA LEL: NA UEL: NA 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 BP: 975° F Fl.Pt. = NA LEL: NA 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 BP: 482° F Fl.Pt. = NA LEL: NA 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. BP: 563° F Fl.Pt. = NA LEL: NA 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 BP: 997° F Fl.Pt. = NA LEL: NA UEL: NA
Naphthalene	91-20-3	TWA 10 ppm	TWA 10 ppm (50 mg/m3) 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.] BP: 424°F Fl.P: 174°F LEL: 0.9% UEL: 5.9% 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. BP: 642° F Fl.Pt. = 340° F LEL: NA UEL: NA
Pyrene	129-00-0	None listed	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 BP: NA Fl.Pt. = NA LEL: NA UEL: NA Combustible Solids
METALS									
Aluminum	7429-90-5	TWA 1 mg/m3	TWA 10 mg/m^3 (total) TWA 5 mg/m^3 (resp)	TWA 15 mg/m^3 (total) 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 BP: 4221°F Fl.Pt. = NA LEL: NA UEL: NA Combustible Solid, finely divided dust is easily ignited; may cause explosions.
Antimony	7440-36-0	TWA 0.5 mg/m3	TWA 0.5 mg/m3 [*Note: The REL also applies to other antimony compounds (as Sb).]	TWA 0.5 mg/m3 [*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 BP: 2975°F Fl.Pt. = NA LEL: NA UEL: NA 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 BP: Sublimes Fl.Pt. = NA LEL: NA UEL: NA Metal: Noncombustible Solid in bulk form, but a slight explosion hazard in the form of dust when exposed to flame

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
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 BP: 1337°F Fl.Pt. = NA LEL: NA UEL: NA
Beryllium	7440-41-7	TWA 0.00005 mg/m^3	Ca C 0.0005 mg/m^3	STEL 0.002 mg/m^3 TWA 0.0002 mg/m^3	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 BP: 4532°F Fl.Pt. = NA LEL: NA UEL: NA Noncombustible Solid in bulk form, but a slight explosion hazard in the form of a powder or dust.
Cadmium	7440-43-9	TWA 0.01 mg/m^3 TWA 0.002 mg/m^3 (as Cd)	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. BP: 1409°F Fl.Pt. = NA LEL: NA UEL: NA 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. BP: 2714°F Fl.Pt. = NA LEL: NA UEL: NA
Chromium	7440-47-3	TWA 0.5 mg/m^3 (metal) TWA 0.003 mg/m^3 (water-soluble Cr III compounds) TWA 0.0002 mg/m^3 (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. BP: 4788°F Fl.Pt. = NA LEL: NA UEL: NA Noncombustible - will burn in dust form if heated in a flame
Cobalt	7440-48-4	TWA 0.02 mg/m3	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 BP: 5612°F Fl.Pt. = NA LEL: NA UEL: NA 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) 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. BP: 4703°F Fl.Pt. = NA LEL: NA UEL: NA Noncombustible - powdered form may ignite
Iron	7439-89-6	TWA 1 mg/m3	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. BP: NA Fl.Pt. = NA LEL: NA UEL: NA 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 BP: 3164°F Fl.Pt. = NA LEL: NA UEL: NA Noncombustible Solid in bulk form

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
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 BP: 1202°F Fl.Pt. = NA LEL: NA UEL: NA
Manganese	7439-96-5	TWA 0.02 mg/m3 [R] TWA 0.1 mg/m3 [I]	TWA 1 mg/m3 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 BP: 3564°F Fl.Pt. = NA LEL: NA UEL: NA Metal: Combustible Solid
Mercury	7439-97-6	TWA 0.01 mg/m3 STEL 0.03 mg/m3	Hg Vapor: TWA 0.05 mg/m3 [skin] 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.] BP: 674°F Fl.Pt. = NA LEL: NA UEL: NA Metal: Noncombustible Liquid
Nickel	7440-02-0	TWA 1.5 mg/m^3 [elemental] TWA 0.1 mg/m^3 [soluble inorganic compound] TWA 0.2 mg/m^3 [insoluble inorganic compound] 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. BP: 5139°F Fl.Pt. = NA LEL: NA UEL: NA 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 BP: 1425°F Fl.Pt. = NA LEL: NA 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.] BP: 1265°F Fl.Pt. = NA LEL: NA UEL: NA Combustible Solid
Silver	7440-22-4	TWA 0.1 mg/m3 [Metal, dust, and fume] 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 BP: 3632°F Fl.Pt. = NA LEL: NA UEL: NA 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 BP: 1621°F Fl.Pt. = NA LEL: NA UEL: NA
Thallium	7440-28-0	0.02 mg/m3 [I]	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 BP: NA Fl.Pt. = NA LEL: NA 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 BP: NA Fl.Pt. = NA LEL: NA 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
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 BP: NA Fl.Pt. = NA LEL: NA UEL: NA
PCBs									
PCBs (total)	11097-69-1, 53469-21-9	TWA 0.5 mg/m3 TWA 1 mg/m3	Ca TWA 0.001 mg/m3 Ca TWA 0.001 mg/m3	TWA 0.5 mg/m3 [skin] TWA 1 mg/m3 [skin]	Ca [5 mg/m3] 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 BP: 689-734°F, 617-691°F Fl.Pt. = NA, NA LEL: NA UEL: NA 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 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 BP: 102°F Fl.Pt. = -45°F LEL: 1.4% UEL: 7.6%

References

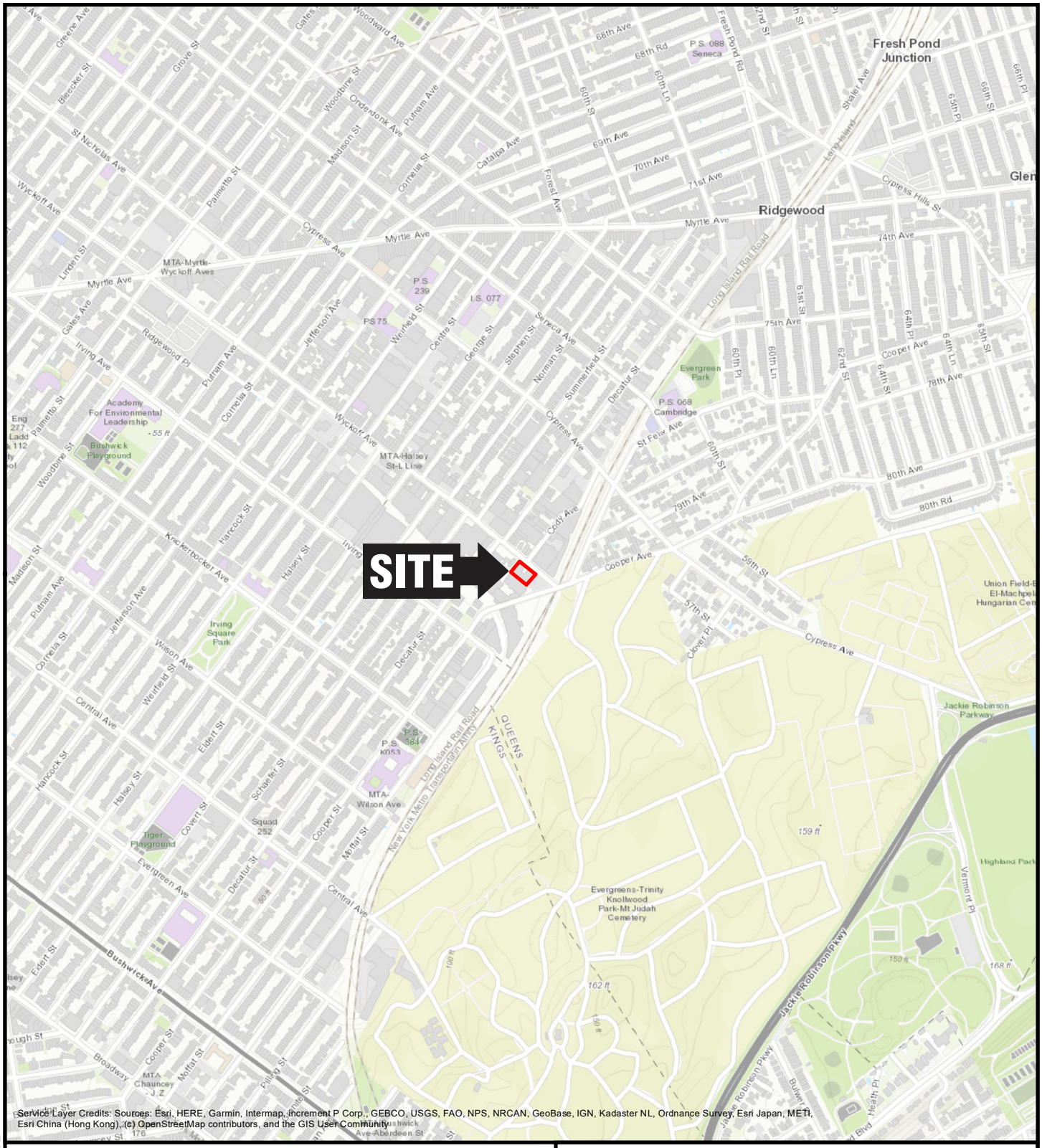
Centers for Disease Control and Prevention, 2018. *NIOSH Pocket Guide to Chemical Hazards.* The National Institute for Occupational Safety and Health.
American Conference of Governmental Industrial Hygienists (ACGIH), 2017. *2017 TLVs® and BEIs®.* Cincinnati, OH.
National Oceanic and Atmospheric Administration (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.
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.
Sax, N.I. and R.J. Lewis, 1989. *Dangerous Properties of Industrial Materials, 7th Edition.* Van Nostrand Reinhold. New York.

Abbreviations:

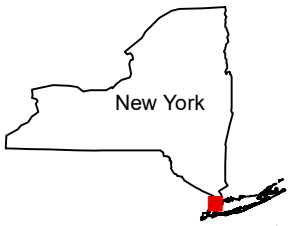
ACGIH – American Conference of Governmental Industrial Hygienists.
BP – boiling point at 1 atmosphere, °F
C – Ceiling, is a concentration that should not be exceeded during and part of the working exposure.
Ca – Carcinogenic.
CAS# - Chemical Abstracts Service registry number which is unique for each chemical.
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
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
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

11-24 WYCKOFF AVENUE, QUEENS, NEW YORK

Prepared for:

1124 WYCKOFF LLC

ROUX

Compiled by: S.S.	Date: 04/03/23
Prepared by: M.S.R.	Scale: AS SHOWN
Project Mgr: S.S.	Project: 4167.0001Y002
File: 4167.0001Y102.1.mxd	

FIGURE 1

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N

LEGEND

MUSTER AREA

SITE BOUNDARY

30 0 30'

Title:

SITE PLAN WITH EMERGENCY MUSTER AREA

11-24 WYCKOFF AVENUE, QUEENS, NEW YORK

Prepared for:

1124 WYCKOFF LLC

ROUX

Compiled by: S.S.	Date: 04/03/23	FIGURE
Prepared by: M.S.R.	Scale: AS SHOWN	2
Project Mgr: S.S.	Project: 4167.0001Y002	
File: 4167.0001Y102.2.mxd		

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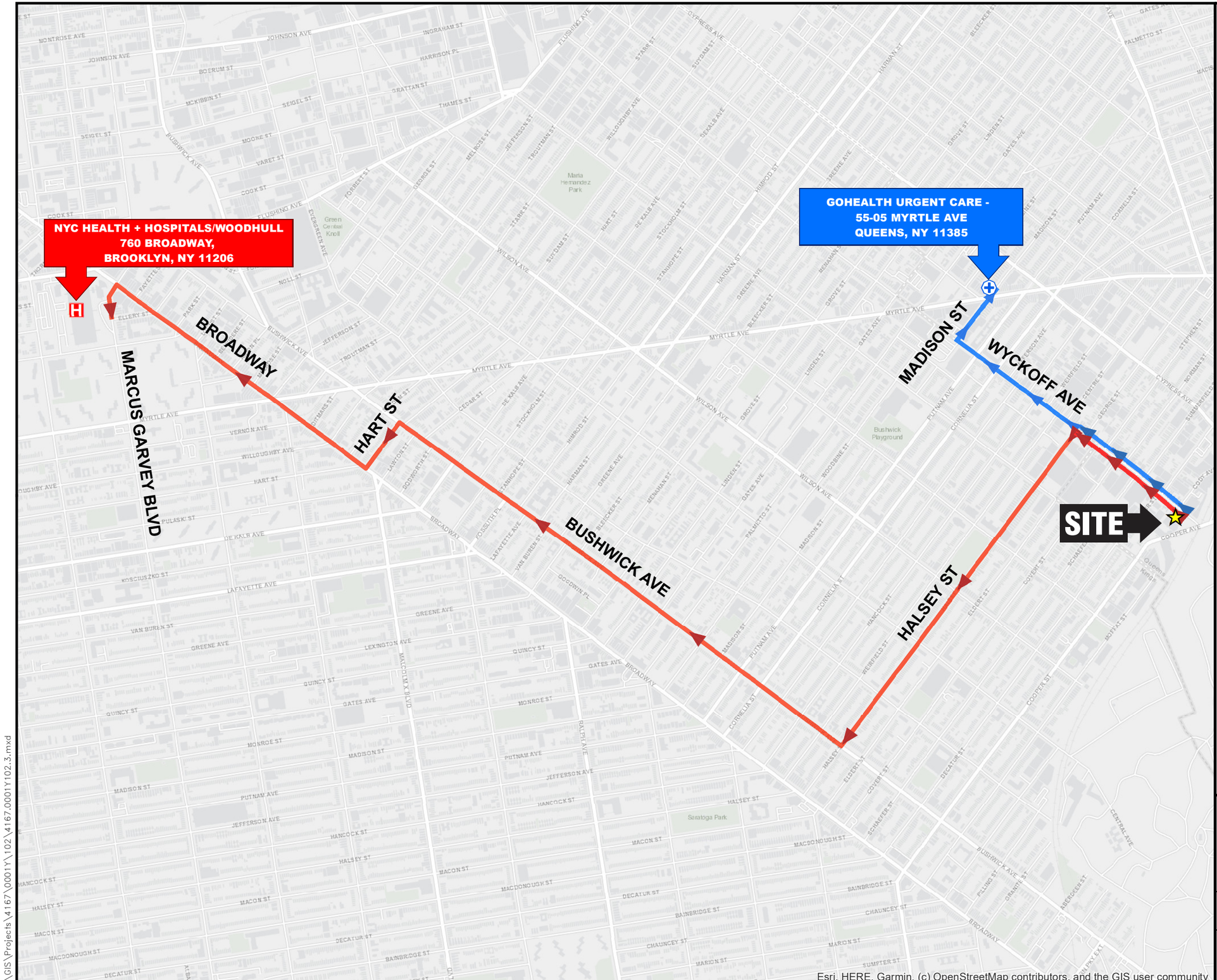

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DIRECTIONS TO HOSPITAL

1. HEAD NORTHWEST ON WYCKOFF AVE TOWARD CODY AVE
2. TURN LEFT ONTO HALSEY ST
3. TURN RIGHT ONTO BUSHWICK AVE
4. TURN LEFT ONTO HART ST
5. TURN RIGHT ONTO BROADWAY
6. TURN LEFT ONTO MARCUS GARVEY BLVD
7. DESTINATION WILL BE ON THE RIGHT

DIRECTIONS TO URGENT CARE

1. HEAD NORTHWEST ON WYCKOFF AVE TOWARD CODY AVE
2. WYCKOFF AVE TURNS RIGHT AND BECOMES MADISON ST
3. DESTINATION WILL BE ON THE LEFT

1,000 0 1,000
Feet

Title:

ROUTES TO URGENT CARE AND HOPSITAL

11-24 WYCKOFF AVENUE, QUEENS, NEW YORK

Prepared for:

1124 WYCKOFF LLC

ROUX

Compiled by: S.S.	Date: 04/03/23	FIGURE
Prepared by: M.S.R.	Scale: AS SHOWN	3
Project Mgr: S.S.	Project: 4167.0001Y002	
File: 4167.0001Y102.3.mxd		

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APPENDICES

A. Job Safety Analysis (JSA) Forms
B. Safety Data Sheets (SDSs) for Chemicals Used
C. COVID-19 Interim Health and Safety Guidance
D. Heat Illness Prevention Program
E. Personal Protective Equipment (PPE) Management Program
F. Subsurface Utility Clearance Management Program
G. Heavy Equipment Exclusion Zone Policy

APPENDIX A

Job Safety Analysis (JSA) Forms

JOB SAFETY ANALYSIS	Ctrl. No. CVD-19	DATE: 06/11/2021	☐ NEW ☒ REVISED	PAGE 1 of 2

JSA TYPE CATEGORY	WORK TYPE	WORK ACTIVITY (Description)
Generic	**Fieldwork**	**Working in Areas Affected by Coronavirus**

DEVELOPMENT TEAM	POSITION / TITLE	REVIEWED BY:	POSITION / TITLE
Kristina DeLuca	Health and Safety Specialist	Brian Hobbs	CHSD

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☐ LIFE VEST	☐ GOGGLES	☐ AIR PURIFYING RESPIRATOR	☒ GLOVES – Leather/cut-resistant in field and nitrile as needed
☒ HARD HAT – In field	☐ FACE SHIELD	☐ SUPPLIED RESPIRATOR	
☐ LIFELINE / BODY HARNESS	☐ HEARING PROTECTION	☒ PPE CLOTHING – High visibility vest in field	☐ OTHER
☒ SAFETY GLASSES – In field	☒ SAFETY SHOES – Steel/composite toe in fie		

REQUIRED AND / OR RECOMMENDED EQUIPMENT

Cloth face covering, nitrile gloves, hand soap, water source, hand sanitizer, disinfectant spray and disinfectant wipes.

Commitment to Safety – All personnel onsite will actively participate in SPSA performance by verbalizing SPSAs throughout the day.

SOCIAL DISTANCING: Maintain 6' of distance between yourself and all other people at all times. If you do not believe the scope of work can be conducted while maintaining this distance, contact your Project Manager immediately.

Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
1. Project Preplanning	N/A	• Review and follow COVID-19 CDC, Roux, Client and local orders/protocols. • Ensure all workers are fit for duty - anyone feeling sick should remain at home even if symptoms do not align with COVID-19. If a worker has been in contact with someone potentially positive or positive for COVID-19, contact your Office Manager. • Determine PPE needs and ensure adequate supply of disinfectant wipes/spray, soap and water or hand sanitizer at Site. Due to high demands and limited supply, plan ahead. • Use the minimum number of employees necessary to safely complete the work.
2. Mobilization	**Exposure:** Becoming infected or infecting co-workers	**Personal/Rental/Roux Owned Vehicle** • Do not carpool, unless all individuals are fully vaccinated. • Verify workers/other people are not approaching vehicle prior to exiting the vehicle. Maintain 6' of distance from general public, as appropriate. **Public Transportation** • Public transit should not be used unless absolutely necessary. Consider renting a car rather than taking public transit. If public transit is required, wear appropriate face covering/mask and apply social distancing (6 ft). Wash hands or use hand sanitizer immediately after. **Hotel Stay (Refer to COVID-19 H&S Guidance for more info)** • If a hotel stay is deemed necessary for the given field work, ensure that you clean your room upon initial arrival. • Place the "Do Not Disturb" placard on the room while away and limit housekeeping services to the extent feasible during your stay to minimize the reintroduction and spread of the virus from others.. Wash hands or use hand sanitizer often.
3. Tailgate Meeting	**Exposure:** Becoming infected or infecting co-workers	• Perform outside or indoors in areas with ample ventilation. • If unvaccinated, maintain at least a 6+ ft distance between you and others. • Discuss primary infection prevention measures listed below. • Discuss COVID-19 symptoms with coworkers and subcontractors to ensure fitness for duty. Anyone exhibiting signs or symptoms should be instructed to leave the Site, contact your Project Manager.

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[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; 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".

4. Site Activities	**Exposure:** Becoming infected or i nfecting co-workers	• Coordinate field activities at the beginning of the day (i.e. Tailgate meeting) to minimize time spent in crowded spaces or overlap while completing job tasks. • Don cloth face coverings as appropriate. • Apply social distancing (6+ ft) when interacting with others if unvaccinated. If anyone comes within 6 ft of you while conducting work and your work prevents you from moving away, politely ask them to move back. If others are unable to move from your space, stop work and leave area. • Minimize shaking hands or touching others. • Minimize sharing of equipment or other items with co-workers and subcontractors unless wearing appropriate PPE (e.g. nitrile gloves), as appropriate. • If anyone is experiencing COVID-19 signs or symptoms in your vicinity, stop work and leave the area. • Do not work in areas with limited ventilation with others. • Cover your mouth and nose with tissue or paper towel or with your elbow when coughing or sneezing and wash hands or use hand sanitizer immediately after. If sick contact SHSO/PM and leave Site immediately. • Clean work surfaces/areas with approved cleaners you're responsible for (ex: desk, office doorknob, computer, etc.) at least daily. • Avoid public spaces and going out to eat by bringing your own lunch to the Site. If performing work in high density urban areas, it is recommended all food must be consumed at or in your vehicle or within designated work trailer. Wash hands or use hand sanitizer before eating and immediately after.

Primary Infection Prevention Measures

- Wash your hands often with soap and water for at least 20 seconds.
  - If soap and water are not available, use an alcohol-based sanitizer that contains at least 60% alcohol. Key times to wash hands include after blowing your nose, coughing or sneezing, after using the restroom, and before eating or preparing food.
- Do not touch your eyes, face, nose and mouth with unwashed hands.
- Cover your mouth and nose with a tissue when you cough or sneeze or use the inside of your elbow. Throw potentially contaminated items (e.g. used tissues) in the trash.
- Avoid close contact/secondary contact with people and potentially contaminated surfaces.
  - Apply appropriate social distance (6+ feet).
  - Minimize handshaking/touching others and use caution when accessing public spaces.
- Clean frequently touched surfaces daily. Commonly touched items can include but are not limited to tables, doorknobs, light switches, countertops, handles, desks, phones, keyboard, toilets, sinks and field equipment. If surfaces are dirty, they should be cleaned with soap and water prior to disinfection. If surface cannot be cleaned/disinfected, then wash hands or use sanitizer as soon as possible.

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DAILY TAILGATE HEALTH & SAFETY CHECKLIST

Job Name: _____ **Date(s):** _____

Location: _____ **Project Number:** _____

Weather: **M-** **T-** **W-** **TH-** **F-**

- Tailgate meeting conducted at least once per day
- Involves everyone working at the Site
- Should familiarize everyone with work area & vicinity
- Work permit procedures
  Emergency access/exits & "muster" point
- Emergency contacts/phone #s, hospital/urgent care route
- Every worker on-site has Stop Work Authority
- Provide details of work planned for the day
- Review the Job Safety Analysis (JSA) and work hazards
- Required PPE for the planned work
- HASP is reviewed
- Ensure safety equipment is on Site
- Test required safety equipment (such as emergency stop controls)
- Can also include inspection of tools and equipment to be used

Instructions: Please only use form for Monday through Friday of one calendar week.

#	CHECKLIST ITEM	DATE				
		M-	T-	W-	TH-	F-
1	The HASP (including emergency phone #s) has been reviewed & signed by Roux staff & subcontractors.					
2	Personnel are OSHA-40 hour HAZWOPER trained (if applicable).					
3	Emergency muster points known, urgent care and ER identified.					
4	Applicable JSAs have reviewed by subcontractors					
5	Roux personnel & subcontractors have discussed hazards associated with Site-specific work.					
6	Any potential slips, trips or fall hazards have been identified.					
7	All workers have appropriate PPE., and have signed in for the day (2nd Page).					
8	Fit for duty: No worker is under the influence of any drugs, alcohol or any other substance that can alter your ability to work safely.					
9	Critical equipment on-Site is noted and is in safe working order.					
10	Site control has been established and exclusion zone is identified (using safety cones or barricades to mark out work area).					
11	Appropriate permits have been obtained (if applicable).					
13	Lock Out / Tag Out procedures have been followed where applicable, for electric and water.					
14	Electrical power operated tools shall be properly grounded and used with a GFCI.					
15	Traffic Safety Control measures have been put in place where applicable.					
16	All vessels containing flammable/corrosive material are labeled and SDSs are available for all chemicals.					
17	Subsurface work : Drilling, trenching, etc. conducted according to required Protocol, including pre-field preparations, utility mark-out and "hand" clearing.					
18	Excavated soils are properly stored; excavations are properly shored/sloped and barricaded.					
19	Good housekeeping procedures are meet; food and beverages are consumed outside of the work area.					
20	A first aid kit, eye wash bottle/station, and fire extinguishers are available for use.					
21	No smoking signs are visibly posted in areas of potential flammable vapors.					
Initial on right to verify that these actions have taken place. If the action does not apply, use N/A.						

Comments and Exceptions _____

_____

NOTE: THIS IS A TWO-SIDED FORM; PLEASE COMPLETE BOTH SIDES

DAILY TAILGATE HEALTH & SAFETY MEETING LOG

Job Name _____ **Date(s)** _____

Location: _____ **Project Number:** _____

SUBCONTRACTORS: _____

Activities Planned _____

_____

_____

Name	Company	Mon.	Tue.	Wed.	Thur.	Fri.

JOB SAFETY ANALYSIS	Ctrl. No. GEN-005	DATE 7/10/2020	☐ NEW ☒ REVISED	PAGE 1 of 2

JSA TYPE CATEGORY	WORK TYPE	WORK ACTIVITY (Description)
GENERIC	**Construction**	**Cutting with Gas-powered Saw, Sawzall or Plasma Cutter**

DEVELOPMENT TEAM	POSITION / TITLE	REVIEWED BY:	POSITION / TITLE
Ray Greenidge	Office Health and Safety Manager	Brian Hobbs	Corporate Health & Safety Manager

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☒ REFLECTIVE VEST ☒ HARD HAT ☐ LIFELINE / BODY HARNESS ☒ SAFETY GLASSES	☐ GOGGLES ☒ FACE SHIELD (gas powered saw and plasma cutter) ☒ HEARING PROTECTION ☒ SAFETY SHOES: Steel-toed boots	☐ AIR PURIFYING RESPIRATOR ☐ SUPPLIED RESPIRATOR ☒ PPE CLOTHING: Fluorescent Long sleeved shirt and / or reflective safety vest	☒ GLOVES: Cut-resistant, leather, nitrile ☒ OTHER: Chaps for gas powered saw. Welding suit for plasma cutting.

REQUIRED AND / OR RECOMMENDED EQUIPMENT

Sawzall/extension cord

COMMITMENT TO SAFETY- All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing SPSAs

Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
1. Set up/ Secure work area.	**1a. CONTACT:** Personnel could enter the work area	1a. Establish the work zone using 42" cones, caution tape, or fixed rigid barrier. Inform others of work activity.
2. Precutting procedure.	**2a. CONTACT:** Improper blade, malfunctioning guards, unsecured materials, flying debris	2a. Inspect all equipment for defects, replace or service if not functioning optimally. Check that all guards are working and in place, replace if missing. Ensure that blades are sharp and clean to avoid binding and/or burning. Cut on flat/secure work surfaces. Do not cut badly warped wood or boards with knots or nails. 2a. Unplug saw before handing it off to another person. 2a. Wear safety glasses, long-sleeved shirt and leather gloves. Utilize job specific PPE such as welding jacket or chaps when using gas powered saw or a plasma cutter.
	2b. EXPOSURE: Loud noises, dust, bright UV light	2b. When using gas powered saw, wet down area to be cut prior to cutting if high dust levels are anticipated. 2b. Wear hearing protection. Wear a dust mask if large amounts of dust are expected; cut upwind if possible. 2b. When plasma cutting, wear a face shield with shaded glasses rated to block UV light generated by the plasma cutter.
	2c. ENERGY SOURCE: Potential for electric shock	2c. Inspect extension cord for damage. If damaged, tag out and repair / replace. Do not operate saw while standing in water. Ensure GFCI protection at outlet or via attachment. 2c. Ensure all electrical equipment is rated for the task.

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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 [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
3. Saw Cutting.	**3a. CONTACT:** Fingers could be cut, lacerated or amputated by reciprocating blade; also flying debris and sparks	3a. Cut away from body. Keep fingers away from moving blade. No loose clothing. Never leave saw running unattended. Unplug saw before changing blades or making adjustments / repairs. Set-up barrier to contain sparks. Cut on flat/secure work surfaces. 3a. Wear Safety glasses 3a. Ensure that the saw blade stops rotating/reciprocating before placing saw on the ground.
	3b. CONTACT: Amputation and line of fire injury.	3b. Maintain a minimum 15-foot exclusion zone and ensure that operator and other personnel are kept out of the line-of- fire of the equipment.
	3c. FALL: Tripping hazards caused by cutting/grinding debris, extension cords.	3c. Keep debris generated in designated storage containers. Keep work area free of Slip, Trip and Fall hazards. 3c. Do not route extension cords through walking/working path.
	3d. EXERTION/ERGONOMICS: Lifting heavy or awkward materials may cause muscle strain.	3d. Maintain Proper Body Position while operating lifting and moving with equipment. Keep load close to body, knees bent, and back straight. 3d. Take frequent breaks or switch personnel if cutting for an extended period of time.
	3e. EXPOSURE: Personnel may be exposed to fire hazard during Hot Work Activities.	3e. Complete Hot Work Permit, Designate Fire Watch. 3e. Conduct work zone inspection: – Verify that all combustible or flammable materials or equipment fuel sources have been removed from within 35 feet of the proposed hot work. – If combustible or flammable materials or equipment fuel sources have not been removed from within 35 feet of the hot work, verify that engineering and procedural controls have been emplaced: curtains, blankets, wetting, ventilation. 3e. Two 20-lb. Type ABC Fire extinguishers required. 3e. Conduct continuous air monitoring / Lower Explosive Limit (LEL) screenings. Action Level: 10% of the LEL. 3e. If ambient air concentrations exceed LEL Action Levels, STOP WORK and contact supervisor. 3e. Wear hard hat, long sleeved-shirt and safety glasses. Utilize job specific PPE such as welding jacket or chaps and welding glasses when using gas powered saw or a plasma cutter.
4. Secure area when leaving tools unattended.	**4a. CONTACT:** Unauthorized personnel may enter the work area	4a. Unplug saw when not being used. Store equipment in designated storage areas when not being used.
	4b. FALL: Slip/trip/fall	4b. Store tool in designated storage location when it is not being used, secure all extension cords, keep all equipment out of walkways. 4b. Keep work area free of Slip, Trip and Fall Hazards.

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JOB SAFETY ANALYSIS	Ctrl. No. GEN-006	DATE 7/10/2020	☐ NEW ☒ REVISED	PAGE 1 of 2

JSA TYPE CATEGORY:	WORK TYPE:	WORK ACTIVITY (Description):	
Generic	**Drilling**	**Direct Push Soil Borings / Well Installation**	
DEVELOPMENT TEAM	**POSITION / TITLE**	**REVIEWED BY:**	**POSITION / TITLE**
Timothy Zei	Project Hydrogeologist	Raymond Olson	Office Health & Safety Manager
		Brian Hobbs	Corporate Health & Safety Manager

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☐ LIFE VEST ☒ HARD HAT ☐ LIFELINE / BODY HARNESS ☒ SAFETY GLASSES	☐ GOGGLES ☐ FACE SHIELD ☒ HEARING PROTECTION: (as needed) ☒ SAFETY SHOES: Composite-toe or steel toe boots	☐ AIR PURIFYING RESPIRATOR ☐ SUPPLIED RESPIRATOR ☒ PPE CLOTHING: Fluorescent reflective vest or high visibility clothing, Long Sleeve Shirt	☒ GLOVES: Leather, Nitrile and cut resistant ☒ 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 & Flags, "Work Area" Signs, Water

COMMITMENT TO SAFETY- All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing SPSAs

EXCLUSION ZONE (EZ): Maintain 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 foot print of a structure to be demolished.

"SHOW ME YOUR HANDS"
Driller and helper should show that hands are clear from 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 prior to mobilization. 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. 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. 1a. When backing up 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. 1a. Inspect the driving path for uneven terrain. Level or avoid if needed. 1a. Drill rig should have a minimum **exclusion zone which encompasses its tip radius** for non-essential personnel (i.e., driller helper, 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. 1b. Do not climb over stored materials/equipment; walk around. Practice good housekeeping. 1b. Use established pathways and walk on stable, secure ground.
	1c. CONTACT: Crushing from roll-over.	1c Geoprobe should cross all hills/obstructions head on with the mast down to reduce risk of roll-over.
2. Raising tower/derrick of drill rig	**2a. CONTACT:** Overhead hazards.	2a. Prior to 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. 2a. Maintain a safe distance of 10' from overhead structures.
	2b. CONTACT: Pinch Points/Amputation Points when raising the rig and instability of rig	2b. Inspect the equipment prior to use and avoid pinch/amputation points. 2b. Lower outriggers to ensure stability prior to raising rig tower/derrick. 2b. If the rig needs to be mounted, be sure to use three points of contact.
3. Advancement of drilling equipment and well installation	**3a. CONTACT:** Flying debris	3a. Be aware of and avoid potential lines of fire 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. 3b. Stand upwind and keep body away from rig. 3b. Dust mask should be worn if conditions warrant. 3b. Wear hearing protection when the drill rig is in operation.

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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 [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
3. Advancement of drilling equipment and well installation (Continued)	**3a. CONTACT:** Flying debris **3b. EXPOSURE:** Noise and dust. **3c. FALL:** Slip/trip/fall hazards. **3d. CAUGHT:** Limb/extremity pinching; abrasion/crushing. **3e. CONTACT:** Equipment imbalance during advancement of drill equipment. **3f. EXPOSURE:** Inhalation of contamination/vapors. **3g. EXERTION:** Potential for muscle strain/injury while lifting and installing well casings, lifting sand bags, and/or lifting rods.	3c. Contain drill cuttings and drilling water to prevent fall hazards from developing in work area. 3c. See 1b. 3d. Ensure all Emergency Safety Stop buttons function properly. 3d. Always wear leather gloves when making connections and using hand tools; wear cut-resistant (i.e., Kevlar) gloves when handling cutting tools. 3d. Inspect the equipment prior to use for potential pinch/amputation points. Keep hands away from pinch/amputation points and use of tools is preferable compared to fingers and hands. 3d. Inspect drill head for worn surface or missing teeth; replace if damaged or blunt. 3d. Ensure all jewelry is removed, loose clothing is secured, and PPE is secured close to the body. 3d. All non-essential personnel should stay away from the immediate work area; position body out of the line-of-fire of equipment. 3d. Drillers and helpers will understand and use the "Show Me Your Hands" Policy. 3d. Spinning rods/casing have an **exclusion zone of tip radius** while in operation. 3e. Drillers will advance the borehole with caution to avoid causing the rig to become imbalanced and/or tip. 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. 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. Monitor ambient air for dangerous conditions using a calibrated photoionization detector (PID) to periodically monitor the breathing zone of the work area. 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. 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. Keep back straight and bend at the knees. 3g. Utilize team lifting for objects over 50lbs. 3g. Use mechanical lifting device for odd shaped objects.
4. Remove sample liner.	**4a. EXERTION:** Potential for muscle strain/injury while removing liner from probe rod. **4b. CONTACT:** Pinch points and cuts **4c. EXPOSURE:** Inhalation and/or dermal contact with contaminants.	4a Utilize team lifting for objects over 50lbs. 4a. Use hydraulic liner extruder if available. 4b. Place liner on sturdy surface when opening. 4b. Don cut-resistant gloves and use appropriate liner cutter when opening liners. 4b. Always cut away from the body. 4c. Wear chemical-resistant disposable gloves when handling liners. 4c. See 3e.
5. Decontaminate equipment.	**5a. EXPOSURE/CONTACT**: To contamination (e.g., Separate Phase Hydrocarbons (SPH), contaminated groundwater, vapors). **5b. EXPOSURE:** To chemicals in cleaning solution including ammonia.	5a. Wear chemical-resistant disposable gloves and safety glasses. 5a. Contain decontamination water so that it does not spill. 5a. Use an absorbent pad to clean spills, if necessary. 5a. Spray equipment from side angle, not straight on, to avoid backsplash. 5a. See 3b. 5b. See 4a. Review SDS to ensure appropriate precautions are taken and understood.

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JOB SAFETY ANALYSIS	Ctrl. No. GEN-007	DATE 7/10/2020	☐ NEW ☒ REVISED	PAGE 1 of 2

JSA TYPE CATEGORY	WORK TYPE	WORK ACTIVITY (Description)
GENERIC	**General Site Activity**	**Driving**

DEVELOPMENT TEAM	POSITION / TITLE	REVIEWED BY:	POSITION / TITLE
Valerie Sabatasso	Staff Scientist	Brian Hobbs	Corporate Health & Safety Manager

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☐ LIFE VEST ☒ HARD HAT: when outside vehicle ☐ LIFELINE / BODY HARNESS ☒ SAFETY GLASSES: when outside vehicle	☐ GOGGLES ☐ FACE SHIELD ☒ HEARING PROTECTION ☒ SAFETY TOE BOOTS: when outside vehicle	☐ AIR PURIFYING RESPIRATOR ☐ SUPPLIED RESPIRATOR ☒ PPE CLOTHING: high visibility vest, when outside vehicle	☒ GLOVES: Leather/ cut-resistant level 2 ☐ OTHER _____

REQUIRED AND / OR RECOMMENDED EQUIPMENT

Motor Vehicle (i.e. car, truck, SUV)

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EXCLUSION ZONE (EZ): Maintain 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 foot print of a structure to be demolished.

Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
1. Driving to/leaving Site	**1a. CONTACT:** 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. *Common factors that may lead to CONTACT incident, but not limited to: • distracted driving (cell phone, GPS, radio, billboards, "rubber necking") • lack of situational awareness • unfamiliarity with traffic patterns/road layout • weather conditions (wet/icy roads, hydroplaning, black ice) • weariness • high speeds • obstructed vision (solar glare, debris on windshield, blind spots) • changes in travel pathway (construction, snow banks, non-operational signals, potholes, detours, special events) • improper vehicle maintenance (non-operational signal light, worn tires, cracked windshield, ineffective wipers) • 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. 1a. Complete a basic vehicle inspection before driving. 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. 1a. Do not hang items in car that can obstruct your view or become projectiles in a collision. 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. 1a. Follow posted speed limits and obey traffic signals and roadway signs. 1a. Always wear your seat belt and shoulder harness when driving. 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. 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 they are when traffic laws require. 1a. Apply the Smith Five Keys® of safe driving • Aim High in Steering® - Expand eye lead time to a minimum of 15 seconds • Get the Big Picture® - Maintain proper a 4 second minimum following distance at all times - Scan mirrors every 5-8 seconds to achieve a circle of awareness - Position your vehicle so you can see relevant/non-relevant objects • Keep Your Eyes Moving® - Try to maintain about 180 degrees of visibility - Avoid blank and fixed stares. Avoid focusing on one object for more than 2 seconds • Leave Yourself an Out® - Avoid traveling in traffic clusters - Surround yourself with space - 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 [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
1. Driving to/leaving Site (cont'd)	**1a. CONTACT:** 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® - Maintain eye contact with on-coming vehicles/pedestrians - Use warning devices (e.g., hand signals, high-lights, horns etc.) - Proper timing is essential 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 1a. Avoid sudden turns and stops. Don't drive recklessly – be in control of vehicle at all times. 1a. In inclement weather, first determine if work can be POSTPONED. Otherwise, plan according to weather conditions including checking forecast along entirety of 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 to operators manual and understand operating procedure prior to engaging 4-wheel drive. If at any point on your drive weather becomes too severe to proceed safely pull over if safe to do so or seek nearest cover (e.g., overpass) 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. 1a. Never operate a vehicle under the influence of alcohol or illegal substances or medications affecting your performance. 1a. Keep your eyes on the road. Do not call or talk on cellular phones. Pull over to a safe location if you must answer or make a call. 1a. When parking, pull-through when possible. If backing is required visually inspect 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:** Personal injury (broken fingers/hand) while entering or exiting vehicles **2b. FALL:** Personal injury (twisted ankle, deep contusion, concussion, broken wrist/arm, etc.) from slip/fall on uneven or unstable or slippery surface while exiting/entering vehicle **2c. CONTACT:** 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 door and vehicle to avoid pinch points. 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 hands to support yourself, by holding the car door and/or steering wheel, when exiting the vehicle. 2c. Check both directions for traffic before opening door. Do not exit vehicle if traffic does not permit you to exit safely 2c. Check anticipated path of door prior to opening, do not open door into any obstructions (e.g., bollards, high curbing)

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JOB SAFETY ANALYSIS	Ctrl. No. GEN-009	DATE: 7/10/2020	☐ 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**		

DEVELOPMENT TEAM	POSITION / TITLE	REVIEWED BY:	POSITION / TITLE
Michael Sarni	Technician	Brian Hobbs	Corporate Health & Safety Manager

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☐ LIFE VEST	☐ GOGGLES	☐ AIR PURIFYING RESPIRATOR	☒ GLOVES: Cut-resistant gloves
☒ HARD HAT	☐ FACE SHIELD	☐ SUPPLIED RESPIRATOR	☐ OTHER:
☐ LIFELINE / BODY HARNESS	☐ HEARING PROTECTION	☒ PPE CLOTHING: Fluorescent long sleeve shirt or long sleeve shirt and reflective safety vest.	
☒ SAFETY GLASSES	☒ SAFETY SHOES: Steel or composite toe		

REQUIRED AND / OR RECOMMENDED EQUIPMENT

Mobile Drum Carrier, safety cones, and caution tape

COMMITMENT TO SAFETY- All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing SPSAs

EXCLUSION ZONE (EZ): Maintain Minimum Heavy Equipment Exclusion Zone around equipment (i.e. forklift) 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 foot print of a structure to be demolished.

Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
1. Preparing for and Inspection of Drum	**1a. FALL:** Tripping/falling due to uneven surface. Loose debris/garbage in work area.	1a. Clear area of loose garbage and debris. Inspect 55-gal drums for proper condition, labeling, check drum ring and bolts for tightness, inspect mobile drum carrier. 1a. Do a Test Lift to get a general sense of the weight of the drum. 1a. Inspect and use established pathways to avoid uneven terrain, weather-related hazards (i.e., debris, puddles, ice, etc.), and other obstructions. 1a. Secure work area and coordinate and communicate the planned work activities with other personnel working in the area. 1a. Delineate work area with 42” safety cones.
	1b. CONTACT/EXPOSURE: Drums could potentially be damaged or contain hazardous material. Mobile drum carrier could potentially be in poor working condition causing malfunctioning during operation.	1b. Prior to inspecting drums don cut-resistant gloves. If drum is not properly labeled, do not open and cease all drum transport activities. Immediately contact project manager and inform him/her of drum situation. 1b. Do not continue drum transport activities until further actions are determined by the project manager. 1b. If the drum is properly labeled, but leaking, improperly sealed or in poor condition, place drum in an over-pack drum. 1b. Inspect 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 ring/ tightening bolts	1c. Keep back straight and knees slightly bent while securing drum ring/tightening bolt. Wear cut-resistant gloves.

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2. Position drum clamp tightly in between drum ribs, securing drum clamp to drum with chain	**2a. CAUGHT:** Pinching fingers between drum clamp and handle/chain.	2a. Attach drum clamp with chain and tighten until snug. Do not place hands between drum clamp and drum as the chain is tightened; wear cut resistant gloves. Keep face away from drum when handling in case of escaping vapors.
Assess **[1]JOB STEPS**	**Analyze** **[2]POTENTIAL HAZARDS**	**Act** **[3]CRITICAL ACTIONS**
3. Disengage safety latches on handle, pull handle down until drum is lifted off ground and safety latches are reengaged; slightly suspending drum off the ground	**3a. EXERTION/ CONTACT:** Potential muscle strain associated with lifting/engaging drum/handle. 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 handle while drum is secured with clamp so that safety latches can be engaged. Keep body out of the line of fire of the handle (do not position head above 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 toe boots.
	3b. CAUGHT: Fingers could be pinched while engaging/disengaging safety latches on handle	3b. Wear cut-resistant gloves while disengaging/reengaging safety latches. 3b. Avoid placing hands in pinch points.
4. Transport drums to designated location and disengage drum clamp (repeat Step 3 in reverse order)	**4a. FALL:** Tripping/ falling due to obstructions and uneven terrain. Potential for drum to fall during transport.	4a. Ensure transport path is free of potential obstructions that may cause the drum/carrier to become unstable. Position drum clamp between the ribs on the drum to prevent possible slipping.

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JOB SAFETY ANALYSIS Ctrl. No. GEN-013	DATE 7/10/2020	☐ NEW ☒ REVISED	PAGE 1 of 2

JSA TYPE CATEGORY	WORK TYPE:	WORK ACTIVITY (Description):
Generic	**Gauging and Sampling**	**Gauging and Sampling**

DEVELOPMENT TEAM	POSITION / TITLE	REVIEWED BY:	POSITION / TITLE
Brandon Tufano	Staff Geologist	Brian Hobbs	Corporate Health & Safety Manager

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☒ LIFE VEST ☒ HARD HAT ☐ LIFELINE / BODY HARNESS ☒ SAFETY GLASSES	☐ GOGGLES ☐ FACE SHIELD ☐ HEARING PROTECTION ☒ SAFETY SHOES: Composite-toe or steel toe boots	☐ AIR PURIFYING RESPIRATOR ☐ SUPPLIED RESPIRATOR ☒ PPE CLOTHING: Fluorescent reflective vest or high visibility clothing	☒ GLOVES: Leather, Nitrile and cut resistant ☒ OTHER: Knee pads, Insect Repellant, sunscreen (as needed)

REQUIRED AND / OR RECOMMENDED EQUIPMENT

42-inch Safety Cones, Caution Tape, Interface Probe 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 SPSAs

Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [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 pathway and plan for most suitable designated pathway prior to mobilization. 1a. Use established pathways, walk and/or drive on stable, secure ground and avoid steep hills or uneven terrain. 1a. If working near open water with an unguarded edge, wear life vest.
	1b. CONTACT: With traffic/third parties.	1b. Identify potential traffic sources and delineate work area with 42-inch traffic safety cones. Position vehicle to protect against oncoming traffic. Use caution tape to provide a more visible delineation of the work area if necessary. 1b. Wear appropriate PPE including high visibility clothing or reflective vest. 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. 4c. Use mechanical assistance or team lifting techniques when equipment is 50 lbs. or heavier. 4c. Make multiple trips to carry equipment.
	1d. EXPOSURE: To biological hazards.	1d. Inspect work area for bees and insects. 1d. Use insect/tick repellent as necessary.
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 well.
	2b. CAUGHT: Pinch/crush points associated with removing/replacing manholes and working with hand tools.	2b. Wear leather gloves or cut resistant gloves when working with well cover and hand tools. 2b. Use proper tools (ratchet and pry bar for well cover) and inspect before use. 2b. Do not put fingers under well cover.
	2c. CAUGHT: Pinch points associated with placing J-plug back onto PVC pipe.	2c. See 2b. 2c. Keep fingers out of line-of-fire when securing cap.
	2d. EXPOSURE: To potential hazardous vapors.	2d. No open flames/heat sources. 2d. To minimize exposure to vapors, allow well to vent after opening it and before sampling activities begin. 2d. Stand up-wind, if possible, to avoid inhaling vapors.
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. 3a. Insert and remove probe slowly to avoid splashing. 3a. Use an absorbent pad to clean probe.
	3b. CONTACT: With traffic.	3b. See 1b.

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Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [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. 4a. Wear cut-resistant gloves and chemical-resistant disposable gloves when sampling. 4a. Fill sample containers over purge container to avoid spilling water onto the ground. 4a. Use an absorbent pad to clean spills. 4a. When using a bailer to purge a well, pull the bailer slowly from the well to avoid splash hazards. 4a. When sampling or purging the water using a bailer, pour out water slowly to reduce the potential for splash hazards with groundwater. 4a. When using a tubing valve always remove the valve slowly after sample collection to release any pressure and avoid pressurized splash hazards. 4a. When collecting a groundwater sample always point sampling apparatus (tubing, bailer, etc.) away from face and body.
	4b. CONTACT: Personal injury from cuts, abrasions, or punctures by glassware or sharp objects.	4b. To avoid spills or breakage, place sample ware on even surface. 4b. Do not over tighten caps on glass sample ware. 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 cutting tools.
	4c. EXERTION: Muscle strain while carrying equipment.	4c. Use proper lifting techniques when handling/moving equipment, bend knees and keep back straight. 4c. Use mechanical assistance or team lifting techniques when equipment is 50 lbs. or heavier. 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 leather gloves when working with groundwater pumps. 4e. Never place hands on or near pinch points such as the wheel, clamps or other moving parts during pump operations. 4e. Use the correct mechanisms, such as a pump reel, to lower pump into well. 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. 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 container and pour liquids slowly so that they do not splash. 5a. Properly dispose of used materials/PPE in appropriate container in 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.). 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 decon area. 6a. Wear chemical-resistant disposable gloves and safety glasses. 6a. Use an absorbent pad to clean spills.
	6b. CAUGHT: Pinch points associated with handling hand tools	6b. See 2b. 6b. Inspect hand tools for sharp edges before decontaminating.

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JOB SAFETY ANALYSIS	Ctrl. No. GEN-015	DATE: 7/10/2020	☐NEW ☒REVISED	PAGE 1 of 2

JSA TYPE CATEGORY	WORK TYPE	WORK ACTIVITY (Description)	
GENERIC	**Site Recon**	**Mobilization/Demobilization**	
DEVELOPMENT TEAM	**POSITION / TITLE**	**REVIEWED BY:**	**POSITION / TITLE**
Rebecca Lowy	Staff Assistant Geologist	Brian Hobbs	Corporate Health & Safety Manager
Tally Sodre	OHSM		

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☐ LIFE VEST ☒ HARD HAT ☐ LIFELINE / BODY HARNESS ☒ SAFETY GLASSES	☐ GOGGLES ☐ FACE SHIELD ☒ HEARING PROTECTION (as needed) ☒ SAFETY SHOES: Steel Toe or composite toe	☐ AIR PURIFYING RESPIRATOR ☐ SUPPLIED RESPIRATOR ☒ PPE CLOTHING: Fluorescent reflective vest of high-visibility clothing; long sleeve shirt; long pants	☒ GLOVES: Leather, nitrile, and cut resistant (as needed) ☐ OTHER

REQUIRED AND / OR RECOMMENDED EQUIPMENT

Required Equipment: Varies

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Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
1. Mobilize/demobilize and establish 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 vehicle. 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. 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. 1a. Wear boots with adequate treads. 1a. Delineate unsafe areas with 42” 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. 1b. When first arriving onsite, park vehicles in designated parking space and/or out of the way locations. Use parking brake on all vehicles and tire chocks on work trucks and trailers. 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. 1b. Identify potential traffic sources. 1b. Wear PPE including high visibility clothing or reflective vest. 1b. Use a spotter while moving work vehicles; plan ahead to avoid backing whenever possible. 1b. Maintain a minimum exclusion zone when vehicles are in motion (i.e. greater than swing/tip radius of equipment). When backing up 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. 1b. Delineate work area with 42” cones, flags, caution tape, and/or other barriers. 1b. Position “Work Area” signs at Site entrances, if possible, or at either side of work area.

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Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
		1b. Position largest vehicle to protect against oncoming traffic. 1b. Face traffic, maintain eye contact with oncoming vehicles, use a spotter, and establish a safe exit route. 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 line-of-fire of vehicle and/or equipment.	1c. Make sure driver has engaged parking brake and placed wheel chocks in a position to prevent movement. Be sure that vehicle is parked in front/down gradient (positioned to best block oncoming traffic) of work area. 1c. Wear leather gloves when handling any tools or equipment. Wear cut-resistant gloves (Kevlar or similar) when handling sharp objects/cutting tools/glass. 1c. Keep body parts away from line-of-fire of equipment. 1c. Always carry tools by the handles and/or designated carrier. Ensure sharp-edged tools are sheathed/secure. 1c. Remove any loose jewelry. Avoid wearing loose clothing and/or ensure loose clothing is secure. 1c. Secure all items on the equipment, tighten up any items or features that have 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. 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 area to avoid contact with biological hazards (i.e. poisonous plants, stinging insects, ticks, etc.). 1e. Wear long sleeved clothes treated with Permethrin, apply insect repellant containing DEET to exposed skin, and inspect clothes and skin for ticks during and after work. 1e. Apply sunscreen (SPF 15+) if exposure to 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. 1f. Watch for cold stress symptoms (severe shivering, slowing of body movement, weakness, stumbling or inability to walk, collapse). Take breaks in warm areas as needed. 1f. Wear clothing appropriate for weather and temperature conditions (e.g., rain jackets, snow pants, multiple layers). 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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JOB SAFETY ANALYSIS	Ctrl. No. GEN-019	DATE: 7/10/2020	☐NEW ☒REVISED	PAGE 1 of 2

JSA TYPE CATEGORY	WORK TYPE	WORK ACTIVITY (Description)
GENERIC	**Site Recon**	**Site Walk and Inspection**

DEVELOPMENT TEAM	POSITION / TITLE	REVIEWED BY:	POSITION / TITLE
Sara Barrientos	Staff Geologist	Brian Hobbs	Corporate Health and Safety Manager
		Joe Duminuco	Vice President

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☐ LIFE VEST ☒ HARD HAT ☐ LIFELINE / BODY HARNESS ☒ SAFETY GLASSES	☐ GOGGLES ☐ FACE SHIELD ☒ HEARING PROTECTION: ear plugs as necessary ☒ SAFETY SHOES: Steel or composite toed	☐ AIR PURIFYING RESPIRATOR ☐ SUPPLIED RESPIRATOR ☒ PPE CLOTHING: High-visibility vest or high-vis outerwear	☒ GLOVES: Leather/cut-resistant/chemical resistant ☒ 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.

Commitment to Safety – All personnel onsite will actively participate in SPSA performance by verbalizing SPSAs throughout the day.

EXCLUSION ZONE (EZ): Maintain 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 foot print of a structure to be demolished.

SITE SECURITY: Prior to site inspection verify appropriate method to address Site Security concerns as it relates to potential criminal activity, homeless population, and/or isolation concerns. Work with the Project Principal and/or Project Manager to address appropriately.

Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
1. Check in with Site contact.	**1a. CONTACT/EXPOSURE/FALL:** Personal injury caused by lack of site specific hazards.	1a. Inquire about hazards and other activities taking place at the Site. 1a. Inform Site contact of work scope, timeline and location(s). 1a. Discuss emergency evacuation procedures and muster points with 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. 2a. Maintain speed limit as posted on-site. 2a. When possible drive on established roadways. 2a. Yield to all pedestrians. 2a. Use pull-through spots or back into parking spots. 2a. Don high visibility clothing/safety vest. If working at 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. 2b. When possible, use established pathways and walk on stable, secure ground. 2b. Communicate traversing hazards with others.
	2c. OVEREXERTION: Muscle strain while carrying equipment.	2c. When carrying equipment to/from work area, use proper lifting techniques; keep back straight, lift with legs, keep load close to 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.
	2d. EXPOSURE: Biological hazards – ticks; bees/wasps; poison ivy; insects; (Ticks are most active any time the temperature is above freezing, typically from March to November.)	2d. Inspect area to avoid contact with biological hazards. 2d. Ticks: • Treat outer clothing including pants, shirts, socks, boots and hats the evening before with Permethrin (allowing at least two hours before use). • Apply DEET to exposed skin before travelling to the Site and reapply after two hours. • Check for ticks during and after work. 2d. Bees: • Use bee spray as appropriate to deter/eliminate bees. • Protect exposed skin with insect repellent.

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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".

	2e. EXPOSURE: Heat Stress & Cold Stress. Personal injury from working in inclement weather conditions.	2d. Poison Ivy: • Identify areas of poison ivy and spray with weed killer. Don Tyvek and rubber boots while traversing poison ivy areas. • If skin contacts poison ivy, wash skin thoroughly with soap and water. 2e. Wear sunscreen with SPF 15 or greater on exposed skin whenever 30 minutes or more of sun exposure is expected. 2e. Watch for heat stress symptoms (muscle cramping, exhaustion, dizziness, rapid and shallow breathing). Take breaks as needed. 2e. Watch for cold stress symptoms (severe shivering, slowing of body movement, weakness, stumbling or inability to walk, collapse). Take breaks as needed. 2e. Wear appropriate rain gear as needed. 2e. Take frequent breaks if tired, wet, or cold/hot. Drink water. 2e. If lightning is observed, wait 30 minutes after last thunder boom/lightning bolt in a sheltered location (car acceptable) before starting work again.
3. Walking near heavy equipment and machinery.	**3a. CONTACT:** Personal injury from Site and roadway traffic. Personal injury from flying debris **3b. OVEREXERTION:** Personal injury from lifting/moving/rotating equipment. **3c. EXPOSURE:** Hearing damage from noise generating equipment/processes. Inhalation/exposure to hazardous vapors and or dust. **3d. EXPOSURE:** Working in a remote area.	3a. See 2a. 3a. Maintain an exclusion zone of at least 10'-25' feet from all engaged equipment. 3a. Keep body parts out of the line of fire of pinch points. 3a. Wear appropriate PPE always. 3b. See 2c. 3c. Wear hearing protection if >85 dBA. (i.e. noise levels which require you to raise your voice to communicate) 3c. Always wear leather gloves when handling any tools or equipment. 3c. Always wear appropriate PPE based off chemicals present. 3d. Use the "buddy system" whenever possible. If working alone, contact PM upon arrival/departure, as well as during work activities prior to commencing work if applicable. 3d. Always carry a communication (i.e., cell phone, walkie-talkie) or directional (i.e., map, compass, etc.) device when traversing remote areas.
4. Working in adverse weather conditions.	**4a. EXPOSURE:** 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. 4a. Watch for cold stress symptoms (severe shivering, slowing of body movement, weakness, stumbling or inability to walk, collapse). Take breaks as needed. 4a. Wear appropriate rain gear as needed. 4a. Take frequent breaks if tired, wet, or cold/hot. Drink water. 4a. If lightning is observed, wait 30 minutes after last thunder boom/lightning bolt in a sheltered location (car acceptable) before starting work again.
5. Departing Site.	**5a. EXPOSURE:** Exposure to unnecessary hazards should personnel believe Roux is on-Site during an emergency and conduct a search.	5a. Sign out or notify Site contact and Roux Project Manager of your departure.

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JSA TYPE CATEGORY: **GENERIC**	WORK TYPE: **Gauging & Sampling**	WORK ACTIVITY (Description): **Soil Sampling**	
DEVELOPMENT TEAM	**POSITION / TITLE**	**REVIEWED BY:**	**POSITION / TITLE**
MaryBeth Lyons	Project Scientist	Brian Hobbs	Corporate Health and Safety Manager

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☐ LIFE VEST ☒ HARD HAT ☐ LIFELINE / BODY HARNESS ☒ SAFETY GLASSES ☒ FLAME RESISTANT CLOTHING (as needed)	☐ GOGGLES ☐ FACE SHIELD: ☒ HEARING PROTECTION: (as needed) ☒ SAFETY SHOES: Composite-toe or steel toe boots	☐ AIR PURIFYING RESPIRATOR ☐ SUPPLIED RESPIRATOR ☒ PPE CLOTHING: Fluorescent reflective vest or high visibility clothing	☒ GLOVES: Leather, Nitrile and cut resistant ☒ OTHER: Insect repellant, sunscreen (as needed)

REQUIRED AND / OR RECOMMENDED EQUIPMENT

Recommended Equipment: 42" traffic cones, caution tape, trowel

COMMITMENT TO SAFETY- All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing SPSAs.

EXCLUSION ZONE (EZ): Maintain 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 foot print of a structure to be demolished.

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 42" traffic cones and/or caution tape to prevent exposure to traffic and inform others of work activity. 1a. Wear reflective vest and/or high visibility clothing. 1a. Face the direction of any vehicular traffic. Position vehicle to protect worker from traffic. 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. 1b. Use established pathways and walk on stable, secure ground. 1b. Stage equipment and tools in a convenient, stable, and orderly manner. Store equipment at lowest potential energy. 1b. Roux employees should stay 5 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: Exposure to sun and excessive heat, possibly causing sunburn, heat exhaustion or heat stroke. Exposure to cold temperatures possibly causing cold stress. Skin burn as a result of fire, if applicable. Exposure to explosive vapors due to tank farm operations. Exposure to airborne dust due to high wind speeds. 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. 1c. Use a tent to shade the work area from direct sunlight particularly when warm temperatures are expected. 1c. Be aware of the location of all Site personnel. 1c. Watch for heat stress symptoms (muscle cramping, exhaustion, dizziness, rapid and shallow breathing). 1c. Watch for cold stress symptoms (severe shivering, slowing of body movement, weakness, stumbling or inability to walk, collapse). 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.). 1c. No open flames/heat sources. 1c. Flame retardant clothing must be worn when specified by Site policy. 1c. Cell phones should be disabled when specified by Site policy. 1c. Pre-treat field clothing with Permethrin prior to site visit to kill ticks and insects. 1c. Wear long sleeved shirts and tuck in (or tape) pant legs into socks or boots to prevent ticks from reaching skin. 1c. Spray insect repellant containing DEET on exposed skin when working in overgrown areas of the Site. 1c. Inspect area to avoid contact with biological hazards. 1c. Wear cut-resistant gloves when handling branches, shrubs, etc. that may lie within the walking path. 1c. Wear spoggles if the average wind speeds are above 15 mph. 1c. Personnel shall examine themselves and co-worker's outer clothing for ticks periodically when onsite. 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 [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
2. Collect Soil Sample	**2a. CONTACT:** Personal injury from pinch points, cuts, and abrasions from sampling equipment tools, and material within soil sample. Personal injury from contact with moving equipment while sampling. 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. 2a. Where possible, use trowel or equivalent tool to avoid contact with soil. 2a. If sampling from bucket of heavy equipment, ensure all equipment is off and operator utilizes the “show me your hands” policy. 2a. See 1a.
	2b. EXPOSURE: 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. 2b. Wear safety glasses to protect eyes from dust or air-borne contaminants that may results from disturbing the soil. 2b. Where possible, remain upgradient from sample location if collecting soil sample from stockpile, drill rig, etc. to avoid breathing contaminant vapors, if they are present. 2b. When collecting soil sample from hand auger, put large zip lock bag over entire auger to prevent spillage of soil on to the ground. 2b. Open sample jars slowly and fill carefully to avoid contact with preservatives.
	2c. EXERTION: 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:** Contamination (e.g., Separate Phase Hydrocarbons (SPH), contaminated vapors and/or soil).	3a. Wear chemical-resistant (nitrile) disposable gloves and safety glasses. 3a. Use an absorbent pad to clean spills. 3a. Properly dispose of used materials/PPE in provided drums in designated drum storage area. 3a. Remain upwind of sample and avoid breathing contaminant vapors, if they are present.
	3b. EXPOSURE: Chemicals in cleaning solution including ammonia.	3b. Wear chemical-resistant (nitrile) disposable gloves and safety glasses. 3b. Work on the upwind side of decontamination area. 3b. Use an absorbent pad to clean spills. 3b. Properly dispose of used materials/PPE in provided drums in designated drum storage area. Ensure that all drums are properly labeled and secured.

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JOB SAFETY ANALYSIS	Ctrl. No. GEN-021	DATE: 7/10/2020	☐ NEW ☒ REVISED	PAGE 1 of 2

JSA TYPE CATEGORY:	WORK TYPE	WORK ACTIVITY (Description)
GENERIC	**Gauging and Sampling**	**Soil Vapor Sampling (Permanent Monitoring Points)**

DEVELOPMENT TEAM	POSITION / TITLE	REVIEWED BY:	POSITION / TITLE
Jeff Wills	Senior Hydrogeologist	Brian Hobbs	Corporate Health and Safety Manager

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☐ LIFE VEST	☐ GOGGLES	☐ AIR PURIFYING RESPIRATOR	☒ GLOVES: Cut-resistant & Nitriles
☒ HARD HAT	☐ FACE SHIELD	☐ SUPPLIED RESPIRATOR	☒ OTHER: Bug Spray, Sun Screen, Knee Pads or kneeling pad
☐ LIFELINE / BODY HARNESS	☐ HEARING PROTECTION	☒ PPE CLOTHING: Fluorescent reflective vest or high visibility clothing	
☒ SAFETY GLASSES	☒ SAFETY SHOES: Steel-toe boots		

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, 42-inch 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 SPSAs.

Work Zone (WZ): A 5-foot exclusion zone will be maintained for non-essential personnel.

Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
1. Define and secure work area.	**1a. FALL:** Potential tripping hazards.	1a. Ensure work area is secure and inform others (third party) of work activity. 1a. Remove tripping hazards and inspect 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 work vehicle to protect employees. 1b. Delineate work area (including vehicles) with traffic safety cones and caution tape or retractable cone bars. 1b. Maintain a 5-foot work zone. 1b. Wear high visibility clothing or reflective safety vest.
	1c. EXERTION: Muscle strain while lifting and carrying equipment.	1c. When carrying equipment to/from work area, keep back straight, lift with legs, keep load close to body, never reach with a load. Ensure that loads are balanced. Use mechanical assistance/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. 2a. Use hand tools with extensions to remove and replace well covers. 2a. Wear cut-resistant gloves. 2a. Use knee pads or kneeling pad when repetitive kneeling on rough ground is anticipated.
	2b. FALL: Potential tripping hazards associated with installing bolts.	2b. Place security bolts in secure location so not to create tripping hazards. Replace security bolts so that they fit flush with monitoring well covers.
	2c. EXERTION: Physical exertion to remove bolts that were over torqued or stripped.	2c. Replace any security bolts that show signs of stripping. Do not over tighten. 2c. Use body positioning and bending techniques that minimize muscle strain; keep back straight, bend at the knees. 2c. See 2a.

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Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
3. Screen vapor point with PID.	**3a. FALL:** Potential tripping hazards associated with equipment. **3b. EXPOSURE:** Inhalation of soil vapor	3a. Place equipment in one area close to the sampling location. 3b. Identify area where equipment is to be stored within the work area (away from main walking path). 3a. Don't leave equipment on the ground. Return equipment to storage area between uses. 3b. Replace brass caps immediately upon completion to avoid soil vapors migrating to the surface through sample tubing. 3b. Stand upwind of sample point during screening activities.
4. Remove / replace brass caps at the end of the sam`ple tubing.	**4a. CONTACT:** Pinch points associated with hand tools and brass caps. **4b. EXPOSURE:** Potential pathway for vapors to migrate to land surface.	4a. Use wrench to remove and replace brass caps. 4a. Wear cut-resistant gloves to protect against pinch points and scrapes. 4b. See 3b. 4b. Stand up wind of sample point location.
5. Set up soil vapor sampling equipment and calibration of meters.	**5a. FALL:** Potential tripping hazards associated with equipment and tubing.5b. **5b. CONTACT:** Pinch points associated with handling equipment. **5c. EXPOSURE:** Inhalation of calibration gas and helium.	5a. See 3a. 5a. Keep tubing slack to a minimum and locate the summa canister as close to the sampling location as possible. 5a. Avoid stepping over equipment and tubing. 5b. Do not place fingers/hands under sampling equipment. 5b. Make multiple trips when unloading equipment in work area. 5b. Wear cut-resistant gloves to protect against pinch points while handling sampling equipment. 5c. Review SDS for each type of calibration gas used before calibrating. 5c. Calibrate meters in a well-ventilated area and keep air flow regulator away from face. 5c. Close valve on canisters after use to avoid inhalation of excess helium or calibration gas. 5c. Stand up wind of bucket during helium tracer gas test.
6. Cleaning Work Area.	**6a. FALL:** Potential tripping hazards associated with equipment and tubing. **6b. CONTACT:** Storing and transport of equipment in car.	6a. See 3a. 6a. See 3b. 6b. Ensure that equipment is placed securely in the vehicle. Do not stack equipment on top of each other. Secure equipment so that it will not slide while being transported. 6b. Wear cut-resistant gloves while handling/loading equipment.

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JOB SAFETY ANALYSIS	Ctrl. No. GEN-022	DATE 7/10/2020	☐ NEW ☒ REVISED	PAGE 1 of 2

JSA TYPE CATEGORY:	WORK TYPE:	WORK ACTIVITY (Description):	
General	**Drilling**	**Rotosonic Soil Borings / Well Installation**	
DEVELOPMENT TEAM	**POSITION / TITLE**	**REVIEWED BY:**	**POSITION / TITLE**
William Poupis	Vice President ADT	Brian Hobbs	Corporate Health & Safety Manager
Ron Lombino	Project Hydrogeologist		

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☐ LIFE VEST ☒ HARD HAT ☐ LIFELINE / BODY HARNESS ☒ SAFETY GLASSES	☐ GOGGLES ☐ FACE SHIELD ☒ HEARING PROTECTION: (as needed) ☒ SAFETY SHOES steel or composite toe	☐ AIR PURIFYING RESPIRATOR ☐ SUPPLIED RESPIRATOR ☒ PPE CLOTHING: fluorescent long sleeve shirt or long sleeve shirt and reflective safety vest.	☒ GLOVES: Leather, Nitrile and cut resistant ☒ 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, 42" 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 SPSAs

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"SHOW ME YOUR HANDS"
Driller and helper should show that hands are clear from 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). **See also Mobilization/Demobilization JSA.**	**1a. CONTACT/CRUSH:** Equipment/property damage during movement. Crush point between moving rig and other objects.	1a. The drill rig's tower/derrick will be lowered and secured prior to mobilization. 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. 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. 1a. When backing up 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. 1a. Inspect the driving path for uneven terrain. Level or avoid if needed. 1a. Drill rig should have a **minimum exclusion zone (i.e. greater than swing/tip radius of 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. 1b. Do not climb over stored materials/equipment; walk around. Practice good housekeeping. 1b. Use established pathways and walk on stable, secure ground.
2. Raising tower/derrick of 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. 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. 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 when raising the rig and instability of rig.	2b. Inspect the equipment prior to use and avoid amputation points when engaging tower/derrick. 2b. Lower outriggers to ensure stability prior to raising rig tower/derrick. 2b. If the rig needs to be mounted, be sure to use three points of contact.

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Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
3. Advancement of soil boring and well installation. **See also Soil Sampling JSA.**	**3a. CONTACT**: Flying debris.	3a. Be aware of and avoid potential lines of fire 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. 3b. Stand upwind and keep body away from rig. 3b. Dust mask should be worn if there is visible dust in the breathing zone. 3b. Wear hearing protection when the drill rig is in operation.
	3c. CAUGHT: Limb/extremity amputation points; abrasion/crushing.	3c. Always wear leather gloves when making connections and using hand tools; wear cut-resistant (i.e., Kevlar) gloves when handling cutting tools. 3c. Inspect the equipment prior to use for potential amputation points. Keep hands away from being between connections and use of tools is preferable compared to fingers and hands. 3c. Inspect drill head for worn surface or missing teeth; replace if damaged or blunt. 3c. Ensure all jewelry is removed, loose clothing is secured, and PPE is secured close to the body. 3c. All non-essential personnel should remain outside the heavy equipment exclusion zone that is at least as far as the boom is high (**minimum exclusion zone based on swing/tip radius**). 3c. Drillers and helpers will understand and use the “Show Me Your Hands” Policy. Operator and helpers will remain a **minimum of 3 feet** from augers/drill rods while in operation.
	3d. CONTACT/CRUSH: Crushed between equipment due to imbalance during advancement of drill equipment.	3d. Drillers will advance the borehole with caution to avoid causing the rig to become imbalanced and/or tip. 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. 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 periodically monitor the breathing zone of the work area. 3e. If readings 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.
	3f. FALL: Slip/trip/fall hazards.	3f. Contain drill cuttings and drilling water to prevent slip/trip/fall hazards from developing in work area. 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. 3g. Utilize team lifting for objects over 50lbs. 3g. Use mechanical lifting device for odd shaped objects.
4. Decontaminate equipment.	**4a. EXPOSURE:** Contamination (e.g., Separate Phase Hydrocarbons (SPH), contaminated groundwater, vapors, chemical in cleaning supplies).	4a. Wear chemical-resistant gloves and safety glasses. 4a. Contain decontamination water so that it does not spill. 4a. Use an absorbent pad to clean spills, if necessary. 4a. See 3b.

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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-023	DATE: 7/10/2020	☐ NEW ☒ REVISED	PAGE 1 of 2

JSA TYPE CATEGORY	WORK TYPE	WORK ACTIVITY (Description)	
Generic	**Construction**	**Spotting Heavy Machinery**	
DEVELOPMENT TEAM	**POSITION / TITLE**	**REVIEWED BY:**	**POSITION / TITLE**
Levi Curnutte	Project Scientist	Brian Hobbs	Corporate Health & Safety Manager

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☐ LIFE VEST	☐ GOGGLES	☐ Particulate Respirator	☒ GLOVES: Cut resistant / leather
☒ HARD HAT	☐ FACE SHIELD	☐ SUPPLIED RESPIRATOR	☐ OTHER:
☒ LONG SLEEVED SHIRT	☐ HEARING PROTECTION	☒ PPE CLOTHING: Fluorescent reflective clothing	
☐ LIFELINE / BODY HARNESS	☒ SAFETY SHOES: Steel-/Composite-toe boots/shoes		
☒ SAFETY GLASSES			

REQUIRED AND / OR RECOMMENDED EQUIPMENT

Heavy Machinery (i.e. excavator, payloader, truck, forklift, etc.)

COMMITMENT TO SAFETY- All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing SPSAs

EXCLUSION ZONE (EZ): Maintain 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 foot print of a structure to be demolished.

Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
1. Prepare for machine activity.	**1a. CONTACT:** Obstructions in the work area may create contact hazards from machinery. **1b. Fall :** Slip/Trip/Fall	1a. Cordon off the work area with safety barrels/cones and a rigid barrier (snow fence, traffic bar, etc.). Communicate that only necessary personnel should be in the work area. Spotter and equipment operator shall enforce the **EZ.** Operator will not operate but shall remain in the hands-off mode while personnel are within the exclusion zone. 1b. Ensure that work area is flat, level and clear of any obstructions or debris before setting up 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 Exclusion Zone. Maintain Exclusion Zone. The Exclusion Zone shall be delineated by using 42-inch traffic cones/barrels and a fixed rigid barrier. 2a. The Minimum Heavy Equipment Exclusion zone is greater than the swing/tip radius of equipment. 2a. Both the spotter and equipment operators shall have 2-way radios/cellular devices on their persons to ensure audible communication in the event any changes or new hazards may arise. 2a. All workers should stay outside of the Exclusion Zone of all equipment unless 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 Exclusion Zone must be reduced due to work area restrictions then the spotter and operator shall enforce the reduced Exclusion Zone. 2a. Spotters must make eye contact with the machine operator or all movement ceases until visual contact can be reestablished. 2a. Spotter shall keep an eye out for any issues with the machine the operator may not see and communicate with other work crews and spotters on behalf of the operator. 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.** 2a. Wear fluorescent clothing/safety vest.

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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 [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
	2b. FALL: Slip/Trip/Fall	2b. Look where walking to identify and avoid slip/trip/fall hazards. Avoid icy and/or wet surfaces. Remove obstacles if possible. 2b. Use designated walkways during spotting whenever possible.
	2c. CAUGHT: 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 operator at all times while being aware of surrounding structures.
	2d. EXPOSURE: 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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[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	Cntrl. No. GEN-027	DATE: 11/3/2020	☒ NEW ☐ REVISED	PAGE 1 of 2

JSA TYPE CATEGORY	WORK TYPE	WORK ACTIVITY (Description)
GENERIC	**Drilling**	**Pre-Drilling Clearance, Vactron and Air Knife**

DEVELOPMENT TEAM	POSITION / TITLE	REVIEWED BY:	POSITION / TITLE
Courtney Rempfer	Staff Scientist	Joseph Midwig	Office Health & Safety Manager
Sara Redding	Senior Hydrogeologist	Brian Hobbs	Corporate Health & Safety Manager

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☐ LIFE VEST ☒ HARD HAT ☐ LIFELINE / BODY HARNESS ☒ SAFETY GLASSES	☐ GOGGLES ☒ FACE SHIELD (While Air Knifing) ☒ HEARING PROTECTION (As needed) ☒ SAFETY SHOES: Composite toe or steel toe boots	☐ AIR PURIFYING RESPIRATOR ☐ SUPPLIED RESPIRATOR ☒ PPE CLOTHING: Fluorescent reflective vest or high visibility clothing; long-sleeve shirt	☒ GLOVES: Leather, Nitrile, cut-resistant ☒ OTHER: Dusk mask, insect repellant, sunscreen (as needed)

REQUIRED AND / OR RECOMMENDED EQUIPMENT

Vac-Truck or Vac Drum, Compressor, Jack Hammer, Air Knife. Circular Saw, Hand Tools, Dust Mask, Photoionization Detector, Multi Gas Meter, 42 inch safety cones and flags, Retractable Cone Bars, Caution Tape, 20 lb. Fire Extinguisher, "Work Area" Signs, Pressurized Water Sprayer

Commitment to Safety – All personnel onsite will actively participate in hazard recognition and mitigation throughout the day by verbalizing SPSAs

EXCLUSION ZONE: All non-essential personnel will maintain a distance of 10 feet from drilling equipment while equipment is moving/engaged

Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
1. Verify pre-clearance protocol	**1a. CONTACT:** Underground utility damage; property damage; personal injury	1a. Confirm that (if applicable) "Call Before You Dig" and local utility companies were contacted prior to starting work in order to confirm utility mark outs. Must have a case # before digging. 1a Walk the Site to evaluate utility markings and review maps (see Site Walk Inspection JSA). Utilities are not always properly marked out ensure use of observational skills through the pre-clearing checklist. 1a. Review pre-clearing checklist fromm and sub-surface clearance form. Pre-clearing protocol indicates that clearance must be conducted to a minimum of 5 vertical feet below ground surface or 10 vertical feet below ground surface in the critical zone using hand tools.
	1b. ENERGY SOURCE/CONTACT: Property damage; Pressurized water mains may cause lacerations or broken bones. Pressurized gas mains may explode causing serious injury, or death. Underground electric may cause severe burns, shock, or death.	1b. Pre-clearing of each soil boring/monitoring well location must be conducted to a minimum of 5 vertical feet below the ground surface (10 feet minimum for Critical Zone) using hand tools (shovel and non-metallic dig bar and hand auger) prior to drilling. Supervisor should be contacted to discuss appropriate pre-clearing depth. 1b. MUST Complete subsurface clearance checklist prior to pre-clearance.
	1c. FALL: Slip, Trip or Fall may cause muscle strains or tears, abrasions, lacerations, or broken bones.	1c. Be aware of the conditions when walking or loading equipment and working. Walk within established pathway avoiding uneven surfaces. Remove potential slip/trip/fall hazards.
2. Mobilize/demobilize and establish work area	**2a. SEE MOBILIZATION / DEMOBILIZATION JSA**	2a. See Mobilization / Demobilization JSA
3. Concrete saw cutting, jack hammer and hand clearance with hand tools, air knife	**3a. CONTACT:** Flying debris striking face or body	3a Maintain 10' minimum exclusion zone. Use the required PPE (i.e., leather/cut proof gloves, safety glasses/face shield). 3a. Use anti-whip devices on compressor hoses. 3a Wear a face shield to protect face from flying debris when using air knife. 3a. Utilize a traffic cone or physical barriers over the hole during air knife activities to keep flying debris close to ground.
	3b. EXPOSURE: Inhalation/exposure to hazardous vapors and/or concrete dust, noise exposure	3b. Monitor breathing zone with a calibrated PID and/or multi-gas meter. If meters sustain readings greater than recommeneded in the HASP for the specific contaminant of concerns (COCs) the Roux field personnel must temporarily cease work, instruct all Site personnel to step away from the area of elevated readings. Contact PM. 3b. Wet concrete while using saw to minimize dust and wear dust mask to prevent inhalation. 3b. Stand upwind and keep body behind saw. Observers and helpers should avoid line of fire for saw blade. Always cut away from body. 3b. No open flames/heat sources. 3b. Wear hearing protection when saw, jackhammer or air compressor are in operation. Otherwise, if sound levels exceed 85 dbA, put on hearing protection.
	3c. ENERGY SOURCE/CONTACT: Property damage; Pressurized water	

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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."

	mains may cause lacerations or broken bones. Pressurized gas mains may explode causing serious injury, or death. Underground electric may cause severe burns, shock, or death.	3c. For air knifing, ensure extention/lance tip reaches the full 5 feet bgs. Air knife should be advanced **AT A MINIMUM** in all four corners of the expected boring location to find any possible arrangement of utilities. 3c. Ensure diameter of soil preclearance hole is at a **MINIMUM 2x** the diameter of any drilling or hard dig equipment that will be entering the hole. 3c. See Complete subsurface clearance protocol for information provided above.
	3d.ERGONOMICS/EXERTION: Muscle strain due to poor body positioning when handling equipment and materials	3d. Use body positioning and lifting techniques that minimizes muscle strain; keep back straight, lift with legs, keep load close to body, and never reach with a load. 3d. Ensure that loads are balanced to reduce the potential for muscle strain. 3d. Two people or a mechanical lifting aid are required when lifting objects over 50 lbs. or when the shape makes the object difficult to lift.
	3e. FALL: Tripping/falling due to uneven terrain, weather conditions, and materials/equipment stored at the Site	3e. Inspect walking path for uneven terrain, weather-related hazards (i.e., ice, puddles, snow, etc.), and obstructions prior to mobilizing equipment. Mob/Demob JSA. 3e. Do not climb over stored materials/equipment; walk around. Practice good housekeeping. 3e. Use established pathways and walk on stable, secure ground. 3e. 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). 3e. Equipment and tools that are not anticipated to be used will be returned to a storage area that is out of the immediate work area. 3e. Ensure power cords and compressed air lines are grouped when used within the work area. 3e. Pre-cleared location will be finished flush to grade as to prevent a slip/trip hazard or coned and taped off.
	3f. CAUGHT: Amputation points associated with the equipment and vacuum hose	3f. Always wear leather gloves when making connections and using hand tools; wear cut-resistant (i.e., Kevlar) gloves when handling cutting tools. 3f. Inspect the equipment prior to use for potential pinch points. 3f. Test all emergency shutdown devices prior to using equipment. 3f. Inspect saw blade for worn surface or missing teeth; switch blade if damaged or blunt. 3f. Ensure all jewelry is removed, loose clothing is secured, and PPE is secured close to the body. 3f. All non-essential personnel shall maintain a 10 foot exclusion zone; position body out of the line-of-fire of equipment. 3f. Drillers and helpers will understand and use the “Show Me Your Hands Policy”.
4. Move drum to staging area using drum cart	**4a. EXPOSURE/CONTACT:** Contamination (e.g., Separate Phase Hydrocarbons (SPH), contaminated groundwater, soil)	4a. Wear Nitrile chemical-resistant gloves under leather or cut proof gloves. 4a. Do not overfill drums. Ensure that the drum lids are attached securely. 4a. All drums will be staged in the designated storage area.
	4b. EXERTION: Muscle strain while maneuvering drums with drum cart/lift gate	4b. See 3d. Do not overfill drums. Use lift gate on back of truck to load and unload drums. Use drum dolly to move drum.
	4c. CAUGHT: Pinch points associated with handling drum lid	4c. Ensure that fingers are not placed under the lid of the drum. Wear leather gloves or cut proof gloves. Use appropriate ratchet while sealing drum lid.
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. 5a. Contain decontamination water so that it does not spill. 5a. Use an absorbent pad to clean spills, if necessary. 5a. Spray equipment from side angle, not straight on, to avoid backsplash. 5a. See 3b.
	5b. EXPOSURE: To chemicals in cleaning solution.	5b. See 4a. Review SDS to ensure appropriate precautions are taken and understood.

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JOB SAFETY ANALYSIS	Ctrl. No. GEN-029	DATE: 8/18/2021	☐ NEW ☒ REVISED	PAGE 1 of 2

JSA TYPE CATEGORY	WORK TYPE	WORK ACTIVITY (Description)	
GENERIC	**Preclearing/Sampling**	**Hand Augering**	
DEVELOPMENT TEAM	**POSITION / TITLE**	**REVIEWED BY:**	**POSITION / TITLE**
Sean Owens	Senior Health & Safety Specialist	Brian Hobbs	Corporate Health & Safety Director

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☐ LIFE VEST	☐ GOGGLES	☐ AIR PURIFYING RESPIRATOR	☒ GLOVES: Cut Resistant / Leather / Nitrile / Chemical resistant
☒ HARD HAT	☐ FACE SHIELD	☐ SUPPLIED RESPIRATOR	☐ OTHER
☐ LIFELINE / BODY HARNESS	☐ HEARING PROTECTION	☒ PPE CLOTHING: Long sleeve high visibility clothing	
☒ SAFETY GLASSES	☒ SAFETY SHOES: Steel or composite toed		

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 SPSAs.

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. 1a. All equipment must be stowed and secured prior to moving. 1a. Drive on established roadways. 1a. Do not back up vehicle without 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.1c.
	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. 1b. Do not climb over stored materials/equipment; walk around. Use established pathways and walk on stable, secure ground. 1b. Use established ramp when descending into/ascending from impoundment areas. 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 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 work area, use proper lifting techniques; keep back straight, lift with legs, keep load close to 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. Wear sunscreen with an SPF of at least 15 whenever 30 minutes or more of exposure is expected.
	1d. EXPOSURE: Exposure to sun, possibly causing sunburn Biological hazards - bees/wasps, poison oak, thorns, insects, etc.	1d. Inspect area to avoid contact with biological hazards. 1d. Wear cut resistant gloves when handling branches, shrubs, etc. that may lie within the walking path. 1d. Avoid any areas onsite that have poison oak.
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. 2a. Wear reflective vest and/or fluorescent clothing. 2a. Face the direction of vehicular traffic. Position vehicle to protect worker from traffic. 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. 2b. Equipment and tools will be staged in a convenient, stable, and orderly manner. 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). 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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Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [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" and local utility companies were contacted prior to hand augering. 3a. Walk the Site to evaluate utility markings and review maps.
4. Augering/advancing borehole	**4a. EXPOSURE:** Contaminated soil/water/vapor **4b. EXERTION:** Muscle strain from lifting, bending, repetitive motion. **4c. CAUGHT:** Personal injury as a result of jewelry/loose clothing caught on equipment, well covers, machinery, hand auger, pry bar etc.. **4d. CONTACT/CAUGHT:** Pinch points, abrasions	4a. Monitor breathing zone with a PID when VOCs area concern. If vapors sustain > 5 ppm, upgrade PPE as per HASP. 4a. Wear chemical-resistant disposable gloves and safety glasses when handling impacted materials. 4a. Place excavated soil on plastic sheeting and store soil waste in designated area. 4a. Work on the upwind side of the boring. 4b. Body positioning and rotating with the auger to reduce strain. Don't twist back. 4c. No form of jewelry should be worn while on-site. 4c. Clothing must be appropriately sized so it is not loose fitting. 4d. Keep head and upper body clear when lifting hand auger, pry bar/post-hole digger. Ensure to not be overly aggressive when using pry bar. 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 handling any preservatives; use containment material or plastic sheeting to protect surrounding areas. 5a. When collecting soil sample from hand auger, put large zip lock bag over entire auger to prevent spillage of soil on to the ground. 5a. Open sample jars slowly and fill carefully to avoid contact with preservatives.
6. Decontaminate equipment	**6a. EXPOSURE:** Contact with contamination (impacted soil and/or lab preservatives, decontamination solution) **6b. CONTACT/CAUGHT:** pinch points and cuts/abrasions	6a. Wear chemical-resistant disposable gloves and safety glasses. 6a. Use an absorbent pad to clean spills. 6a. Properly dispose of used materials/PPE trash bags. 6b. Keep fingers/hands out of pinch points when dis-assembling hand auger during decontamination.

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APPENDIX B

Safety Data Sheets (SDSs) for Chemicals Used

Five Part Reactive Gas Mixture:
Hydrogen Sulfide / Carbon Monoxide / Methane / Oxygen / Nitrogen

1. Chemical Product and Company Identification

SpecAir Specialty Gases, Chemicals & Technology
22 Albiston Way
Auburn, ME
04210

TELEPHONE NUMBER: 800-292-6218
FAX NUMBER: 207-777-6215
E-MAIL: Info@SpecAir.com

24-HOUR EMERGENCY NUMBER: 1-800-535-5053

PRODUCT NAME: Five Part Reactive Gas Mixture
CHEMICAL NAME: Methane, Carbon Monoxide, Hydrogen Sulfide, Oxygen in Nitrogen
COMMON NAMES/ SYNONYMS: None
TDG (Canada) CLASSIFICATION: 2.2
WHIMIS CLASSIFICATION: A

2. Composition / Information on Ingredients

	% VOLUME	PEL-OSHA2	TLV-ACGIH3	LD50 OR LC50 Route/Species
Hydrogen Sulfide Formula: H2S CAS: 7783-06-4 RTECS#: MX1225000	0.0005% to 0.01% 5 PPM to 100 PPM	20 PPM Ceiling Concentration	10 PPM TWA 15 PPM STEL	LC50 712 PPM Inhalation/Rat 1 Hr.
Methane Formula: CH4 CAS: 0074-82-8 RTECS#: TX2275000	0.0001% to 2.5%	None Established	None Established Simple Asphyxiant	Not Available
Carbon Monoxide Formula: CO CAS: 0630-08-0 RTECS#: FG3500000	0.0001% to 0.1%	TWA 50 PPM	25 PPM TWA	LC50 1807 PPM / 4 Hr. (Rat)
Oxygen Formula: O2 CAS: 7782-44-7 RTECS#: RS206000	2.0% to 23%	None	None	Not Available
Nitrogen Formula: N2 CAS: 7727-37-9 RTECS#: QW9700000	74.39% to 98.0%	None Established	None Established Simple Asphyxiant	Not Available

3. HAZARDS IDENTIFICATION

EMERGENCY OVERVIEW

This product is a colorless gas, which has a rotten-egg odor. The odor cannot be relied on as an adequate warning of the presence of this product, because olfactory fatigue occurs after over-exposure to hydrogen sulfide. Hydrogen sulfide and carbon monoxide are toxic to humans in relatively low concentrations. Over-exposure can cause skin or eye irritation, nausea, dizziness, headaches, collapse, unconsciousness, coma, and death.

ROUTE OF ENTRY:

Skin Contact	Skin Absorption	Eye Contact	Inhalation	Ingestion
Yes	No	Yes	Yes	No

CARCINOGENICITY:

NTP: No

IARC: No

OSHA: No

EYE EFFECTS:
Hydrogen sulfide can cause eyes to become scratchy, irritated and even teary. Above 50 ppm of hydrogen sulfide, there is an intense tearing blurring of vision, and pain when looking at light.

SKIN EFFECTS:
Over-exposure to carbon monoxide can be indicated by the lips and fingernails turning bright red. High concentrations of hydrogen sulfide may also be irritating to the skin.

INGESTION EFFECTS:
Ingestion unlikely. Gas at room temperature.

INHALATION EFFECTS:
Due to the small size of this cylinder, no unusual heath effects from over-exposure are anticipated under routine circumstances of use. Over-exposure to hydrogen sulfide can cause dizziness, headache, and nausea. At 12- 16% Oxygen, breathing and pulse rate is increased, muscular coordination is slightly disturbed.

NFPA Hazard Codes

Health:	4
Flammability:	0
Reactivity:	0

HMIS Hazard Codes

Health:	4
Flammability:	0
Reactivity:	0

Rating System

0=	No Hazard
1=	Slight Hazard
2=	Moderate Hazard
3=	Serious Hazard
4=	Severe Hazard

4. FIRST AID MEASURES

EYES:
PERSONS WITH POTENTIAL EXPOSURE SHOULD NOT WEAR CONTACT LENSES. Flush contaminated eyes with copious quantities of water. Part eyelids to assure complete flushing. Continue for a minimum of 15 minutes. Seek immediate medical attention.

SKIN:
Remove contaminated clothing as rapidly as possible. Flush affected area with copious quantities of water. Seek immediate medical attention.

INGESTION:
Contact Poison Control. Seek Medical Attention

INHALATION:
PROMPT MEDICAL ATTENTION IS MANDATORY IN ALL CASED OF OVEREXPOSURE. RESCUE PERSONNEL SHOULD BE EQUIPPED THE SELF-CONTAINED BREATHING APPARATUS. Victims should be assisted to an uncontaminated area and inhale fresh air. Quick removal from the contaminated area is most important. If breathing has stopped administer artificial resuscitation and supplemental oxygen. Further treatment should be symptomatic and supportive.

5. FIRE-FIGHTING MEASURES

These containers hold gas under pressure, with no liquid phase. If involved in a major fire, they should be sprayed with water to avoid pressure increases, otherwise pressures will rise and ultimately they may distort or burst to release the contents. The gases will not add significantly to the fire, but containers or fragments may be projected considerable distances - thereby hampering firefighting efforts.

6. ACCIDENTAL RELEASE MEASURES

In terms of weight, these containers hold very little contents, such that any accidental release by puncturing etc. will be of no practical concern.

7. HANDLING AND STORAGE

Suck back of water into the container must be prevented. Do not allow back feed into the container. Use only properly specified equipment which is suitable for this product, its supply pressure and temperature. Use only in well-ventilated areas. Do not heat cylinder by any means to increase rate of product from the cylinder. Do not allow the temperature where cylinders are stored to exceed 130°F (54°C).

8. EXPOSURE CONTROLS/PERSONAL PROTECTION

Use adequate ventilation for extended use of gas.

9. PHYSICAL AND CHEMICAL PROPERTIES

PARAMETER:	VALUE:
Physical state	: Gas
Evaporation point	: N/A
pH	: N/A
Odor and appearance	: Colorless gas with a rotten-egg odor

10. STABILITY AND REACTIVITY

Stable under normal conditions. Expected shelf life 12 months.

11. TOXICOLOGICAL INFORMATION

This gas mixture contains components that may cause embryo toxic effects in humans; however, due to the small size of the cylinder no toxicological damage is anticipated.

12. ECOLOGICAL INFORMATION

No ecological damage is expected to be caused by this product.

13. DISPOSAL INFORMATION

Do not discharge into any place where its accumulation could be dangerous. Used containers are acceptable for disposal in the normal waste stream as long as the cylinder is empty and valve removed or cylinder wall is punctured; but SpecAir Specialty Gases encourages the consumer to return cylinders back to manufacturer.

14. TRANSPORT INFORMATION

	United States DOT	Canada TDG
Proper Shipping Name:	Compressed Gas N.O.S. (Hydrogen Sulfide, Nitrogen)	Compressed Gas N.O.S. (Hydrogen Sulfide, Nitrogen)
Hazard Class:	2.2	2.2
Identification Number:	UN1956	UN1956
Shipping Label:	Non-Flammable Gas	Non-Flammable Gas

15. REGULATORY INFORMATION

The components of this product are listed under the accident prevention provisions of section 112(r) of the Clean Air Act (CAA) with a threshold quantity (TQ) of 10,000 pounds each.

16. OTHER INFORMATION

This MSDS has been prepared in accordance with the Chemicals (Hazard Information and Packaging for Supply (Amendment) Regulation 1996. The information is based on the best knowledge of SpecAir Specialty Gases and its advisors and is given in good faith, but we cannot guarantee its accuracy, reliability or completeness and therefore disclaim any liability for loss or damage arising out of use of this data. Since conditions of use are outside the control of the Company and its advisors we disclaim any liability for loss or damage when the product is used for other purposes than it is intended.

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.

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

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 Eye Irrit. 2; H319	12-28
CAS number: 68081-81-2	Sodium Alkylbenzene Sulfonate	Acute Tox. 4; H303 Skin Irrit. 2 ; H315 Eye Irrit. 2; H319	8-22
CAS number: 7722-88-5	Tetrasodium Pyrophosphate	Skin Irrit. 2 ; H315 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.

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

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.

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

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.

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

Flammability (solid, gaseous):	Not determined or not available.	**Viscosity**:	a. Kinematic: Not determined or not available. 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

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

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.

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

	Comments: None	**Comments:** None
14.4	**Packing group:** 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.

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

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

Safety Data Sheet (SDS)

OSHA HazCom Standard 29 CFR 1910.1200(g) and GHS Rev 03.

Issue date 05/28/2015 Reviewed on 05/28/2015

* 1 Identification

· **Product identifier**

· **Trade name: Precision Calibration Gas Mixture**
· **Product number:** G-4015
· **Relevant identified uses of the substance or mixture and uses advised against**
Used for calibration of gas measuring devices. Not suitable for human consumption.
· **Product description** Calibration gas mixture consisting of Helium.

· **Details of the supplier of the safety data sheet**
· **Manufacturer/Supplier:**
Gasco Affiliates, LLC
320 Scarlett Blvd.
Oldsmar, Fl 34677

TELEPHONE NUMBER: (800) 910-0051
FAX NUMBER: (866) 755-8920
E-MAIL: info@gascogas.com
· **Emergency telephone number:**
Inside the US: 1-800-424-9300 (CHEMTREC, 24 hours)
Outside the US: 1-703-527-3887 (CHEMTREC, 24 hours)

* 2 Hazard(s) identification

· **Classification of the substance or mixture**

GHS04 Gas cylinder

Press. Gas H280 Contains gas under pressure; may explode if heated.

GHS07

STOT SE 3 H336 May cause drowsiness or dizziness.

Simple Asphyxiant May displace oxygen and cause rapid suffocation.

· **Label elements**
· **GHS label elements**
The substance is classified and labeled according to the Globally Harmonized System (GHS).
· **Hazard pictograms**

GHS04 GHS07

· **Signal word** Warning

· **Hazard-determining components of labeling:**
Helium
· **Hazard statements**
Contains gas under pressure; may explode if heated.
May cause drowsiness or dizziness.
May displace oxygen and cause rapid suffocation.

(Contd. on page 2)

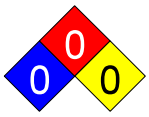
Safety Data Sheet (SDS)

OSHA HazCom Standard 29 CFR 1910.1200(g) and GHS Rev 03.

Issue date 05/28/2015 Reviewed on 05/28/2015

Trade name: Precision Calibration Gas Mixture

· ***Precautionary statements***
Avoid breathing dust/fume/gas/mist/vapors/spray.
Use only outdoors or in a well-ventilated area.
IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing.
Call a poison center/doctor if you feel unwell.
Store locked up.
Protect from sunlight.
Store in a well-ventilated place. Keep container tightly closed.
Dispose of contents/container in accordance with local/regional/national/international regulations.
· ***Unknown acute toxicity:***
100 percent of the mixture consists of ingredient(s) of unknown toxicity.
· ***Classification system:***
· ***NFPA ratings (scale 0 - 4)***



Health = 0
Fire = 0
Reactivity = 0

· ***HMIS-ratings (scale 0 - 4)***

Health = 0
Fire = 0
Reactivity = 0

· ***Hazard(s) not otherwise classified (HNOC):*** None known

*
3 Composition/information on ingredients

· ***Chemical characterization: Mixtures***
· ***Description:*** Mixture of substances listed below with nonhazardous additions.

· ***Dangerous Components:***		
CAS: 7440-59-7 RTECS: MH 6520000	Helium Press. Gas, H280; STOT SE 3, H336; Simple Asphyxiant	99 - 99.999%

*
4 First-aid measures

· ***Description of first aid measures***
· ***After inhalation:***
Generally the product does not irritate with inhalation.
Supply fresh air. If required, provide artificial respiration. Consult doctor if symptoms persist.
In case of unconsciousness, place patient securely on side position for transportation.
· ***After skin contact:***
Generally the product does not irritate the skin.
In cases of contact with liquified material, frostbite may occur. Immerse frostbite in cool-warm water and seek medical attention.
· ***After eye contact:***
Not anticipated under normal use.
Rinse opened eye for several minutes under running water. If symptoms persist, consult a doctor.
· ***After swallowing:***
Not a normal route of entry.
If swallowed and symptoms occur, consult a doctor.
· ***Information for doctor:***
· ***Most important symptoms and effects, both acute and delayed:*** No further relevant information available.

(Contd. on page 3)

Safety Data Sheet (SDS)

OSHA HazCom Standard 29 CFR 1910.1200(g) and GHS Rev 03.

Issue date 05/28/2015 Reviewed on 05/28/2015

Trade name: Precision Calibration Gas Mixture

· **Indication of any immediate medical attention and special treatment needed**
No further relevant information available.

*
5 Fire-fighting measures

· **Extinguishing media**
· **Suitable extinguishing agents:**
Use fire fighting measures that suit the environment.
Use water spray to cool fire-exposed containers.
· **Special hazards arising from the substance or mixture**
Closed containers may explode when exposed to extreme heat.
· **Advice for firefighters**
This gas mixture is not flammable; however, containers, when involved in fire, may rupture or burst in the heat of the fire.
· **Protective equipment:**
As in any fire, wear self-contained breathing apparatus pressure-demand (NIOSH approved or equivalent), and full protective gear to prevent contact with skin and eyes.

*
6 Accidental release measures

· **Personal precautions, protective equipment and emergency procedures**
In a confined area, NIOSH approved respiratory protection may be required.
Treat any fumes as toxic.
· **Environmental precautions:** No special measures required.
· **Methods and material for containment and cleaning up:**
Dispose of the collected material according to regulations.
· **Reference to other sections**
See Section 7 for information on safe handling.
See Section 8 for information on personal protection equipment.
See Section 13 for disposal information.

*
7 Handling and storage

· **Handling:**
· **Precautions for safe handling**
Ensure good ventilation/exhaustion at the workplace.
Open and handle receptacle with care.
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 the potential for oxygen deficiency (simple asphyxiation). Do not attempt to adjust, repair or in any other way modify the cylinders containing this gas mixture. If there is a malfunction or another type of operational problem, contact nearest distributor immediately.
· **Information about protection against explosions and fires:**
Pressurized container: protect from sunlight and do not expose to temperatures exceeding 50 °C, i.e. electric lights. Do not pierce or burn, even after use.
Keep protective respiratory device available.
Do not cut, grind or weld on container that contains or contained product.
Do not spray on a naked flame or any incandescent material.

· **Conditions for safe storage, including any incompatibilities** Store away from strong oxidizing agents.
· **Storage:**
· **Requirements to be met by storerooms and receptacles:**
Store in a cool location.
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. Cylinders should be stored in dry, well-ventilated areas, away from sources of heat, ignition, and direct sunlight. Protect cylinders against physical damage.

(Contd. on page 4)

Safety Data Sheet (SDS)

OSHA HazCom Standard 29 CFR 1910.1200(g) and GHS Rev 03.

Issue date 05/28/2015 Reviewed on 05/28/2015

Trade name: Precision Calibration Gas Mixture

Full and empty cylinders should be segregated. Use a "first-on, first-out" inventory system to prevent full containers from being stored for long periods of time.
· **Information about storage in one common storage facility:** Not required.
· **Further information about storage conditions:** Store in cool, dry conditions in well sealed receptacles.
· **Specific end use(s)** No further relevant information available.

* 8 Exposure controls/personal protection

· **Additional information about design of technical systems:** No further data; see section 7.

· **Control parameters**
All ventilation should be designed in accordance with OSHA standard (29 CFR 1910.94). Use mechanical (general) ventilation for storage areas. Use appropriate ventilation as required to keep Exposure Limits in Air below TLV & PEL limits.

· **Components with occupational exposure limits:**	
7440-59-7 Helium	
TLV	TLV withdrawn, see App. F; simple asphyxiant

· **Additional information:** The lists that were valid during the creation of this SDS were used as basis.

· **Exposure controls**
· **Personal protective equipment:**
· **General protective and hygienic measures:**
Keep away from foodstuffs, beverages and feed.
Wash hands before breaks and at the end of work.
· **Breathing equipment:**
Not necessary if room is well-ventilated.
In case of brief exposure or low pollution use respiratory filter device. In case of intensive or longer exposure, use respiratory protective device that is independent of circulating air.
· **Protection of hands:** Not required.
· **Eye protection:** Not necessary under normal conditions.

* 9 Physical and chemical properties

· **Information on basic physical and chemical properties**
· **General Information**
· **Appearance:**
Form: Gaseous
Color: Clear, colorless
· **Odor:** Odorless
· **Odor threshold:** Not determined.

· **pH-value:** Not determined.

· **Change in condition**
Melting point/Melting range: -272 °C (-458 °F)
Boiling point/Boiling range: -268 °C (-450 °F)

· **Flash point:** Not applicable.

· **Flammability (solid, gaseous):** Product is not flammable.

· **Ignition temperature:**

Decomposition temperature: Not determined.

· **Auto igniting:** Product is not self-igniting.

(Contd. on page 5)

Safety Data Sheet (SDS)

OSHA HazCom Standard 29 CFR 1910.1200(g) and GHS Rev 03.

Issue date 05/28/2015 Reviewed on 05/28/2015

Trade name: Precision Calibration Gas Mixture

· **Danger of explosion:**	Not determined.
· **Explosion limits:**	
Lower:	Not determined.
Upper:	Not determined.
· **Vapor pressure:**	Not determined.
· **Density @ 20 °C (68 °F):**	0.178 g/cm³ (1.485 lbs/gal)
· **Relative density**	Not determined.
· **Vapor density**	Not determined.
· **Evaporation rate**	Not applicable.
· **Solubility in / Miscibility with**	
Water @ 20 °C (68 °F):	8.6 g/l
· **Partition coefficient (n-octanol/water):**	Not determined.
· **Viscosity:**	
Dynamic:	Not determined.
Kinematic:	Not determined.
· **Solvent content:**	
Organic solvents:	0.0 %
· **Other information**	No further relevant information available.

* 10 Stability and reactivity

· **Reactivity** No further relevant information available.
· **Chemical stability** Stable under normal conditions.
· **Thermal decomposition / conditions to be avoided:** No decomposition if used according to specifications.
· **Possibility of hazardous reactions** No dangerous reactions known.
· **Conditions to avoid** No further relevant information available.
· **Incompatible materials:** Strong oxidizing agents.
· **Hazardous decomposition products:** None.

* 11 Toxicological information

· **Information on toxicological effects** The toxicity of this product is unknown.
· **Acute toxicity:**
· **Primary irritant effect:**
· **on the skin:** No irritating effect.
· **on the eye:** No irritating effect.
· **Additional toxicological information:**

· **Carcinogenic categories**
· **IARC (International Agency for Research on Cancer)**
Group 1 - Carcinogenic to humans
Group 2A - Probably carcinogenic to humans
Group 2B - Possibly carcinogenic to humans
Group 3 - Not classifiable as to its carcinogenicity to humans
Group 4 - Probably not carcinogenic to humans

None of the ingredients are listed.

· **NTP (National Toxicology Program)**

None of the ingredients are listed.

(Contd. on page 6)

Safety Data Sheet (SDS)

OSHA HazCom Standard 29 CFR 1910.1200(g) and GHS Rev 03.

Issue date 05/28/2015 Reviewed on 05/28/2015

Trade name: Precision Calibration Gas Mixture

· **OSHA-Ca (Occupational Safety & Health Administration)**
None of the ingredients are listed.

* 12 Ecological information

· **Toxicity** The hazards for the aquatic environment are unknown.
· **Aquatic toxicity:** No further relevant information available.
· **Persistence and degradability** No further relevant information available.
· **Behavior in environmental systems:**
· **Bioaccumulative potential** No further relevant information available.
· **Mobility in soil** No further relevant information available.
· **Additional ecological information:**
· **General notes:** Generally not hazardous for water.
· **Results of PBT and vPvB assessment**
· **PBT:** Not applicable.
· **vPvB:** Not applicable.
· **Other adverse effects** No further relevant information available.

* 13 Disposal considerations

· **Waste treatment methods**
· **Recommendation:**
Release all residual gas pressure in a well ventilated area. Verify the cylinder is completely empty (0 PSIG).
Remove or cover any hazard labels. Return empty cylinder for recycling.
NOTE: Check with the local easte authority before placing any gas cylinder into waste container for pickup.
GASCO encourages the consumer to return all cylinders.
· **Waste disposal key:**
The U.S. EPA has not published waste disposal numbers for this product's components.

· **Uncleaned packagings:**
· **Recommendation:** Return cylinder and unused product to supplier.

* 14 Transport information

· **UN-Number**
· **DOT, ADR, IMDG, IATA** UN1046
· **UN proper shipping name**
· **DOT** Helium, compressed
· **ADR** UN1046 Helium, compressed
· **IMDG, IATA** HELIUM, COMPRESSED
· **Transport hazard class(es)**

· **DOT**

· **Class** 2.2
· **Label** 2.2

(Contd. on page 7)

Safety Data Sheet (SDS)

OSHA HazCom Standard 29 CFR 1910.1200(g) and GHS Rev 03.

Issue date 05/28/2015 Reviewed on 05/28/2015

Trade name: Precision Calibration Gas Mixture

· **ADR**

· **Class**	2.2 1A
· **Label**	2.2

· **IMDG, IATA**

· **Class**	2.2
· **Label**	2.2
· **Packing group**	
· **ADR, IMDG, IATA**	Non-Regulated Material
· **Environmental hazards:**	Not applicable.
· **Special precautions for user**	Not applicable.
· **Danger code (Kemler):**	20
· **EMS Number:**	F-C,S-V
· **Transport in bulk according to Annex II of MARPOL73/78 and the IBC Code**	Not applicable.

· **Transport/Additional information:**

· **DOT**

· **Quantity limitations**	On passenger aircraft/rail: 75 kg On cargo aircraft only: 150 kg

· **ADR**

· **Excepted quantities (EQ)**	Code: E1 Maximum net quantity per inner packaging: 30 ml Maximum net quantity per outer packaging: 1000 ml

· **IMDG**

· **Limited quantities (LQ)**	120 ml
· **Excepted quantities (EQ)**	Code: E1 Maximum net quantity per inner packaging: 30 ml Maximum net quantity per outer packaging: 1000 ml
· **UN "Model Regulation":**	UN1046, Helium, compressed, 2.2

* 15 Regulatory information

· **Safety, health and environmental regulations/legislation specific for the substance or mixture**
· **Sara**

· **Section 355 (extremely hazardous substances):**
None of the ingredients are listed.

· **Section 313 (Specific toxic chemical listings):**
None of the ingredients are listed.

· **TSCA (Toxic Substances Control Act):**
All ingredients are listed.

(Contd. on page 8)

Safety Data Sheet (SDS)

OSHA HazCom Standard 29 CFR 1910.1200(g) and GHS Rev 03.

Issue date 05/28/2015 Reviewed on 05/28/2015

Trade name: Precision Calibration Gas Mixture

· **California Proposition 65**

· **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.

· **Carcinogenic categories**

· **EPA (Environmental Protection Agency)**
None of the ingredients are listed.

· **TLV (Threshold Limit Value established by ACGIH)**
None of the ingredients are listed.

· **NIOSH-Ca (National Institute for Occupational Safety and Health)**
None of the ingredients are listed.

· **GHS label elements**
The substance is classified and labeled according to the Globally Harmonized System (GHS).

· **Hazard pictograms**

GHS04 GHS07

· **Signal word** Warning

· **Hazard-determining components of labeling:**
Helium

· **Hazard statements**
Contains gas under pressure; may explode if heated.
May cause drowsiness or dizziness.
May displace oxygen and cause rapid suffocation.

· **Precautionary statements**
Avoid breathing dust/fume/gas/mist/vapors/spray.
Use only outdoors or in a well-ventilated area.
IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing.
Call a poison center/doctor if you feel unwell.
Store locked up.
Protect from sunlight.
Store in a well-ventilated place. Keep container tightly closed.
Dispose of contents/container in accordance with local/regional/national/international regulations.

· **National regulations:**
The product is subject to be classified according with the latest version of the regulations on hazardous substances.

(Contd. on page 9)

Safety Data Sheet (SDS)

OSHA HazCom Standard 29 CFR 1910.1200(g) and GHS Rev 03.

Issue date 05/28/2015 Reviewed on 05/28/2015

Trade name: Precision Calibration Gas Mixture

· **State Right to Know**		
CAS: 7440-59-7 RTECS: MH 6520000	Helium Press. Gas, H280; STOT SE 3, H336; Simple Asphyxiant	99 - 99.999%
All ingredients are listed.		

· **Chemical safety assessment:** A Chemical Safety Assessment has not been carried out.

* 16 Other information

· **Relevant phrases**

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· **Date of preparation / last revision** 05/28/2015 / -

· **Abbreviations and acronyms:**

ADR: The European Agreement concerning the International Carriage of Dangerous Goods by Road
ADN: The European Agreement concerning the International Carriage of Dangerous Goods by Inland Waterways
IMDG: International Maritime Code for Dangerous Goods
DOT: US Department of Transportation
IATA: International Air Transport Association
ACGIH: American Conference of Governmental Industrial Hygienists
EINECS: European Inventory of Existing Commercial Chemical Substances
ELINCS: European List of Notified Chemical Substances
CAS: Chemical Abstracts Service (division of the American Chemical Society)
NFPA: National Fire Protection Association (USA)
HMIS: Hazardous Materials Identification System (USA)
Press. Gas: Gases under pressure: Compressed gas
STOT SE 3: Specific target organ toxicity - Single exposure, Hazard Category 3

· ***** Data compared to the previous version altered.**

SDS created by MSDS Authoring Services www.msdsauthoring.com +1-877-204-9106

MESA Specialty Gases & Equipment
2427 S. Anne Street
Santa Ana, California 92704 USA
Domestic US: (866) 470-6372; International 714-434-7102
www.mesagas.com

SAFETY DATA SHEET

SECTION 1 – IDENTIFICATION

Chemical Name:	Isobutylene in Air
Chemical Formula:	C4H8 Balance Air
Chemical Family:	Inert Gas Mixture
Hazard Classification:	Compressed Gas, N.O.S., Non-Flammable Gas, UN1956, Green Label
Product Use Description:	Analytical Standard and General Laboratory Applications
Company:	MESA Specialty Gases & Equipment 2427 South Anne Street Santa Ana, California 92704 USA
Phone Number for Information:	Infotrac
Emergency Contact:	800-535-5053 (Int'l: 352-323-3500)

SECTION 2 – HAZARD(S) IDENTIFICATION

SIGNAL WORD - WARNING

HAZARD STATEMENTS: Contains gas under pressure; may explode if heated.
PRECAUTIONARY STATEMENTS:
General: Use in accordance with Safety Data Sheets.
Do not ingest or inhale. Avoid contact with skin and clothing.
Prevention: Keep away from heat, hot surfaces, sparks, open flames, and other ignition sources. No smoking.
Response: Leaking gas fire: Do not extinguish unless leak can be stopped safely.
In case of leakage, eliminate all ignition sources.
Do not open valve until prepared to use.
Always use a back flow preventative device in piping.
Storage: Protect from sunlight. Store in a well-ventilated place.
OTHER HAZARDS: High pressure gas.
Self-contained breathing apparatus (SCBA) may be required.

SECTION 3 – COMPOSITION/INFORMATION ON INGREDIENTS

COMPONENT	CAS NO.	CONCENTRATION
Isobutylene	115-11-7	0.1 ppm - 1.9 Mol%
Air (79.1% N2 + 20.9% O2)	See Below	Balance
Oxygen	7782-44-7	
Nitrogen	7727-37-9	

SECTION 4 – FIRST AID MEASURES

ROUTE OF EXPOSURE:
Inhalation: Remove person to uncontaminated area. SCBA may be required to prevent asphyxiation of rescue workers. Keep warm and at rest. Lay victim face down with head and chest lower than hips to improve drainage from lungs. If breathing is labored, administer pure oxygen. If breathing has stopped, start artificial respiration. Get immediate medical attention for serious exposure.
Eye contact: Immediately flush eyes with plenty of water, occasionally lifting the upper and lower eyelids. Check for and remove any contact lenses. Continue to rinse for at least 10 minutes. Get medical attention if irritation occurs.
Skin contact: Flush contaminated skin with plenty of water. Remove contaminated clothing and shoes. Get medical attention if symptoms occur. Wash clothing before reuse. Clean shoes thoroughly before reuse.
Ingestion: N/A

SYMPTOMS: Overexposure can cause tingling of the fingers and toes, abnormal sensations, and impaired coordination and confusion. Decompression sickness, the “bends”, is possible following rapid decompression. Symptoms of frostbite include change in skin color to white or grayish-yellow. The pain after contact with liquid can quickly subside.

SECTION 5 – FIRE FIGHTING MEASURES

EXTINGUISHING MEDIA: Use appropriate media for surrounding fire such as CO2 foam extinguishers

UNUSUAL FIRE AND EXPLOSION HAZARDS: Gas cylinders may rupture violently when exposed to fire. Continue to cool fire exposed cylinders until flames are extinguished. Cylinder valve is equipped with a pressure relief device to safely vent the cylinder if it is exposed to elevated pressure in a fire.

SPECIAL FIRE FIGHTING PROCEDURES: Wear NIOSH/MSHA approved SCBA and full protective equipment. Stop flow of gas if this can be done safely. Use water spray to keep cylinders cool.

SECTION 6 – ACCIDENTAL RELEASE MEASURES

PERSONAL PRECAUTIONS, PROTECTIVE EQUIPMENT, AND EMERGENCY PROCEDURES: Wear self-contained breathing apparatus when entering area unless atmospehre is proved to be safe. Monitor oxygen level. Ventilate the area.
ENVIRONMENTAL PRECAUTIONS: Prevent spreading of vapors through sewers, ventilation systems, and confined areas. Do not discharge materials into any place where their accumulation could be dangerous.

METHODS AND MATERIALS FOR CONTAINMENT AND CLEANING UP: Stop the folow of gas or remove cylinder to outdoor location if this can be done without risk. Ventilate enclosed areas. Move leaking cylinder to fume hood or safe outdoor area. Use monitoring equipment if hazardous conditions are suspected or likely to occur.

SECTION 7 – HANDLING AND STORAGE

PRECAUTIONS FOR SAFE HANDLING: Only experienced and properly instructed persons should handle compressed gases. Person is to know and understand the properties and hazards of the product before use. Do not remove or deface labels provided by the supplier for the identification of the product.

Protect cyinders from physical damage to prevent valve damage or leakage. Move cylinders properly; do not drag, slide, or drop cylinders when transporting. Use adjustable strap wrench to remove tight/rusted caps.Ensure the complete gas system has been checked for leaks before use. Never insert any object into valve cap openings; doing so may damage valve causing leakage.

Gas or liquefied gas are to be used with the appropriate pressure regulating control and high pressure equipment. Suck-back into cylinder may cause rupture. Always use a back flow preventative device in piping. Never lift cylinder by its valve protection cap. Use only in ventilated areas.

CONDITIONS FOR SAFE STORAGE: Cylinders should be secured with mounting brackets away from heavily traveled areas. Use oldest cylinders in stock first to prevent full cylinders from being stored for excessive periods of time. Full and empty cylinders should be segregated. Keep cylinder in dry, cool, well ventilated area away from heat, flame, sparks or corrosive chemicals. Cylinders should be moved by suitable hand trucks. Close valve after each use and when empty.Cylinder valve guards or caps should be in place. Cylinder temperature should not exceed 52°C (125°F). Store containers in location free from fire risk and away from any sources of heat and ignition. Keep cylinder away from combustible material. Use equipment rated for cylinder pressure.

SECTION 8 – EXPOSURE CONTROLS/PERSONAL PROTECTION

COMPONENT	OSHA PEL	ACGIH TLV
Isobutylene	None	None
Air (79.1% N2 + 20.9% O2)	See Below	See Below
Oxygen	None	None
Nitrogen	None	None

APPROPRIATE ENGINEERING CONTROLS: Ventilation: Enclosed area must be provided with general or local exhaust ventilation to avoid hazardous conditions.

INDIVIDUAL PROTECTIVE MEASURES: Safety glasses, work gloves, and safety shoes should be worn when handling high pressure cylinders or hazardous materials.
Respiratory Protection (Specify Type): Use self-contained breathing apparatus in emergency or rescue situations.

SECTION 9 – PHYSICAL AND CHEMICAL PROPERTIES

Appearance: Colorless	Upper/lower flammability/explosive limits: No data available
Odor: Odorless	Vapor Pressure: N/A
Odor threshold: No data available	Vapor Density (Air=1): Varies
pH: N/A	Relative Density (Water=1): Varies
Melting point/range: N/A	Solubility (in water): N/A
Boiling point/range: N/A	Partition coefficient (n-octanol/water): N/A
Flash Point: N/A	Auto-ignition temperature: No data available
Evaporation Rate (Butyl Acetate=1): N/A	Decomposition temperature: No data available
Flammability (solid, gas): No data available	Visocity: N/A

SECTION 10 – STABILITY AND REACTIVITY DATA

Reactivity: Refer to possibility of hazardous reactions and/or incompatible materials sections	**Conditions to avoid:** Flame, excessive heat
Chemical Stability: Stable	**Incompatible materials:** None
Possibility of hazardous reactions: No data available	**Hazardous Decomposition or Byproducts:** None

SECTION 11 – TOXICOLOGICAL INFORMATION

LIKELY ROUTES OF EXPOSURE:
Inhalation: Overexposure by inhalation at elevated or reduced pressure. Remove person to uncontaminated area. SCBA may be required to prevent asphyxiation of rescue workers. Keep warm and at rest. Lay victim face down with head and chest lower than hips to improve drainage from lungs. If breathing is labored, administer pure oxygen. If breathing has stopped, start artificial respiration. Get immediate medical attention for serious exposure.
Eye contact: Immediately flush eyes with plenty of water, occasionally lifting the upper and lower eyelids. Check for and remove any contact lenses. Continue to rinse for at least 10 minutes. Get medical attention if irritation occurs.
Skin contact: Flush contaminated skin with plenty of water. Remove contaminated clothing and shoes. Get medical attention if symptoms occur. Wash clothing before reuse. Clean shoes thoroughly before reuse.
Ingestion: N/A

SYMPTOMS/EFFECTS FROM EXPOSURE: May cause frostbite. Overexposure can cause tingling of the fingers and toes, abnormal sensations, and impaired coordination and confusion. Decompression sickness, the “bends”, is possible following rapid decompression. Symptoms of frostbite include change in skin color to white or grayish-yellow. The pain after contact with liquid can quickly subside.

ACUTE/CHRONIC TOXICITY: This is a customized mixtures of liquids and/or gases. No data is available on the final product itself. Refer to Sections 2 and 3 for hazards related to specific components of the combined mixture.

CARCINOGENICITY: No data available.

SECTION 12 – ECOLOGICAL INFORMATION

Ecotocity (aquatic and terrestrial): No known effects. Any adverse effect on animals would be related to oxygen deficient environments, as well as respiratory system damage. Additionally, frost produced in the presence of rapidly expanding gases may adversely affect plant life.
Persistence and degradability: No data available
Bioaccumulative potential: No data available
Mobility in soil: No data available

Other Effects: The mixture does not contain any class I or Class II ozone depleting chemicals.

SECTION 13 – DISPOSAL CONSIDERATIONS

Disposal: Waste disposal must be in accordance with appropriate National, Federal, State, and local regulations. Do not dispose or discharge into the environment. Do not discharge into enclosed environment. Contact supplier if additional guidance is required.

SECTION 14 – TRANSPORTATION INFORMATION

DOT Classification:

Proper Shipping Name: Compressed Gas, N.O.S.
Class: 2.2
UN/ID No.: UN1956
Label: Non-Flammable Gas, Green Label

IATA Classification:

Proper Shipping Name: Compressed Gas, N.O.S.
Class: 2.2
UN/ID No.: UN1956
Label: Non-Flammable Gas, Green Label

Environment hazard: No

Transport in bulk (according to Annex II of MARPOL 73/78 and the IBC Code: N/A

SPECIAL PRECAUTIONS FOR USER: Avoid transport on vehicles where the load space is not separated from driver's compartment. Ensure that transporter is aware of the potential hazards of the load and knows what to do in event of an emergency. Contact supplier for complete transportation information.

SECTION 15 – REGULATORY INFORMATION

U.S. SARA REPORTING REQUIREMENTS: The components of this gas mixture are subject to the reporting requirements of Sections 302, 304 and 313 of Title III of the Superfund Amendments and Reauthorization Act, as follows.

CHEMICAL NAME SARA 302
(40 CFR 355, Appendix A) SARA 304
(40 CFR Table 302.4) SARA 313
(40 CFR 372.65)
HEXANE NO YES YES

U.S. SARA THRESHOLD PLANNING QUANTITY: Not applicable.

U.S. CERCLA REPORTABLE QUANTITY (RQ): Not applicable.

CANADIAN DSL/NDSL INVENTORY STATUS: The components of this gas mixture are on the DSL Inventory.

U.S. TSCA INVENTORY STATUS: The components of this gas mixture are on the TSCA Inventory.

CANADIAN WHMIS : Class A: Compressed Gas

LABELING (For Compressed Gas):

WARNING: CONTAINS GAS UNDER PRESSURE; MAY EXPLODE IF HEATED. MAY CAUSE RAPID SUFFOCATION BY DISPLACING OXYGEN IN THE AIR. MAY INCREASE RESPIRATION AND HEART RATE. May cause dizziness, nausea, drowsiness, vomiting, excess salivation, and loss of mobility/consciousness. May cause frostbite. Keep away from heat, hot surfaces, sparks, open flames and other ignition sources—No smoking. Use and store in well-ventilated areas. Leaking gas fire: Do not extinguish unless leak can be stopped safely. In case of leakage, eliminate all ignition sources. Do not open valve until prepared to use. Always use a backflow preventative device in piping. Use only with equipment rated for cylinder pressure. Close valve after each use and when empty. Cylinder temperature should not exceed 52°C (125°F). Use in accordance with Safety Data Sheet. FIRST AID: IF INHALED, remove to fresh air. If breathing is difficult, give Oxygen. Call a physician. IN CASE OF FROSTBITE, obtain immediate medical attention. DO NOT REMOVE THIS LABEL.

SECTION 16 – OTHER INFORMATION

Information contained in this data sheet is offered without charge for use by technically qualified personnel at their discretion and risk. All statements, technical information and recommendations contained herein are based on tests and data which we believe to be reliable. But the accuracy and completeness thereof, is not guaranteed and no warranty of any kind, either expressed or implied, is made with respect thereto. Since MESA Specialty Gases and Equipment Division of MESA International Technologies, Inc. shall have no control over the use of the product described herein, we assume no liability for loss or damage incurred from the proper or improper use of such product.

HISTORY:

Date of printing:	12/17/2018
Date of issue/revision:	12/17/2018
Date of previous issue:	12/1/2014

DISCLAIMER

Information contained in this data sheet is offered without charge for use by technically qualified personnel at their discretion and risk. All statements, technical information and recommendations contained herein are based on tests and data which we believe to be reliable. But the accuracy and completeness thereof, is not guaranteed and no warranty of any kind, either expressed or implied, is made with respect thereto. Since MESA Specialty Gases and Equipment Division of MESA International Technologies, Inc. shall have no control over the use of the product described herein, we assume no liability for loss or damage incurred from the proper or improper use of such product.

Section 1: IDENTIFICATION

Product Name: Simple Green® All-Purpose Cleaner
Additional Names:

Manufacturer's Part Number: **Please refer to Section 16*

Recommended Use: Cleaner & Degreaser for water tolerant surfaces.
Restrictions on Use: Do not use on non-rinsable surfaces.

Company: Sunshine Makers, Inc.
15922 Pacific Coast Highway
Huntington Beach, CA 92649 USA
Telephone: 800-228-0709 ● 562-795-6000 *Mon – Fri, 8am – 5pm PST*
Fax: 562-592-3830
Email: info@simplegreen.com
Emergency Phone: Chem-Tel 24-Hour Emergency Service: 800-255-3924

Section 2: HAZARDS IDENTIFICATION

This product has been assessed in accordance to 2012 OSHA Hazard Communication Standards (29 CFR 1910.1200) and has been determined to not be classifiable as hazardous.

OSHA HCS 2012
Label Elements
Signal Word: None **Hazard Symbol(s)/Pictogram(s):** None required

Hazard Statements: None
Precautionary Statements: None
Hazards Not Otherwise Classified (HNOC): None
Other Information: None Known

Section 3: COMPOSITION/INFORMATION ON INGREDIENTS

Ingredient	CAS Number	Percent Range
Water	7732-18-5	> 84.8%*
C9-11 Alcohols Ethoxylated	68439-46-3	< 5%*
Sodium Citrate	68-04-2	< 5%*
Sodium Carbonate	497-19-8	< 1%*
Tetrasodium Glutamate Diacetate	51981-21-6	< 1%*
Citric Acid	77-92-9	< 1%*
Methylchloroisothiazolinone	26172-55-4	< 0.002%*
Methylisothiazolinone	2682-20-4	< 0.001%*
Fragrance	Proprietary Mixture	< 1%*
Liquitint Colorant	Proprietary Mixture	< 1%*

**specific percentages of composition are being withheld as a trade secret*

Section 4: FIRST-AID MEASURES

Inhalation: Not expected to cause respiratory irritation. If adverse effect occurs, move to fresh air.
Skin Contact: Not expected to cause skin irritation. If adverse effect occurs, rinse skin with water.
Eye Contact: Not expected to cause eye irritation. If adverse effect occurs, flush eyes with water.
Ingestion: May cause upset stomach. Drink plenty of water to dilute. See section 11.

Most Important Symptoms/Effects, Acute and Delayed: None known.

Indication of Immediate Medical Attention and Special Treatment Needed, if necessary: Treat symptomatically

Section 5: FIRE-FIGHTING MEASURES

Suitable & Unsuitable Extinguishing Media: Use Dry chemical, CO2, water spray or "alcohol" foam. Avoid high volume jet water.
Specific Hazards Arising from Chemical: In event of fire, fire created carbon oxides may be formed.
Special Protective Actions for Fire-Fighters: Wear positive pressure self-contained breathing apparatus; Wear full protective clothing.

This product is non-flammable. See Section 9 for Physical Properties.

Section 6: ACCIDENTAL RELEASE MEASURES

Personal Precautions, Protective Equipment and Emergency Procedures: *For non-emergency and emergency personnel:* See section 8 – personal protection. Avoid eye contact. Safety goggles suggested.

Environmental Precautions: Do not allow into open waterways and ground water systems.

Methods and Materials for Containment and Clean Up: Dike or soak up with inert absorbent material. See section 13 for disposal considerations.

Section 7: HANDLING AND STORAGE

Precautions for Safe Handling: Ensure adequate ventilation. Keep out of reach of children. Keep away from heat, sparks, open flame and direct sunlight. Do not pierce any part of the container. Do not mix or contaminate with any other chemical. Do not eat, drink or smoke while using this product.

Conditions for Safe Storage including Incompatibilities: Keep container tightly closed. Keep in cool dry area. Avoid prolonged exposure to sunlight. Do not store at temperatures above 109°F (42.7°C). If separation occurs, mix the product for reconstitution.

Section 8: EXPOSURE CONTROLS / PERSONAL PROTECTION

Exposure Limit Values: No components listed with TWA or STEL values under OSHA or ACGIH.

Appropriate Engineering Controls: Showers, eyewash stations, ventilation systems

Individual Protection Measures / Personal Protective Equipment (PPE)
Eye Contact: Use protective glasses or safety goggles if splashing or spray-back is likely.
Respiratory: Use in well ventilated areas or local exhaust ventilations when cleaning small spaces.
Skin Contact: Use protective gloves (any material) when used for prolonged periods or dermally sensitive.
General Hygiene Considerations: Wash thoroughly after handling and before eating or drinking.

Section 9: PHYSICAL AND CHEMICAL PROPERTIES

Appearance:	Green Liquid	**Partition Coefficient: n-octanol/water:**	Not determined
Odor:	Added sassafras odor	**Autoignition Temperature:**	Non-flammable
Odor Threshold:	Not determined	**Decomposition Temperature:**	42.7°C (109°F)
pH ASTM D-1293**:**	8.5 – 9.2	**Viscosity:**	Like water
Freezing Point ASTM D-1177**:**	0-3.33°C (32-38°F)	**Specific Gravity** ASTM D-891**:**	1.01 – 1.03
Boiling Point & Range ASTM D-1120**:**	101°C (213.8°F)	**VOCs:**	***Water & fragrance exemption in calculation
Flash Point ASTM D-93**:**	> 212°F	SCAQMD 304-91 / EPA 24:	0 g/L / 0 lb/gal / 0%
Evaporation Rate ASTM D-1901**:**	½ Butyl Acetate @ 25°C	CARB Method 310**:	2.5 g/L / 0.021 lb/gal / 0.25%
Flammability (solid, gas):	Not applicable	SCAQMD Method 313:	Not tested
Upper/Lower Flammability or Explosive Limits:	Not applicable	**VOC Composite Partial Pressure:**	Not determined
Vapor Pressure ASTM D-323**:**	0.60 PSI @77°F, 2.05 PSI @100°F	**Relative Density** ASTM D-4017**:**	8.42 – 8.59 lb/gal
Vapor Density:	Not determined	**Solubility:**	100% in water

Section 10: STABILITY AND REACTIVITY

Reactivity: Non-reactive.
Chemical Stability: Stable under normal conditions 70°F (21°C) and 14.7 psig (760 mmHg).
Possibility of Hazardous Reactions: None known.
Conditions to Avoid: Excessive heat or cold.
Incompatible Materials: Do not mix with oxidizers, acids, bathroom cleaners, or disinfecting agents.
Hazardous Decomposition Products: Normal products of combustion - CO, CO2.

Section 11: TOXICOLOGICAL INFORMATION

Likely Routes of Exposure:
Inhalation - Overexposure may cause headache.
Skin Contact - Not expected to cause irritation, repeated contact may cause dry skin.
Eye Contact - Not expected to cause irritation.
Ingestion - May cause upset stomach.

Symptoms related to the physical, chemical and toxicological characteristics: no symptoms expected under typical use conditions.
Delayed and immediate effects and or chronic effects from short term exposure: no symptoms expected under typical use conditions.
Delayed and immediate effects and or chronic effects from long term exposure: headache, dry skin, or skin irritation may occur.
Interactive effects: Not known.

Numerical Measures of Toxicity

Acute Toxicity:
Oral LD_{50} (rat) > 5 g/kg body weight
Dermal LD_{50} (rabbit) > 5 g/kg body weight
Calculated via OSHA HCS 2012 / Globally Harmonized System of Classification and Labelling of Chemicals

Skin Corrosion/Irritation: Non-irritant per Dermal Irritection® assay modeling. No animal testing performed.
Eye Damage/Irritation: Non/Minimal irritant per Ocular Irritection® assay modeling. No animal testing performed.
Germ Cell Mutagenicity: Mixture does not classify under this category.
Carcinogenicity: Mixture does not classify under this category.
Reproductive Toxicity: Mixture does not classify under this category.
STOT-Single Exposure: Mixture does not classify under this category.
STOT-Repeated Exposure: Mixture does not classify under this category.
Aspiration Hazard: Mixture does not classify under this category.

Section 12: ECOLOGICAL INFORMATION

Ecotoxicity: Volume of ingredients used does not trigger toxicity classifications under the Globally Harmonized System of Classification and Labelling of Chemicals.
Aquatic: Aquatic Toxicity - Low, based on OECD 201, 202, 203 + Microtox: EC_{50} & IC_{50} ≥100 mg/L. Volume of ingredients used does not trigger toxicity classifications under the Globally Harmonized System of Classification and Labelling of Chemicals.
Terrestrial: Not tested on finished formulation.

Persistence and Degradability: Readily Biodegradable per OCED 301D, Closed Bottle Test. Reaches 100% biodegradability within 1 year or less.
Bioaccumulative Potential: No data available.
Mobility in Soil: No data available.
Other Adverse Effects: No data available.

Section 13: DISPOSAL CONSIDERATIONS

Unused or Used Liquid: May be considered hazardous in your area depending on usage and tonnage of disposal – check with local, regional, and or national regulations for appropriate methods of disposal.

Empty Containers: May be offered for recycling.

Never dispose of used degreasing rinsates into lakes, streams, and open bodies of water or storm drains.

Section 14: TRANSPORT INFORMATION

U.N. Number: Not applicable
U.N. Proper Shipping Name: Cleaning Compound, Liquid NOI
Transport Hazard Class(es): Not applicable
Packing Group: Not applicable
Environmental Hazards: Marine Pollutant - NO
Transport in Bulk (according to Annex II of MARPOL 73/78 and IBC Code): Unknown.
Special precautions which user needs to be aware of/comply with, in connection with transport or conveyance either within or outside their premises: None known.

U.S. (DOT) / Canadian TDG: Not Regulated for shipping.
IMO / IDMG: Not classified as Hazardous
ICAO/ IATA: Not classified as Hazardous
ADR/RID: Not classified as Hazardous

Section 15: REGULATORY INFORMATION

All components are listed on: TSCA and DSL Inventory.

SARA Title III: Sections 311/312 Hazard Categories – Not applicable.
Sections 313 Superfunds Amendments and Reauthorizations Act of 1986 – Not applicable.
Sections 302 – Not applicable.

Clean Air Act (CAA): Not applicable
Clean Water Act (CWA): Not applicable

State Right To Know Lists: No ingredients listed
California Proposition 65: No ingredients listed
Texas ESL:

Ethoxylated Alcohol	68439-46-3	60 µg/m³ long term	600 µg/m³ short term
Sodium Citrate	68-04-2	5 µg/m³ long term	50 µg/m³ short term
Sodium Carbonate	497-19-8	5 µg/m³ long term	50 µg/m³ short term
Citric Acid	77-92-9	10 µg/m³ long term	100 µg/m³ short term

This product has been classified as "not classifiable as hazardous" in accordance with Consumer Product Safety Commission (16 CFR Chapter 2), and labelled and packaged accordingly.

Section 16: OTHER INFORMATION

Size	UPC	Size	UPC
2 fl. oz.	043318131035	67.6 fl. oz.	043318130144
4 fl. oz.	043318130014	67.6 fl. oz.	043318000393
16 fl. oz.	043318130021	1 gallon	043318000799
22 fl. oz.	043318130229	1 gallon	043318130052
24 fl. oz.	043318130137	1 gallon	043318004957
32 fl. oz.	043318002557	1 gallon w/ dilution bottle	043318480492
32 fl. oz.	043318130335	140 fl. oz. w/ dilution bottle	043318001468
32 fl. oz.	043318000652	2.5 gallon	043318004889

USA items listed only. Not all items listed. USA items may not be valid for international sale.

Section 16: OTHER INFORMATION - continued

NFPA:

Health – None
Flammability – Non-flammable
Stability – Stable
Special - None

Acronyms

NTP	National Toxicology Program	IARC	International Agency for Research on Cancer
OSHA	Occupational Safety and Health Administration	CPSC	Consumer Product Safety Commission
TSCA	Toxic Substances Control Act	DSL	Domestic Substances List

Prepared / Revised By: Sunshine Makers, Inc., Regulatory Department.
This SDS has been revised in the following sections: Clarification on hazards in section 2, expanded transparency in section 3, revised layout in section 9, 14 & 16, added statement in section 15.

DISCLAIMER: 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.

APPENDIX C

COVID-19 Interim Health and Safety Guidance

COVID-19 INTERIM HEALTH AND SAFETY GUIDANCE

CORPORATE HEALTH AND SAFETY MANAGER	:	**Brian Hobbs, CIH, CSP**
EFFECTIVE DATE	:	**03/2020**
REVISION DATE	:	**09/21/2022**
REVISION NUMBER	:	**9**

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1. PURPOSE

This guidance has been implemented to establish work practices, administrative procedures, and engineering controls to minimize potential exposure to SARS-CoV-2, the virus that causes COVID-19. The following guidance has been developed based on local, state and federal recommendations/requirements regarding COVID-19. The purpose of this document is to supplement existing site-specific Health and Safety Plans (HASPs) and provide interim health and safety guidance to minimize potential exposure to SARS-CoV-2. Should additional scientific information or regulatory information change, this document shall be updated accordingly.

2. SCOPE AND APPLICABILITY

This guidance covers all Roux employees and the subcontractors that Roux oversees. Site specific HASPs shall be developed to incorporate elements of mitigative measures against COVID-19 exposure. If work cannot be carried out in compliance with this guidance, the project shall be further evaluated by the Project Principal (PP), Office Manager (OM), and Corporate Health and Safety Director (CHSD) prior to work authorization.

Roux subcontractors are required to review, comply with, and implement Roux's COVID-19 Interim Health and Safety Guidance while on Site. Subcontractors may implement additional preventative measures as they see fit. All work shall be conducted in a manner consistent with the federal, state, and local guidance as it relates to COVID-19.

3. BACKGROUND

What is COVID-19?

COVID-19 is a respiratory disease caused by SARS-CoV-2, a coronavirus discovered in 2019. The virus spreads mainly from person to person through respiratory droplets produced when an infected person coughs, sneezes, or talks. Some people who are infected may not have symptoms. Multiple variants of the virus that causes COVID-19 are circulating globally. There are currently several vaccines which have been developed which are authorized, recommended and effective at protecting you from getting sick.

What are the symptoms of COVID-19?

Reported illnesses have ranged from mild symptoms to severe illness and death for confirmed COVID-19 cases. Symptoms may appear 2 to 14 days following exposure to the virus. People with these symptoms or combinations of symptoms may have COVID-19:

- Fever or chills
- Cough
- Shortness of breath or difficulty breathing
- Fatigue
- Muscle or body aches
- Headache
- New loss of taste or smell
- Sore throat
- Congestion or runny nose
- Nausea or vomiting
- Diarrhea

This list is not all possible symptoms. The CDC will continue to update this list as they learn more about the virus. For an updated symptom list please reference the following link for CDC Symptoms of Coronavirus.

If someone develops emergency warning signs for COVID-19, they should be instructed to get medical attention immediately. Emergency warning signs can include those listed below; however, this list is not all inclusive. Please consult your medical provider for any other symptoms that are severe or concerning.

- Trouble breathing
- Persistent pain or pressure in the chest
- New confusion
- Inability to wake or stay awake
- Pale, gray, or blue-colored skin, lips, or nail beds, depending on skin tone

How does COVID-19 spread?[1]

Individuals who are within close contact (within 6 feet) of a person with COVID-19 or have direct contact with that person are at greatest risk of infection.

COVID-19 spreads in three main ways:

- Breathing in air when close to an infected person who is exhaling small droplets and particles that contain the virus.
- Having these small droplets and particles that contain virus land on the eyes, nose, or mouth, especially through splashes and sprays like a cough or sneeze.
- Touching eyes, nose, or mouth with hands that have the virus on them.

Transmission of SARS-CoV-2 from inhalation of virus in air farther than six feet from an infectious source can occur.

Some infections can be spread by exposure to virus in small droplets and particles that can linger in the air for minutes to hours. These viruses may be able to infect people who are further than 6 feet away from the person who is infected or after that person has left the space. This kind of spread is referred to as **airborne transmission** and is an important way that infections like tuberculosis, measles, and chicken pox are spread. Per published reports, factors that increase the risk of SARS-CoV-2 infection under these circumstances include:

- Enclosed spaces with inadequate ventilation or air handling within which the concentration of exhaled respiratory fluids, especially very fine droplets and aerosol particles, can build-up in the air space.
- Increased exhalation of respiratory fluids if the infectious person is engaged in physical exertion or raises their voice (e.g., exercising, shouting, singing).
- Prolonged exposure to these conditions, typically more than 15 minutes.

Spread from contact with contaminated surfaces or objects is less common.

Respiratory droplets can also land on surfaces and objects. It is possible that a person could get COVID-19 by touching a surface or object that has the virus on it and then touching their own mouth, nose, or eyes. Spread from touching surfaces is not thought to be a common way that COVID-19 spreads.

4. TRAINING REQUIREMENTS

All employees with potential exposure to COVID-19 shall be provided training that incorporates COVID-19 exposure mitigation strategies, such as implementation of proper social distancing, personal hygiene (e.g., handwashing), as well as disinfection procedures, as outlined by CDC guidelines.

5. EXPOSURE RISK POTENTIAL

Worker risk of occupational exposure to COVID-19 can vary from very high, high, medium, or lower (caution) risk. This level of exposure is dependent on several factors, which can include industry type; need for contact within 6 feet of people known to be or suspected of being infected with COVID-19; density of work environment; and industrial setting (i.e., healthcare building, occupied interior work area, minimal ventilation).

Provided below is background risk level information taken from the U.S. Department of Labor Occupational Safety and Health Administration Guidance on preparing workplaces for COVID-19. Risk evaluations for each project shall be conducted by the PP and OM in consultation with the CHSD to ensure Roux employees and subcontractors remain within the lower exposure (caution) category. If it is identified there is a medium exposure risk or higher, further evaluation and mitigative measures shall be evaluated to reduce overall exposure risk prior to work authorization.

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1 How COVID-19 Spreads https://www.cdc.gov/coronavirus/2019-ncov/prevent-getting-sick/how-covid-spreads.html#edn1

Very High Exposure Risk (Activities not conducted by Roux)

Very high exposure risk includes occupations/work activities with high potential for exposure to known or suspected sources of COVID-19 during specific medical, postmortem, or laboratory procedures. This can include but is not limited to:

- Healthcare workers (e.g., doctors, nurses, dentists, paramedics, emergency medical technicians) performing aerosol-generating procedures (e.g., intubation, cough induction procedures, bronchoscopies, some dental procedures and exams, or invasive specimen collection) on known or suspected COVID-19 patients.
- Healthcare or laboratory personnel collecting or handling specimens from known or suspected COVID-19 patients (e.g., manipulating cultures from known or suspected COVID-19 patients).
- Morgue workers performing autopsies, which generally involve aerosol-generating procedures on the bodies of people who are known to have, or suspected of having, COVID-19 at the time of their death.

High Exposure Risk (Activities not conducted by Roux)

High exposure risk occupations/work activities include exposure to known or suspected COVID-19 positive individuals. This can include but not limited to:

- Healthcare delivery and support staff (hospital staff who must enter patients' rooms) exposed to known or suspected COVID-19 patients.
- Medical transport workers (ambulance vehicle operators) moving known or suspected COVID-19 patients in enclosed vehicles.
- Mortuary workers involved in preparing bodies for burial or cremation of people known to have, or suspected of having, COVID-19 at the time of death.
- Those who have frequent or sustained contact with coworkers, including under close working conditions indoors or in poorly ventilated spaces in various types of industrial, manufacturing, agriculture, construction, and other critical infrastructure workplaces.
- Those who have frequent indoor or poorly ventilated contact with the general public, including workers in retail stores, grocery stores or supermarkets, pharmacies, transit and transportation operations, law enforcement and emergency response operations, restaurants, and bars.

Medium Exposure Risk

Medium exposure risk occupations/work activities include those that require frequent and/or close contact with (i.e., within 6 feet for a cumulative total of 15 minutes or more over a 24-hour period)) people who may be infected with COVID-19, but who are not known or suspected to be COVID-19 positive. For most of our worksites, it is assumed there is on-going community transmission for COVID-19. Therefore, workers who work at sites and may have contact with the general public, other contractors, high-population-density work environments (i.e., greater than 10 people) fall within medium exposure risk group category. This can include, but is not limited to, sampling events that require two or more workers to collect and log samples in close contact or work occurring in an interior space with limited ventilation and several workers present.

Lower Exposure Risk (Caution)

Lower exposure risk (caution) occupations/work activities are those that do not require close contact (within 6 feet for a cumulative total of 15 minutes or more over a 24-hour period) with other people. During these activities, there is limited contact (i.e., within 6 feet of) the general public or other workers. Workers in this category have minimal occupational contact with the public and other coworkers. This includes construction oversight that does not require close contact, sampling or gauging events performed by one worker and our remote workers as well as office workers who do not have frequent close contact with coworkers, clients, or the public.

6. CDC FULLY VACCINATED GUIDANCE

You are up to date with your COVID-19 vaccines if you have completed a COVID-19 vaccine primary series and received the most recent booster dose recommended for you by CDC. Additional information concerning vaccinations can be found at the following link.

7. COVID-19 HEALTH SCREENING

7.1 Roux Employees

Depending on local/state/client requirements, Roux employees may self-attest to a COVID-19 Daily Health Questionnaire that is to be completed at home through a mobile application on scheduled workdays. The purpose of this program is to ensure business continuity as well as mitigate any potential exposure to our employees and others if it is determined employees are at-risk for contracting COVID-19. As part of this self-attestation, all employees are required to take their temperatures daily at home to confirm they do not have a fever (≥ 100.4). Employees who answer yes to any of these questions are instructed to contact their Office Manager and/or Department Head immediately and should not enter the office or go to a field site. Information shall be used to determine appropriate internal response in consultation with the Human Resources Director (HRD) and CHSD.

Below, you will find our COVID-19 Daily Health Questionnaire that all Roux employees are required to self-attest to **every scheduled workday by 9:30 AM.** If employees do not promptly fill out the questionnaire by the time listed above, there will be additional follow up by HR, H&S, and/or OMs.

According to the U.S. Centers for Disease Control and Prevention & the World Health Organization, COVID-19 Symptoms include:

- *Fever (≥100. 4°F) or chills*
- *Cough*
- *Shortness of breath or difficulty breathing*
- *Fatigue*
- *Muscle or body aches*
- *Headache*
- *New loss of taste or smell*
- *Sore throat*
- *Congestion or runny nose*
- *Nausea or vomiting*
- *Diarrhea*

Have you experienced any of the COVID-19 related symptoms noted above in the last 14 days? Please Note: We do not expect employees to answer "yes" to the symptoms question if these are symptoms you normally experience due to another condition or medication.

- *Yes*
- *No*

Have you been in close contact with someone who is suspected or confirmed to have COVID-19 or who is under investigation for COVID-19 within the last 14 days? * Close contact as defined by the CDC is being within 6 feet of someone who has COVID-19 for a cumulative total of 15 minutes or more over a 24-hour period.*

- *Yes*
- *No*

Have you traveled outside of the country, been on a cruise ship and/or traveled to areas within the United States which have state mandated travel restrictions in the last 14 days?

- *Yes*
- *No*

Have you tested positive for COVID-19 within the last 14 days?

- *Yes*
- *No*

7.2 Subcontractors

Depending on local/state/client requirements, Subcontractors who shall perform work onsite may be required to attest to the fitness of their work crew on a daily basis. This requires each worker to self-assess by asking themselves the four questions listed in the section above and also contained within the Roux Subcontractor Work Crew COVID-19 Daily Health Attestation. If any crew member answers "Yes" to any of the questions, that worker is not to report to the field site and should seek proper medical advice in accordance with local, state and federal guidelines. In addition if required by local/state/client requirements, the Sub-Contractor shall self-attest to vaccination status in order for the Field Team to ensure conformance with updated guidance for fully vaccinated individuals should state/local/client requirements allow. See Section 6. CDC Fully Vaccinated Guidance.

On a daily basis, the subcontractor supervisor must provide the Subcontractor Work Crew COVID-19 Daily Health Attestation complete with the names of all work crew fit to be on the Site for that day (i.e., who have answered "No" to all questions on the self-assessment) to Roux's Project Manager or Site Supervisor. The Subcontractor must notify Roux if there have been any "Yes" responses daily. Subcontractors shall not be required to provide the name or any other personal information of any employee who has answered "Yes" to any of the self-assessment questions, however, the Subcontractor should provide the date and times that the employee has been onsite in the prior 14 days. Records shall be maintained within the project files indicating health screening has been performed, records shall be retained for not less than 14 days following the date of submission. The Roux Subcontractor Work Crew COVID-19 Daily Health Check Attestation can be found within Appendix A.

8. SELF-ISOLATION & QUARANTINE

8.1 Self-Isolation

What if I am asked to self-isolate at home and when can I return from home isolation?

Depending on the situation, if you are COVID-19 positive or suspected to have COVID-19, employees may be required to self-isolate in their homes, as per CDC or local health department guidelines. Roux shall follow CDC guidance in areas where local/state requirements allow. The following table below outlines CDC isolation guidance.

Day 0 is your first day of symptoms or a positive viral test. Day 1 is the first full day after your symptoms developed or your test specimen was collected. If you have COVID-19 or have symptoms, isolate for at least 5 days.

IF YOU Tested positive for COVID-19 or have symptoms, regardless of vaccination status:	**Stay home for at least 5 days** Stay home for 5 days and isolate from others in your home. Wear a well-fitted mask if you must be around others in your home.	**Ending isolation if you had symptoms** End isolation after 5 full days if you are fever-free for 24 hours (without the use of fever-reducing medication) and your symptoms are improving. **Ending isolation if you did NOT have symptoms** End isolation after at least 5 full days after your positive test. **If you were severely ill with COVID-19** You should isolate for at least 10 days. Consult your doctor before ending isolation.	**Take precautions until Day 10** **Wear a mask** Wear a well-fitted mask for 10 full days any time you are around others inside your home or in public. Do not go to places where you are unable to wear a mask. **Avoid travel** **Avoid being around people who are at high risk**

8.2 Quarantine

Employees may be required to self-quarantine due to potential exposure with a suspected and/or confirmed COVID-19 positive individual as well as recent travel as per local/state guidelines. Consult with your OM regarding whether your situation requires quarantine following close contact. People in quarantine should stay home, separate themselves from others, monitor their health, and follow directions from their state or local health department.

8.2.1 Travel Related Quarantine/Testing

All travel out of state must be communicated with the OM and/or Department Head prior to departure. Please note, some federal/state/local entities require submissions of traveler health forms and potentially require additional testing for COVID-19. It is expected all Roux employees will comply with such federal/state/local travel requirements.

9. WORKPLACE CONTROLS

During the project planning phase, worksite evaluations shall be carried out by the PP and OM in consultation with the CHSD to determine risk exposure levels for work activities. If it is determined there is a high exposure risk level or higher, additional workplace controls shall be evaluated and implemented as required in addition to the basic infection prevention measures outlined below in Section 10. Additional workplace controls can include engineering controls (i.e., ventilation, physical barriers), administrative controls (i.e., minimizing contact between workers, rotating shifts, site specific training), and additional personal protective equipment (i.e., respiratory protection). If exposure risk cannot be mitigated, potential project postponement may be necessary at the discretion of the OM in consultation with the CHSD.

A Job Safety Analysis (JSA) has been developed and is provided in Appendix B, which summarizes and applies concepts within this guidance, including the infection prevention measures listed below. This JSA shall be required for all fieldwork in areas where there is community-based transmission of COVID-19.

10. INFECTION PREVENTION MEASURES

The following is basic infection prevention and personal hygiene practices which shall be implemented for all Roux field activities as well as in the office setting.

- ***Personal Hygiene***
  - Wash your hands often with soap and water for at least 20 seconds.
    - If soap and water are not available, use an alcohol-based sanitizer that contains at least 60% alcohol.
    - Key times to wash your hands include after blowing your nose, coughing or sneezing, after using the restroom, and before eating or preparing food.
  - Do not touch your eyes, face, nose and mouth with unwashed hands.
  - Cover your mouth and nose with a tissue when you cough or sneeze or use the inside of your elbow.
  - Throw potentially contaminated items (e.g., used tissues) in the trash.
- ***Avoid Close Contact/Secondary Contact with People and Potentially Contaminated Surfaces***
  - Apply appropriate social distance (6+ feet), as appropriate.
  - Do not work in areas with limited ventilation with other Site workers (e.g., small work trailer which lacks HVAC system).
  - Morning tailgate/safety meetings are recommended to occur outside or in well ventilated work trailers.
  - Contact your lab/equipment vendor to confirm equipment is properly disinfected prior to being shipped.
  - Do not carpool with others unless all individuals are comfortable with traveling together.
  - For company owned vehicles limit sharing of vehicles with coworkers. If unable to limit sharing of company owned vehicles, properly clean vehicle before driving with a focus on commonly touched surfaces (e.g., steering wheels, shifters, buttons, etc.).
  - Use caution when using public restrooms, portable toilets. Use paper towel as a barrier when touching door handles and faucets.
- ***Cleaning and Disinfecting***
  - Clean high touched surfaces daily. Examples of high-touch surfaces include: counters, tables, doorknobs, handles, stair rails, desks, toilets, faucets, and sinks. In most situations, regular cleaning (at least once a day) is enough to sufficiently remove virus that may be on surfaces. However, if certain conditions apply, you may choose to disinfect after cleaning. When there is no confirmed or suspected COVID-19 cases known to have been in a space, cleaning once a day is usually enough to sufficiently remove virus that may be on surfaces and help maintain a healthy facility.
  - You may want to either clean more frequently or choose to disinfect in addition to cleaning in shared spaces if the space:
    - Is a high traffic area, with a large number of people,
    - Is poorly ventilated,
    - Does not provide access to handwashing or hand sanitizer, or
    - The space is occupied by individuals at increased risk for severe illness.

If a someone who tested or is presumed COVID-19 positive and has been in your facility within the last 24 hours, you should clean and disinfect the space. This will be done in consultation with the CHSD.

The following outlines cleaning and disinfection protocols for specific types of surfaces as required. Please consult with the CHSD when developing site-specific cleaning and disinfection protocols.

- ***Hard (Non-porous) Surfaces***
  - If surfaces are dirty, they should be cleaned with a detergent/soap and water prior to disinfection.
  - Refer to the manufacturer's instructions to ensure safe and effective use of the product and wear appropriate personal protective equipment (e.g., gloves, safety glasses, face shield).
  - Many products require:
    - Keeping surface wet for a period of time (i.e., contact time).
      - Refer to manufacturer's instructions outlining adequate contact time.
    - Precautions such as wearing gloves and making sure you have good ventilation during use of the product.
  - Disposable gloves should be removed aseptically and discarded after cleaning. Wash hands immediately following removal of gloves. Refer to Appendix C for how to remove gloves aseptically.
  - If products on EPA List N: Disinfectants for Coronavirus (COVID-19) are not available, bleach solutions can be used if appropriate for the surface and will be effective against coronaviruses when properly diluted.
    - Most household bleach contains 5%–9% sodium hypochlorite. Do not use a bleach product if the percentage is not in this range or is not specified, such as some types of laundry bleach or splash-less bleach as these are not appropriate for disinfection.
    - Follow the directions on the bleach bottle for preparing a diluted bleach solution. If your bottle does not have directions, you can make a bleach solution for disinfecting by mixing:
      - 5 tablespoons (1/3 cup) of bleach per gallon of room temperature water; OR
      - 4 teaspoons of bleach per quart of room temperature water.
    - Follow the manufacturer's application instructions for the surface. If instructions are not available, leave the diluted bleach solution on the surface for at least 1 minute before removing or wiping. This is known as the "contact time" for disinfection. The surface should remain visibly wet during the contact time.
    - Ensure proper ventilation during and after application (for example, open windows).
    - Never mix household bleach (or any disinfectants) with any other cleaners or disinfectants. This can cause vapors that may be very dangerous to breathe in.
    - Make a new diluted bleach solution daily. Bleach solutions will not be as effective after being mixed with water for over 24 hours. Products with EPA-approved emerging viral pathogen claims are expected to be effective against COVID-19. Follow the manufacturer's instructions for all cleaning and disinfecting products (e.g., concentration, application method and contact time, etc.).
- ***Soft (Porous) Surfaces***
  - For soft (porous) surfaces, remove visible contamination if present and clean with appropriate cleaners indicated for use on the surfaces. After cleaning:
    - Launder items as appropriate in accordance with the manufacturer's instructions. If possible, launder using the warmest appropriate water setting for the item and dry items completely; or
    - Use products with the EPA-approved emerging viral pathogens that claim they are suitable for porous surfaces.

- ***Electronics***
  - For electronics such as tablets, touch screens, keyboards, remote controls, etc. remove visible contamination if present.
    - Follow the manufacturer's instructions for all cleaning and disinfection products.
    - Consider use of wipeable covers for electronics.
    - If no manufacturer guidance is available, consider the use of alcohol-based wipes or sprays containing at least 70% alcohol to disinfect touch screens. Dry surfaces thoroughly to avoid pooling of liquids.
- ***Linens, Clothing, and Other Items that Go in the Laundry***
  - Although it is unlikely field clothing would become potentially contaminated with COVID-19, it is recommended that field staff regularly launder field clothing following any field event upon returning home.
  - In order to minimize the possibility of dispersing the virus from potentially contaminated clothing, do not shake dirty laundry.
  - Wash items as appropriate in accordance with the manufacturer's instructions. If possible, launder items using the warmest appropriate water setting for the items and dry items completely.
  - Clean and disinfect hampers or other containers used for transporting laundry according to guidance listed above.
- ***Office/Site Specific-Cleaning and Disinfection Protocols***
  - Each office and long-term field site shall develop internal cleaning and disinfecting practices, which can be broken into three categories: routine cleaning; enhanced cleaning and disinfecting; and deep cleaning and disinfecting.
  - In the instance there is someone who is suspected or confirmed positive for COVID-19 and has worked at the office or field site within the last 24 hours, deep cleaning and disinfecting shall be considered. The CHSD shall work with the OM and Office Health and Safety Manager (OHSM) to evaluate site-specific measures that shall be carried out prior to deep cleaning and disinfecting. If more than 24 hours have passed since the person who is sick or diagnosed with COVID-19 has been in the space, cleaning shall be carried out. You may choose to also disinfect depending on certain conditions and in consultation with the CHSD.
  - If deep cleaning and disinfection is carried out the following will be considered:
    - Closing off all areas potentially affected and wait at least several hours before you clean and disinfect.
    - Areas should remain closed off until cleaning and disinfecting takes place; if able, ventilation shall be increased in the space (e.g., opening doors, windows, increasing CFM).

11. FACE COVERINGS

The CDC recommends the use of face coverings/masks in public settings where other social distancing measures are difficult to maintain. Masks are required on planes, buses, trains and other forms of public transportation traveling into, within, or out of the United States and in U.S. indoor transportation hubs such as airports and stations. The use of face coverings is to supplement and NOT replace the existing practices outlined above.

Based on existing studies and on-going recommendations and/or requirements from federal, state, and local entities, Roux is recommending the use of face coverings, when appropriate. Appropriate use is defined when local authorities or clients require the use of face coverings in conjunction with established social distancing, or if an employee elects to use a cloth covering on their own accord. Roux will provide appropriate face coverings that shall meet the basic requirements outlined by the CDC guidance.

Face Coverings (i.e., masks) should:

- Have two or more layers;
- Completely cover the nose and mouth;
- Fit snugly against the sides of the face and not have any gaps; and
- Have a nose wire to prevent air from leaking out of the top of the mask.

When donning and doffing the face covering, individuals should avoid touching their eyes, nose, and mouth. Following removal of the face covering, employees should wash their hands immediately using the guidelines described in Section 10 Infection Prevention Measures-Personal Hygiene above. Face coverings should be routinely washed depending on the frequency of use.

APPENDIX A

Roux Subcontractor Work Crew
COVID-19 Daily Health Screening Questionnaire

Subcontractor Work Crew
COVID-19 Daily Health Attestation

Date:
Company Name:
Supervisor Name: Signature:
Project Name:
Site Address:
Number of Workers on site:

Prior to entry onto a field site, the following questions shall be asked by the Subcontractor Supervisor to their work crew. **Subcontractors and Field Teams shall self-attest to vaccination status in order to ensure compliance with state/local guidance for fully vaccinated and unvaccinated individuals.**

It is preferred this questionnaire is completed for each individual prior to their arrival at the field site. If the answer to any of these questions is YES, the worker is not to report to the field site and seek proper medical advice, in accordance with CDC Guidelines. **The Subcontractor Supervisor must provide this form on a daily basis to the Roux primary contact for the project and notify Roux of any YES responses.**

1. Have you experienced any signs/symptoms of COVID-19 such as fever (≥100.4°F), cough, shortness of breath, chills, fatigue, muscle/body aches, headache, new loss of taste or smell, sore throat, congestion or runny nose, nausea/vomiting or diarrhea in the last 5 days?

2. Have you been in close contact* with someone who is suspected or confirmed to have COVID-19 or who is under investigation for COVID-19 within the last 5 days?

*Close contact as defined by the CDC is being within 6 feet of someone who has COVID-19 for a cumulative total of 15 minutes or more over a 24-hour period. Those who are up to date on COVID-19 vaccinations or had confirmed COVID-19 within the past 90 days (you tested positive using a viral test) you do not need to quarantine.

3. Have you traveled outside of the country, been on a cruise ship and/or traveled to areas within the United States which have state mandated travel restrictions in the last 5 days?

4. Have you tested positive for COVID-19 within the last 5 days?

Please list the crew member's names on site for the day.

1.	8.
2.	9.
3.	10.
4.	11.
5.	12.
6.	13.
7.	14.

APPENDIX B

Job Safety Analysis-Working in Areas Affected by COVID-19

JOB SAFETY ANALYSIS	Ctrl. No. CVD-19	DATE: 01/10/2022	☐ NEW ☒ REVISED	PAGE 1 of 2

JSA TYPE CATEGORY	WORK TYPE	WORK ACTIVITY (Description)
Generic	**Fieldwork**	**Working in Areas Affected by Coronavirus**

DEVELOPMENT TEAM	POSITION / TITLE	REVIEWED BY:	POSITION / TITLE
Kristina DeLuca	Health and Safety Specialist	Brian Hobbs	CHSD

REQUIRED AND / OR RECOMMENDED PERSONAL PROTECTIVE EQUIPMENT

☐ LIFE VEST	☐ GOGGLES	☐ AIR PURIFYING RESPIRATOR	☒ GLOVES – Leather/cut-resistant in field and nitrile as needed
☒ HARD HAT – In field	☐ FACE SHIELD	☐ SUPPLIED RESPIRATOR	
☐ LIFELINE / BODY HARNESS	☐ HEARING PROTECTION	☒ PPE CLOTHING – High visibility vest in field	☐ OTHER
☒ SAFETY GLASSES – In field	☒ SAFETY SHOES – Steel/composite toe in fie		

REQUIRED AND / OR RECOMMENDED EQUIPMENT

Face covering/mask, nitrile gloves, hand soap, water source, hand sanitizer, disinfectant spray and disinfectant wipes.

Commitment to Safety – All personnel onsite will actively participate in SPSA performance by verbalizing SPSAs throughout the day.

SOCIAL DISTANCING: Maintain 6' of distance between yourself and all other people at all times. If you do not believe the scope of work can be conducted while maintaining this distance, contact your Project Manager immediately.

Assess [1]JOB STEPS	Analyze [2]POTENTIAL HAZARDS	Act [3]CRITICAL ACTIONS
1. Project Preplanning	N/A	• Review and follow COVID-19 CDC, Roux, Client and local orders/protocols. • Ensure all workers are fit for duty - anyone feeling sick should remain at home even if symptoms do not align with COVID-19. If a worker has been in contact with someone potentially positive or positive for COVID-19, contact your Office Manager. • Determine PPE needs and ensure adequate supply of disinfectant wipes/spray, soap and water or hand sanitizer at Site. Due to high demands and limited supply, plan ahead. • Use the minimum number of employees necessary to safely complete the work.
2. Mobilization	**Exposure:** Becoming infected or infecting co-workers	**Personal/Rental/Roux Owned Vehicle** • Avoid carpooling, unless all individuals are up to date on vaccinations. • Verify workers/other people are not approaching vehicle prior to exiting the vehicle. Maintain 6' of distance from general public, as appropriate. **Public Transportation** • Public transit should not be used unless absolutely necessary. Consider renting a car rather than taking public transit. If public transit is required, wear appropriate face covering/mask and apply social distancing (6 ft). Wash hands or use hand sanitizer immediately after. **Hotel Stay (Refer to COVID-19 H&S Guidance for more info)** • If a hotel stay is deemed necessary for the given field work, ensure that you clean your room upon initial arrival. • Place the "Do Not Disturb" placard on the room while away and limit housekeeping services to the extent feasible during your stay to minimize the reintroduction and spread of the virus from others.. Wash hands or use hand sanitizer often.
3. Tailgate Meeting	**Exposure:** Becoming infected or infecting co-workers	• Perform outside or indoors in areas with ample ventilation. • If unvaccinated, maintain at least a 6+ ft distance between you and others. • Discuss primary infection prevention measures listed below. • Discuss COVID-19 symptoms with coworkers and subcontractors to ensure fitness for duty. Anyone exhibiting signs or symptoms should be instructed to leave the Site, contact your Project Manager.

[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; 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".

4. Site Activities	**Exposure:** Becoming infected or infecting co-workers	• Coordinate field activities at the beginning of the day (i.e. Tailgate meeting) to minimize time spent in crowded spaces or overlap while completing job tasks. • Don face coverings as appropriate. • Apply social distancing (6+ ft) when interacting with others if unvaccinated. If anyone comes within 6 ft of you while conducting work and your work prevents you from moving away, politely ask them to move back. If others are unable to move from your space, stop work and leave area. • Minimize shaking hands or touching others. • Minimize sharing of equipment or other items with co-workers and subcontractors unless wearing appropriate PPE (e.g. nitrile gloves), as appropriate. • If anyone is experiencing COVID-19 signs or symptoms in your vicinity, stop work and leave the area. • Do not work in areas with limited ventilation with others. • Cover your mouth and nose with tissue or paper towel or with your elbow when coughing or sneezing and wash hands or use hand sanitizer immediately after. If sick contact SHSO/PM and leave Site immediately. • Clean work surfaces/areas with approved cleaners you're responsible for (ex: desk, office doorknob, computer, etc.) at least daily. • Avoid public spaces and going out to eat by bringing your own lunch to the Site. If performing work in high density urban areas, it is recommended all food must be consumed at or in your vehicle or within designated work trailer. Wash hands or use hand sanitizer before eating and immediately after.

Primary Infection Prevention Measures

- Wash your hands often with soap and water for at least 20 seconds.
  - If soap and water are not available, use an alcohol-based sanitizer that contains at least 60% alcohol. Key times to wash hands include after blowing your nose, coughing or sneezing, after using the restroom, and before eating or preparing food.
- Do not touch your eyes, face, nose and mouth with unwashed hands.
- Cover your mouth and nose with a tissue when you cough or sneeze or use the inside of your elbow. Throw potentially contaminated items (e.g. used tissues) in the trash.
- Avoid close contact/secondary contact with people and potentially contaminated surfaces.
  - Apply appropriate social distance (6+ feet).
  - Minimize handshaking/touching others and use caution when accessing public spaces.
- Clean frequently touched surfaces daily. Commonly touched items can include but are not limited to tables, doorknobs, light switches, countertops, handles, desks, phones, keyboard, toilets, sinks and field equipment. If surfaces are dirty, they should be cleaned with soap and water prior to disinfection. If surface cannot be cleaned/disinfected, then wash hands or use sanitizer as soon as possible.

[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".

APPENDIX C

How to Remove Gloves

How to Remove Gloves

To protect yourself, use the following steps to take off gloves

1

Grasp the outside of one glove at the wrist. Do not touch your bare skin.

2

Peel the glove away from your body, pulling it inside out.

3

Hold the glove you just removed in your gloved hand.

4

Peel off the second glove by putting your fingers inside the glove at the top of your wrist.

5

Turn the second glove inside out while pulling it away from your body, leaving the first glove inside the second.

6

Dispose of the gloves safely. Do not reuse the gloves.

7

Clean your hands immediately after removing gloves.

APPENDIX D

Heat Illness Prevention Program

HEAT ILLNESS
PREVENTION PROGRAM

CORPORATE HEALTH AND SAFETY DIRECTOR	:	**Brian Hobbs, CIH, CSP**
EFFECTIVE DATE	:	**10/2019**
REVISION DATE	:	**1/2023**
REVISION NUMBER	:	**2**

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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 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 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) and Office Health and Safety Manager (OHSM). The CHSD has the authority and 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 regards 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 field work 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 which can increase heat stress can 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 which produce heat (e.g., welding)
- Physical contact with warm or hot objects or liquids
- The clothing and PPE employees are required to wear
- 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 which rotate workers to limit employee exposure. Typically, the Site Supervisor (SS) or Site Health and Safety Officer (SHSO) shall conduct worksite observations to assure 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.
  - Heat wave 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 available and cool. Water containers (and all spouts and levers) must be kept clean. If able provide single use drinking cups with appropriate waste receptacle. Accessible sanitation facilities shall also be maintained at work Sites as appropriate.

SS/SHSO are 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 choosing 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
  - 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

Shade shall be strong enough to cool employees down and other shadows should not be visible in the shade. 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 setup 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 sections below.

8. 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.

9. HEAT WAVE 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 assured through the use of periodic check ins with the SS/SHSO via phone. 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.

10. 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 are 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.

11. 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, work trailers). Supervisors, prior to supervising personnel in the field, as well as all personnel involved with the field work of a project, are trained in this HASP which 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.

11. 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 ones 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 E

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	:	**01/2023**
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") has 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 evaluated on a case-by-case basis to insure its adequacy, maintenance and sanitation.

2. SCOPE AND APPLICABILITY

These guidelines apply to all PPE selection decisions to be made in implementing 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. 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.

The proper selection of equipment is important in preventing exposures. The Project Manager (PM) or health and safety personnel making the selection will have to take several factors into consideration. 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. An accurate assessment of all these factors must be made before work can be safely carried out.

3.1.1 Training

Training shall be provided to all field-based employees in the proper use and care of PPE. Training shall include but is not 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 necessary; how to don, doff, adjust and wear PPE; the limitations of PPE; the proper care, maintenance, useful life 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. Training certifications are provided to employees, which include the date of training and certification subject.

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/or size shall be evaluated to ensure adequate protection for all employees. 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 - Not specifically regulated.
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) - Not specifically regulated.

3.3 Protective Clothing Selection Criteria

3.3.1 Chemicals Present

The most important factor in selecting PPE is the determination of what chemicals the employee may be exposed to. On field investigations, the number of chemicals 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 samples of the air, soil, water, or other site media. 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 taken into consideration 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, be permeated by, or otherwise fail to protect under given circumstances. Fortunately, most manufacturers make guides to the use of 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 in conjunction 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 of time; therefore, the selected material may be adequate for the particular chemical of most concern and 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 different layers of protective materials) affords the best protection.

3.3.2 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/or solid state. Airborne and liquid chemical concentrations should be compared to the OSHA standards and/or 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 it is 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 both the OSHA and ACGIH exposure limit tables.

3.3.3 Physical State

The characteristics of a chemical may range from nontoxic to extremely 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 is dependent on the type of soil and concentration of the chemical. Generally speaking, "dry" soils which have not become airborne do not pose a hazard to site personnel if they take minimal precautions, such as wearing some type of lightweight gloves.

3.3.4 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 different materials can be used which would be considered inadequate under long-term exposures. It should be kept in mind that during manufacturer’s permeation testing, a pure (100% composition) liquid is usually placed in direct contact with the material producing a worst-case situation.

3.3.5 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. The use of leather gloves and a heavy apron over other normal protective clothing will help prevent damage to the normal PPE, and thus reduce worker exposures.

3.3.6 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 a thick glove. Therefore, the PPE selection process must consider the task being performed and provide PPE alternatives or techniques that allow dexterity to be maintained while still protecting the worker (e.g., wearing tight latex gloves over more bulky hand protection to increase dexterity).

3.3.7 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 it can be reused. If the chemical has completely permeated the material, the clothing cannot be adequately decontaminated, and the material should be discarded.

3.3.8 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 very cold temperatures. If there are any questions about the stability of the protective materials under different conditions, the manufacturer should be contacted prior to using PPE in the field.

3.3.9 Work Load

Like climactic conditions, the type of work activity may affect work duration and the ability of personnel 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 ^{o}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 both 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 field work.

3.4 Types of Protective Materials

1. Cellulose or Paper: suitable for nuisance dusts and coarse fibers.
2. Natural and Synthetic Fibers
    a. Tyvek™: suitable small sized hazardous particles, including lead, asbestos, and mold.
    b. Tychem™: suitable for a variety of hazards from light liquid splashes to heavy exposures of industrial chemicals and agents.
    c. Nomex™: 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.

3.5 Protection Levels

3.5.1 Level A Protection

Level A protection (a fully encapsulated suit) is used when skin hazards exist or when there is no known data that 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 the use of 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 suggest a need for Level A protection:

- confined facilities where probability of skin contact is high;
- sites containing known skin hazards;
- sites with no established history to rule out skin and other absorption hazards;
- atmosphere immediately dangerous to life and health (IDLH) through the skin absorption route;
- site exhibiting signs of acute mammalian toxicity (e.g., dead animals, illnesses associated with past entry into site by humans);
- sites at which sealed drums of unknown materials must be opened;
- 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
- 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.

The following items constitute Level A protection:

- open circuit, pressure-demand self-contained breathing apparatus (SCBA);
- totally encapsulated suit;
- gloves, inner (surgical type);
- gloves, outer;
- chemical protective;
- boots, chemical protective, steel toe and shank;
- radiation detector (if applicable); and
- communications.

3.5.2 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.

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 or particulates, or splashes of materials that will affect the skin of personnel.

Personal protective equipment for Level B includes:

- open circuit, pressure-demand SCBA;
- chemical protective clothing:
- overalls and long-sleeve jacket; or
- coveralls;
- gloves, inner (surgical type); gloves, outer, chemical protective;
- boots, chemical protective, steel toe and shank; and
- communications optional.

3.5.3 Level C Protection

Level C protection is utilized when both skin and respiratory hazards are well defined and the criteria for the use of 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 carrying an emergency escape respirator during certain initial entry and site reconnaissance situations, or when applicable thereafter.

Personal protective equipment for Level C typically includes:

- full facepiece air-purifying respirator;
- emergency escape respirator (optional);
- chemical protective clothing:
  - overalls and long-sleeved jacket; or
  - coveralls;
- gloves, inner (surgical type);
- gloves, outer, chemical protective; and
- boots, chemical protective, steel toe and shank.

3.5.4 Level D Protection

Level D is the basic work uniform. Personal protective equipment for Level D includes:

- coveralls;
- safety boots/shoes;
- eye protection;
- hand protection;

- reflective traffic safety vest (mandatory for traffic areas or railyard);
- hard hat (with face shield is optional); and
- emergency escape respirator is optional.

3.5.5 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.

3.5.6 Additional Considerations

Field work 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. The use of disposable shoe coverings when contacting liquid contamination can cut down on overall decontamination requirements. If non-liquids are to be encountered, a Tyvek™ bootie could be used. If the ground contains any sharp objects, the advantage of booties is questionable. Boots should be worn with either cotton or wool socks to help absorb perspiration.
2. If the site situation requires the use of hard hats, chin straps should be used if a person will be stooping over where his/her hat may fall off. Respirator straps should not be placed over the hard hats, as this will affect the fit of the respirator.

   Some types of protective materials conduct heat and cold readily. In cold conditions, natural material clothing should be worn under the protective clothing. Protective clothing should be removed prior to 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 in such a way 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 a 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 that have higher possibilities of vapors, gases or particulates.

APPENDIX F

Subsurface Utility Clearance Management Program

SUBSURFACE UTILITY CLEARANCE MANAGEMENT PROGRAM

CORPORATE HEALTH AND SAFETY DIRECTOR	**:**	**Brian Hobbs, CIH, CSP**
EFFECTIVE DATE	**:**	**01/2019**
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APPENDICES

Appendix A – Roux Subsurface Utility Clearance Checklist

Appendix B – Utility Verification/Site Walkthrough Record

Appendix C – Private Utility Technology Applications and Considerations

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. For the purpose of this program, a structure is defined as any underground feature that may present a 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.

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. Intrusive work activities can include, but are not limited to, digging or scraping the ground 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 concrete slabs on grade.

Deviation from the program, regardless of the specific work activity or location, must be pre-approved based on the client's site knowledge, experience, site conditions, and additional documentation on the site. Any exceptions shall be documented through Roux Subsurface Variance Form located within the Roux Health & Safety Application or through OKTA. Approval is required by the Project Principal (PP) and the Operations Manager (OM) in consultation with the Office Health and Safety Manager (OHSM) or Corporate Health and Safety Director (CHSD) prior to mobilization. Depending on the work location, more stringent federal, state, local, or client requirements may apply. It is the responsibility of the Project Team to identify such requirements prior to mobilization.

3. PROCEDURES

3.1 Before Intrusive Activities/Job Planning Process

Obtain, review, and field verify relevant historical site data that may include: as-builts/site plans; easement/right-of-way information; historical aerial photos/development plans; local/state permitting records; previous site investigation/boring logs; and/or interviews with site representative/client.

If there is the potential for unexploded ordinances or munitions, consultation with your OM and CHSD is required prior to site operations.

During the project kick-off meeting for intrusive activities, the Project Manager (PM) will review the Roux Subsurface Utility Clearance Checklist and Utility Verification (Appendix A) / Site Walkthrough Record (Appendix B) and the below bullet points with the project field team:

- Review the work scope to be performed with the site owner/tenant to determine if it may impact any utilities;
- 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. Use of private utility locating firms, however, does not eliminate the legal requirement for the subcontractor (e.g., driller, excavation 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;
    - A mark-out is defined as 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 must completed prior to intrusive work.
- 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 Utility Mark Out

- 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 Project Team 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 utility mark-outs have 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 the utility locators have located said utilities.

  (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.)
- For non-subscribing utility owners or if public one-call service is not available, perform due diligence and request further information regarding utility type, construction details, location and burial depth directly from public service providers/utility owners. Request utility mark-out by each public service provider/utility owner as applicable. Confirm contact and response by each public utility owner on the Subsurface Utility Clearance Checklist.
- If utilizing a Private Markout, ensure the contractor has a plan regarding what types of technology will be used based on Table 1 in Appendix C: Private Utility Technology Applications and Considerations. If possible, it is recommended that multiple technologies are used to sweep each location/work area. Use tracer wire to locate the utility (when tracer wires are present). Record the results of the private utility mark-out on the Utility Verification/Site Walkthrough Record form.

3.3 Site Visit/Utility Walkthrough

Before mobilization with the subcontractor or during the pre-work safety tailgate with the subcontractor, perform a site walkthrough after utility mark-outs have been completed to determine whether additional potential hazards are present and look for visible signs of utilities that may be present. Outlined below are considerations regarding a site walk before intrusive activities.

- Identify overhead utilities that may impede equipment mobilization or work zones to ensure adequate clearance distance, as specified within the site-specific Health and Safety Plan (HASP).

The presence of any of the following may indicate potential subsurface structures:

- Locations of buildings, equipment, and features like area lights, signs, sprinkler systems, phones, drains, natural gas meters, manholes, etc.;
- Warning tape, which is often a sign of underground services;
- Material like pea gravel, sand, or other non-native materials can indicate the presence of tanks or lines or any deviations from the established native soil or backfill conditions;
- Red concrete, which is often used for electrical duct banks;
- Evidence of damaged utilities, such as piping materials, insulation, or odors present in the work area; and
- Other utilities including fire hydrants, electrical transformers, pipeline markers, valve covers, steam lines, and valve box covers, clean-outs, etc.

By observing the path between the main service line and the connection point, it may be possible to determine the likely routing of on-site utilities. However, this method should not be relied upon solely, and proper risk mitigation strategies should be in place before proceeding with any ground disturbance activities.

For sites with potential underground storage tanks present (UST) the following guidance applies:

- Identify if the product piping is either rigid or flexible.
- Conduct a visual examination of the tank field, observation wells, dispensers, vent stacks, and UST fill points to determine their location.
- Locate and become familiar with the emergency shutoff button/switch, if present.
- Determine the orientation, arrangement, and location of the tanks, as well as their size and capacity, through the examination of visible features at grade, such as fill ports and extractor covers, and by consulting any available as-built drawings.

3.4 Guidance on Preferred Methods of Clearing the Subsurface

At least one of the methods listed below should be carried out during pre-clearing activities. The Project Team is responsible for evaluating risks associated with the Scope of Work to determine which method is selected.

The following approaches shall be considered and implemented based on the Scope of Work.

- Soft Digging
    - This is the preferred method of utility clearance when clearing higher-risk utilities. Soil should be cleared through the use of a vacuum truck/equivalent, an accompanying air knife (preferred), or a water lance to break up and loosen the soil for removal with the vacuum.

- Hand Digging
    - Soil should be broken up and removed using a shovel or other appropriate hand tools without excessive force. Limit the use of dig bars or other tools that, if used incorrectly, can significantly damage utilities.
- Hand Augering
    - The hand auger must be turned slowly without excessive force. Rounded edge augers are preferred. Hand augers should not be used in pea stone/pea gravel where utilities may be present and could potentially be damaged by the hand auger. The abrupt absence of soil recovery in a hand auger could indicate utilities as pea gravel or sand may have spilled out of the auger (exception: native soil conditions that typically result in poor hand auger recoveries).

3.4.1 Guidance for Tools and Equipment

Personnel performing pre-clearance activities shall keep tools and equipment in safe working order and be properly inspected before use. If tools or equipment are broken, they will be tagged and removed from service. The following specify additional guidelines regarding tools and equipment:

- Hand digging tools must have a non-conductive handle, such as fiberglass, wood, or composite, or fully insulated handles and potential contact surfaces.
- Blades on shovels and post-hole diggers should have rounded or blunt edges.
- Pick axes or pointed spades should not be used for physical clearance.
- Crow bars, pinch bars, or pry bars should not be used to break hardened soil or backfill, except when authorized by the SS lead to loosen materials like bricks or larger stones.
- Electric-powered equipment must have ground fault protection.
- Should there be refusal or difficulty with advancing hand tools, the contractor shall stop work and notify the Roux SS/SHSO immediately.

3.5 During Intrusive Activities

Remove any surface coverings (i.e., pavement, brush, debris, etc.) to ensure workers have clear visibility of the work area and subsurface conditions. Avoid mechanical jack hammering over known lines unless they are de-energized, locked-out/tagged-out and potential repairs are planned or if the utility has been completely disconnected prior.

Install Pre-Clearance exploratory test holes using one of the techniques, as outlined in Section 3.4, for the first 5-ft below land surface (BLS) at each location before conducting intrusive mechanized 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 direct push applications (CPT, Geoprobe®, etc.) the borehole clearance diameter shall be at least 125% to the diameter of the largest tool being pushed through the subsurface (e.g., tip, rod, sampler, etc.), to the minimum required depth.
- 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 pre-clearance test holes specified in Section 3.4 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. If considered a high risk utility, additional requirements may be necessary and required by the Operator of the utility. Consultation with the OM and CHSD may be appropriate.

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 the 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 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/mechanical intrusive work 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. State law dictates ticket life; however, at a maximum, ticket life shall not exceed 20 working days.

3.6 Stop Work Authority

Each Roux employee has Stop Work Authority that 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 in consultation with the OHSM after such action is reviewed and resolved. The PM will initiate and execute all management notifications and contact emergency facilities and personnel when this action is appropriate.

Appendix A - Roux Subsurface Utility Clearance Checklist

Roux Subsurface Utility Clearance Checklist ***Date of Revision: 2/2023***

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 (if applicable).				
HASP is available and reviewed by site workers / visitors.				
Subsurface Utility Clearance Procedure has been reviewed with all site workers.				
Mechanical intrusive work activities may not be performed at any location without authorization from the Roux Site Supervisor (SS). Clearance activities may not be performed at any location unless the SS is physically present.				
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. Intrusive work activities cannot be performed in areas that are in direct conflict with any markings made by public or private locators.				
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).				
Mechanical intrusive work is prohibited within 2-feet distance in all directions from subsurface structures that will be intentionally exposed during pre-clearance. Any removal of material within 2-feet of the subsurface structure may only proceed by hand using non-conductive tools/compressed air if authorized by state law and the owner/operator of the utility.				
All equipment onsite must maintain the appropriate horizontal distance from any point on the equipment to the nearest overhead electrical power line. Refer to site-specific HASP and local/utility company requirements.				
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 relied upon solely.

- 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 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 B - Utility Verification/Site Walkthrough Record

Employee Name: _____

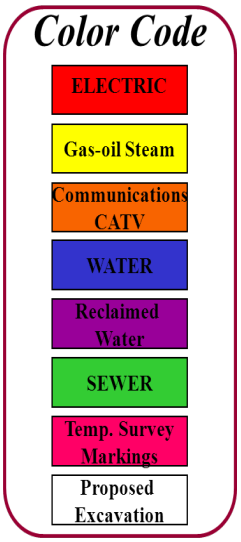
Date: _____

Instructions: For each utility suspected at the job site, indicate the 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 C – Private Utility Technology Applications and Considerations

Technology ⇒ Utility/Object ⇓	Radio Frequency Electro-Magnetic Detector (connection to utility, or induction without connection)	Radio Frequency Electro-Magnetic Detector (passive sweep)	Ground Penetrating Radar (GPR)⊙	Acoustic Plastic Pipe Locator	Beacon, Sonde or Conductive Rodder Insertion	EM-61 (time domain electromagnetics) Ξ	
Power/Instrument Line (Energized/Signaled) □	❋ G	G	G	R	R	G	
Power Line (Non-energized) □	❋ G	R	G	R	R	G	
Sewer/Water Line (Metallic) □	❋ G	Y	G	Y	G	G	
Sewer/Water Line (Non-metallic)	R	R	G	G	❋ G	R	
Instrument / Telecomm Lines (Non-energized)	❋ G	R	G	R	R	Y Only if metallic	
Hydrocarbon Transmission Line (Pipeline)◆ □	❋ G	R	G	R	R	G	
Metallic/Non-Metallic Line (with Tracer Wire)	❋ G	Y	G	Y	Y	G metallic	Y non-metallic
Metallic/Non-Metallic Line (without Tracer Wire)	G metallic	R non-metallic	R	❋ G	Y	Y	G metallic	R non-metallic
Metal or Fiberglass UST	R	R	❋ G	R	R	G metallic	R non-metallic

Additional Considerations

Technology ⇒ Variable ⇓	Radio Frequency Electro-Magnetic Detector	Ground Penetrating Radar (GPR)⊙	Acoustic Pipe Locator	Beacon, Sonde, or Conductive Rodder Insertion	EM-61 Ξ
Moist Soil	G	Y	G	G	G
Dry Soil	Y	G	Y	G	G
Clay	Y	R	G	G	G
Concrete w/Rebar	R	Y	G	G	R
Long Horizontal Profile	G	G	G	G	G
Short Horizontal but Deep Vertical Profile	Y	G	R	R	G
Access to Line✚	G	N/A	G	G	G
No Access to Line✚	G (induction or passive) R (direct connect)	G	R	R	G G
Ferrous Metal	G	G	G	G	G
Non-ferrous Metal	Y	G	G	G	G
Adjacent or crossing conductive utility(ies)	Y	N/A	N/A	N/A	Y

Each site will be unique. Do not use this table as the sole criteria for technology selection. Use it as a starting point to assess available, applicable technology(s).

❋ Indicates best technology for given object. Site structures, rebar in concrete, shallow groundwater tables, perched storm water, etc. can significantly affect performance and reliability of any electro/magnetic method. Other utilities which cross or are adjacent to the target line can cause the EM signal to bleed or jump to the other utility line.

□ Metallic lines that have power running through them or can be connected to a tracer signal generator.

◆ Natural gas pipeline locating technicians must be trained/certified (in the U.S. requires DOT and Office of Pipeline Safety standards, other regions may have similar certification or requirements).

⊙ Most sensitive to interpretation. The skill, training and experience of operator are critical.

Ξ Emerging technology with limited availability.

✚ Access: induce unique electronic signature, apply acoustical impulse or insert conductive rodder/beacon/sonde.

Green Generally, an applicable technology **Yellow** May or may not be applicable **Red** Not generally applicable

APPENDIX G

Heavy Equipment Exclusion Zone Policy

HEAVY EQUIPMENT EXCLUSION ZONE MANAGEMENT PROGRAM

CORPORATE HEALTH AND SAFETY DIRECTOR	**:**	**Brian Hobbs, CIH, CSP**
EFFECTIVE DATE	**:**	**01/2019**
REVIEWED DATE	**:**	**01/2023**
REVISION NUMBER	**:**	**2**

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1. PURPOSE

The Exclusion Zone Management Program aims 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 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 fieldwork 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 (e.g., 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 fall zone of equipment and their contents; and
    - Greater than the tip-over distance of the heavy equipment.

    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 Planning Process and incorporated in the pre-job tailgate meeting with the subcontractor(s), including any updates to our Job Safety Analysis (JSA).
2. The spotter (or another authorized individual) should be assigned responsibility for enforcing the Exclusion Zone and not conduct any other task. 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 a spotter must be within the Exclusion Zone they must be in a designated area which is outside the swing/tip radius, fall zone, line of fire of lifted loads, etc. If multiple pieces of equipment are being used, one spotter may be adequate so long as there is clear line of sight, and the spotter has the ability to control the zone(s). Radios would be required since hand signals would not be adequate if two pieces of equipment were running.

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 (e.g., 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 bring the boom/arm to the ground ensuring a “Zero Energy State” and 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 exited the Exclusion Zone's limits 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 fences should be used. For certain types of widespread 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 the 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. Logistics involving heavy equipment should be understood and discussed prior to the field event during the job planning phase.

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 (PP), Office Health and Safety Manager (OHSM) 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. However, all Roux employees are provided initial training and annually thereafter on our Heavy Equipment Exclusion zone policy.

5. STOP WORK AUTHORITY

All Roux employees and their subcontractors have stop-work authority. Upon initial hire all Roux employees are provided initial new hire safety orientation which includes stop work authority. All Roux employees and

subcontractors are encouraged to stop work whenever any task or operation presents unreasonable risk or employees are unsure how to carry out work safely. This includes upon observing any questionable safety-related behavior or condition; they are to stop work immediately and discuss the behavior or condition with the individual(s) involved. Upon stopping work, the following actions will be taken:

- Notify the affects parties to include the Project Manager (PM) and Site Health and Safety Officer (SHSO);
- Work to correct the situation or not proceed until the situation is corrected by the PM or SHSO;
- Resume work only when told to proceed by either the PM or SHSO;
- The SHSO will document the Stop-Work intervention in the field logbook;
- Stop-Work interventions will be reported to the PP for review to ensure the interventions are closed; and
- The PP will share learnings from the interventions, as appropriate, throughout the firm, to demonstrate the importance of the Stop-Work interventions.

Please note: Any form of retribution or intimidation directed at any individual or company for exercising their right to issue a stop work authority will not be tolerated.